ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

2021

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2021

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Edita
Instituto de Ciencia de Materiales de Sevilla (ICMS)
Centro Mixto entre el Consejo Superior de Investigaciones
Científicas y la Universidad de Sevilla
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Coordinadora de Divulgación Científica ICMS
T. Cristina Rojas Ruiz

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Presentación

A través de esta Memoria 2021, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo del pasado año. El ICMS es un centro mixto del Consejo Superior de Investigaciones Científicas (CSIC) y la Universidad de Sevilla (US), cofinanciado por la Junta de Andalucía. Fue creado en 1986 y está integrado por personal científico de la Universidad de Sevilla y el CSIC. A finales de 2021, contaba con 129 empleados, de los cuales 55 eran científicos permanentes.

De acuerdo con la estructura establecida en el vigente Plan Estratégico 2018-2021, nuestro centro se organiza en torno a 5 departamentos, cuyos miembros abordan temáticas tanto fundamentales como aplicadas. Así, nuestras investigaciones abordan problemas candentes de la Química y Física del Estado Sólido, la Catálisis Heterogénea, la Cerámica, la Óptica, la Ciencia de Superficies, las Energías Renovables y el Medio Ambiente, etc. Entre ellas, podemos resaltar el aprovechamiento de las energías solar y de biomasa, la generación de H_2 y otros combustibles limpios y de origen renovable, el aprovechamiento y conversión química del CO_2, la depuración de efluentes químicos, el secuestro de residuos radioactivos, la mejora de la selectividad y rendimiento de reacciones químicas básicas, el aumento de la eficiencia de los dispositivos generadores de energía renovable (celdas fotovoltaicas, electrodos, generadores piezoeléctricos, celdas de combustible o baterías) y de los emisores de luz (LEDs), el desarrollo y la mejora de sensores químicos y agentes de contraste radiológico, de materiales biocompatibles para implantes quirúrgicos, de pigmentos cerámicos, de recubrimientos hidrofóbicos o hidrofílicos, recubrimientos hielofóbicos,… y un largo etcétera.

Nuestros principales empeños para los años venideros son realizar investigaciones que se sitúen en la vanguardia de la Ciencia e Ingeniería de Materiales y que éstas sirvan de apoyo al desarrollo en Andalucía de un nuevo sistema económico, en el que las bases científica y tecnológica pasen a ser pilares fundamentales.

Dr. Juan Pedro Espinós Manzorro
Director del Instituto de Ciencia de Materiales de Sevilla

Presentation

Through this Activity Report 2021, the Institute of Materials Science of Seville (ICMS) is communicating the best of its scientific activities carried out for the last year. The ICMS is a joint centre of the Spanish Research Council (CSIC) and the University of Seville (US), also funded by the Junta de Andalucía. It was founded in 1986, and integrated by scientific staff of the University of Seville and the CSIC. At the end of last year, it includes 129 people, 55 of them as permanent scientific staff.

The current Strategic Plan 2018-2021 organizes our Centre in 5 departments, which personnel address both fundamental and applied research. Thus, our researchers address hot topics related with disciplines of Solid State Chemistry and Physics, Heterogeneous Catalysis, Ceramics, Optics, Surface Science, Renewable Energy and Environment, etc., Among them, we could mention the exploitation of solar energy and biomass, the generation of H_2 and other clean and renewable fuels, the exploitation and chemical conversion of CO_2, the removal of pollutants, the storage of radioactive wastes, the improvement of the selectivity and yield of basic chemical reactions, the efficiency of renewable energy generators (photovoltaic cells, electrodes, piezoelectric devices, fuel cells and batteries) and light emitting devices (LEDs), the development and improvement of chemical sensors and radiological contrast agents, the production of biocompatible scaffolds for surgical implants, the production of ceramic pigments, many different coatings and thin films, … and a long et cetera.

Our main efforts for near future are to cover the most modern and innovative aspects of the current Material Science and Material Engineering, as well as to support in the region of Andalusia, the development of a new economic system, in which scientific and technological basis should become fundamental pillars.

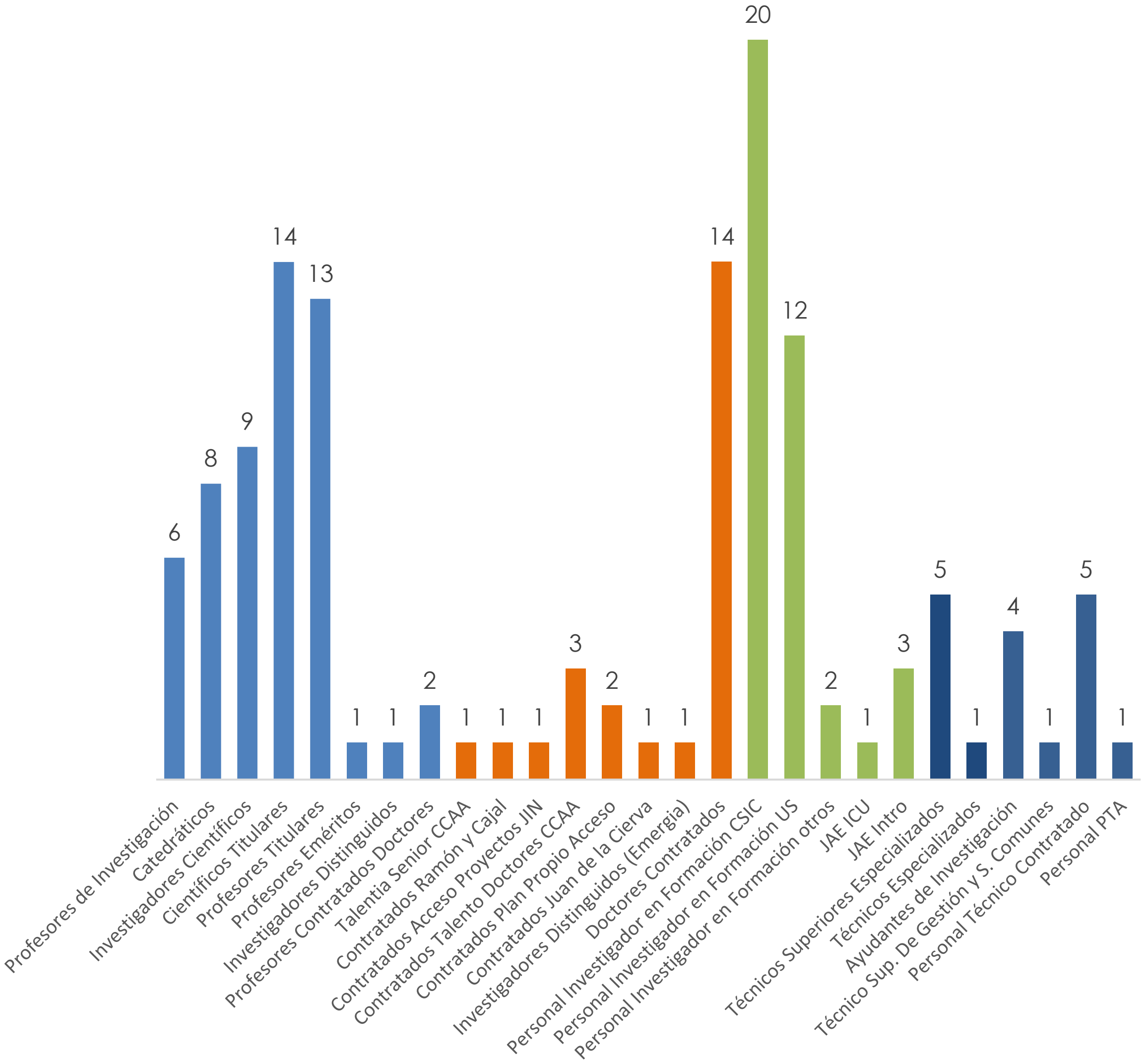
Dr. Juan Pedro Espinós Manzorro
Director del Instituto de Ciencia de Materiales de Sevilla

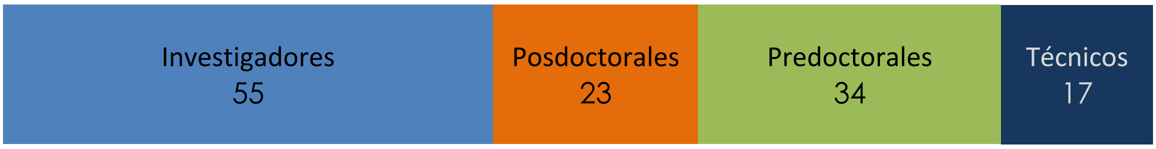
El ICMS en 2021
ICMS in 2021

Datos Estadísticos del ICMS
Statistical Data of ICMS

RECURSOS HUMANOS / HUMAN RESOURCES

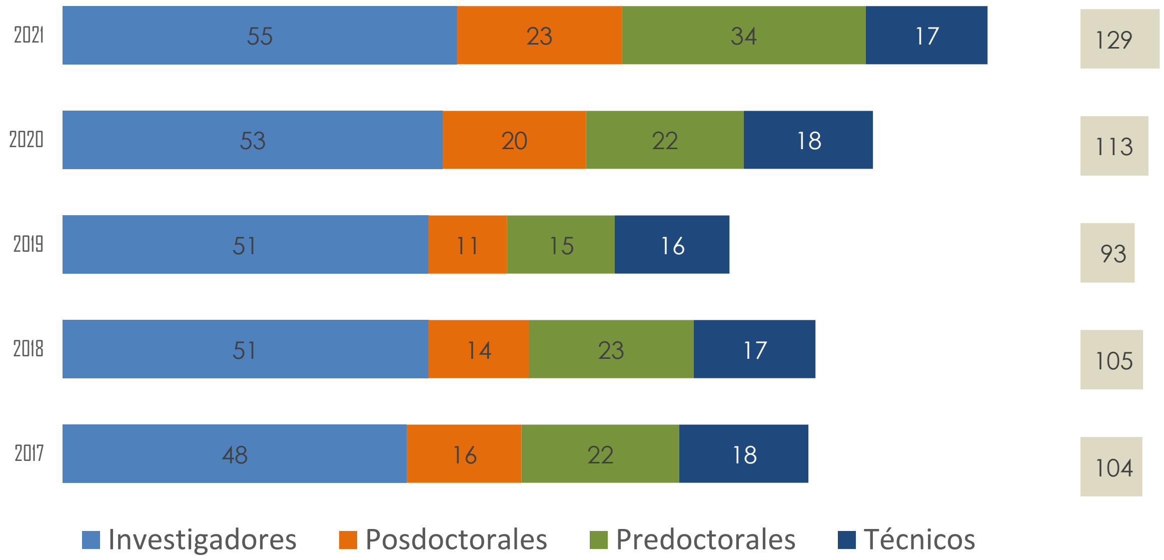
Distribución del personal por categorías – 2021
Distribution by professional category – 2021



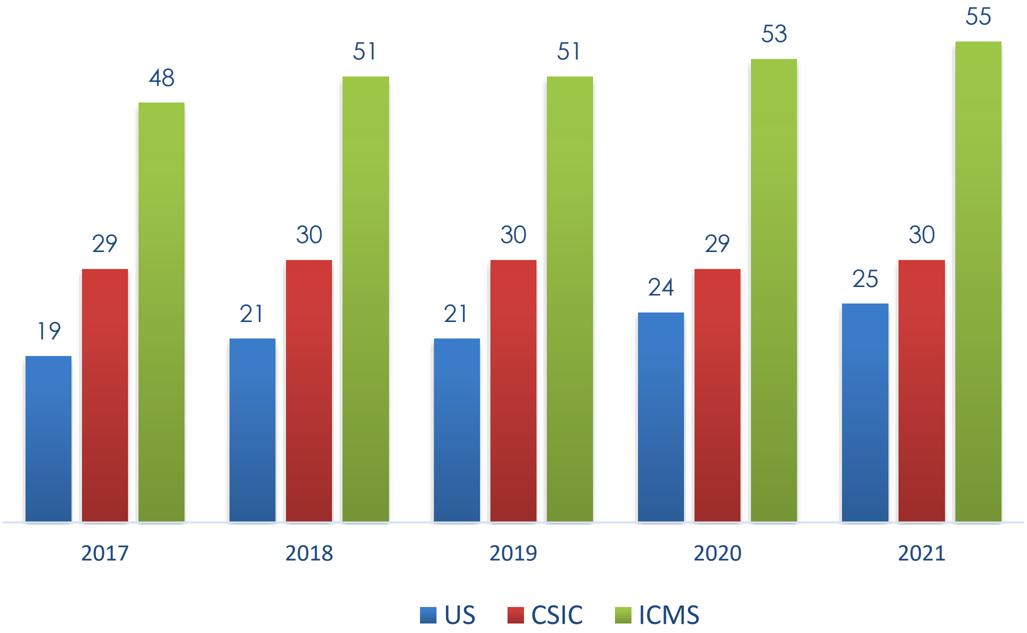


* Datos a 31 de Diciembre de 2021

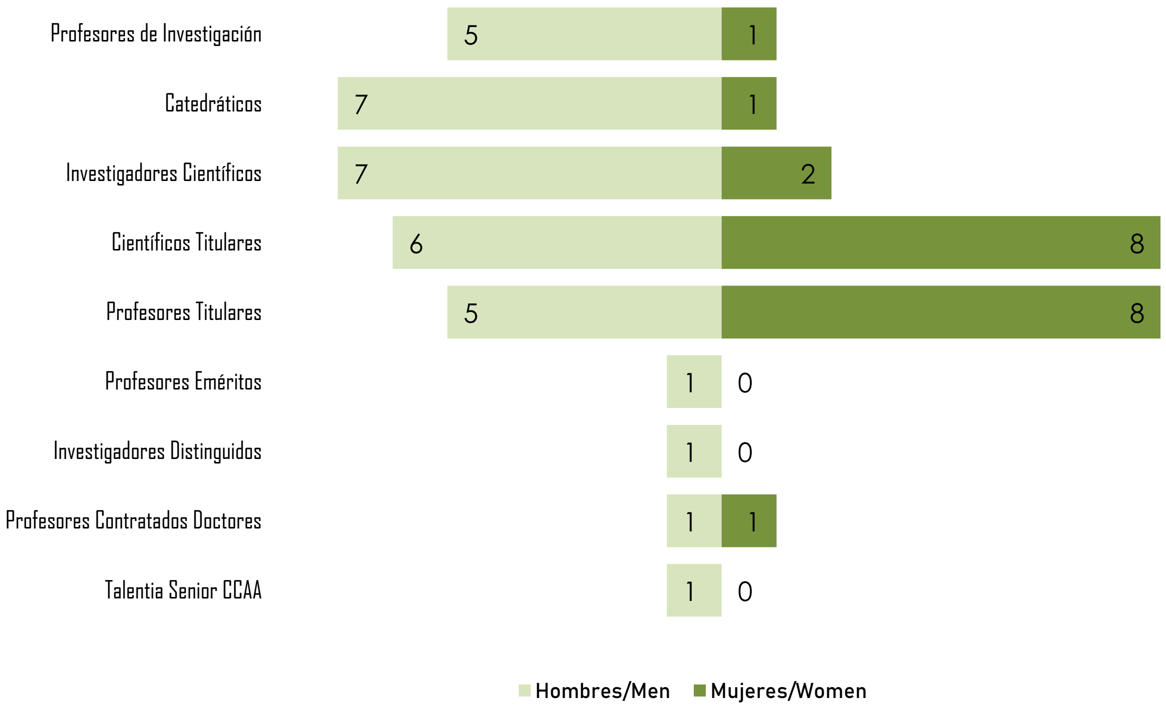
Evolución 2017-2021 del personal
Evolution of Staff

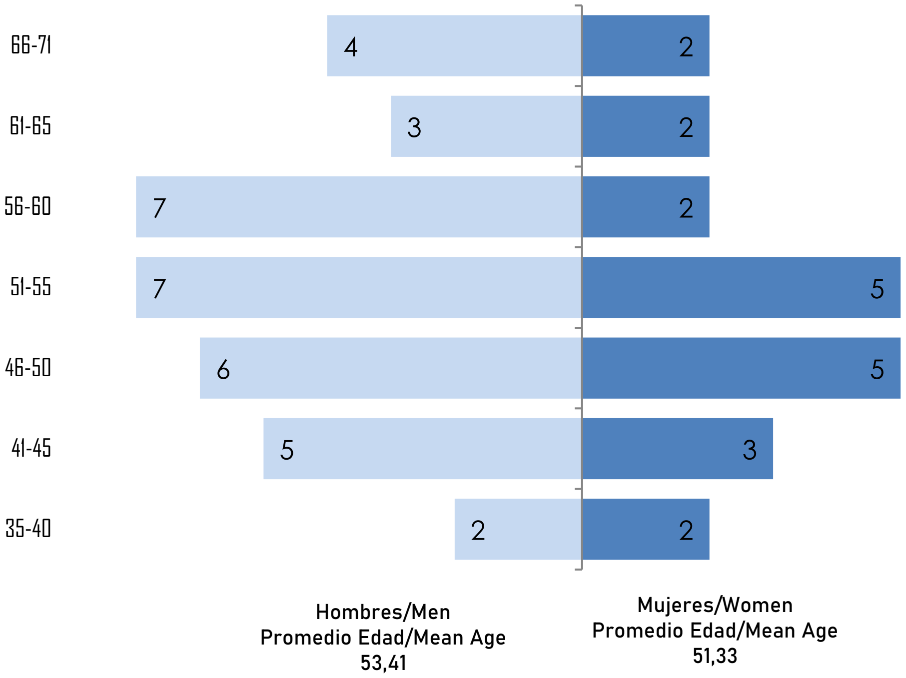


Evolución 2017-2021 del Personal Investigador
Evolution of Research Staff



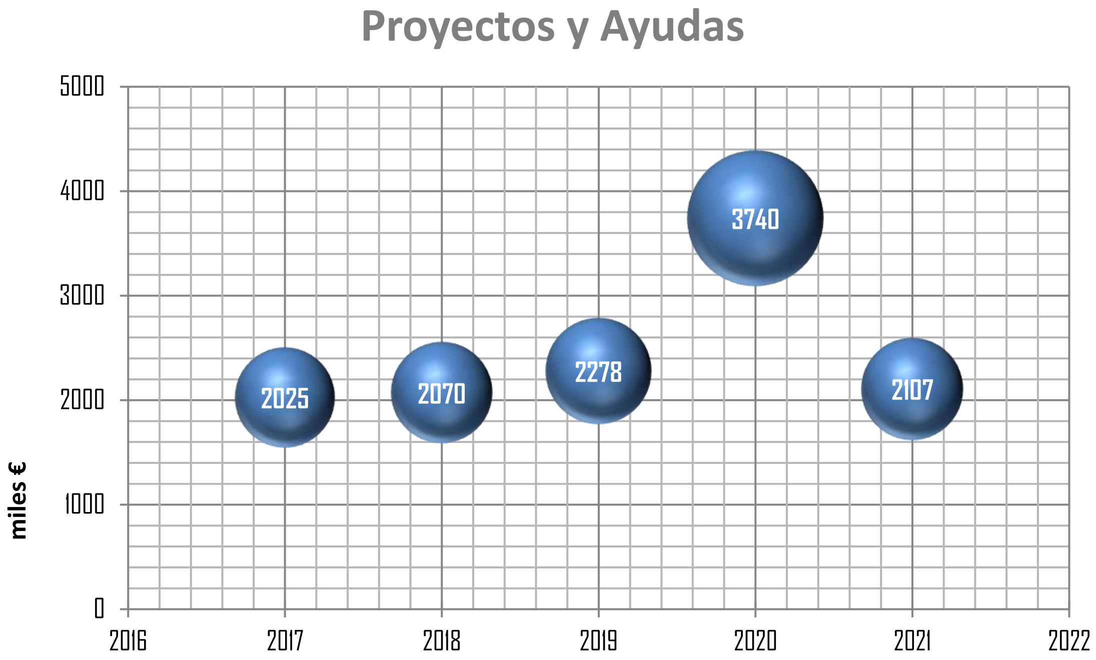
Distribución del personal Investigador (sexo y edad)
Distribution by Scientific staff (gender and age)





FINANCIACIÓN / FUNDING

Evolución 2017-2021 de la Financiación conseguida por año (miles€)(PCO)
Evolution of the funding of the ICMS



Producción Científica / Scientific production

Revistas SCI en las que se ha publicado algún artículo, por orden de Factor de Impacto
Number of Scientific Papers published in a Specific Journal Arranged by Their Impact Factor (SCI)

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT	
APPLIED CATALYSIS B-ENVIRONMENTAL	3	24,319	D1	Q1
CHEMICAL ENGINEERING JOURNAL	3	16,744	D1	Q1
MATERIALS HORIZONS	1	15,717	D1	Q1
SCIENCE OF THE TOTAL ENVIRONMENT	2	10,753	D1	Q1
CARBOHYDRATE POLYMERS	1	10,723	D1	Q1
CHEMISTRY OF MATERIALS	1	10,508	-	Q1
ACS APPLIED MATERIALS & INTERFACES	3	10,380	-	Q1
ADVANCED OPTICAL MATERIALS	4	10,050	D1	Q1
JOURNAL OF COLLOID AND INTERFACE SCIENCE	2	9,965	-	Q1
JOURNAL OF POWER SOURCES	1	9,794	-	Q1
PROGRESS IN NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY	1	9,447	D1	Q1
ACS SUSTAINABLE CHEMISTRY & ENGINEERING	2	9,224	D1	Q1
SENSORS AND ACTUATORS B-CHEMICAL	1	9,221	D1	Q1
SOLAR RRL	2	9,173	-	Q1
SEPARATION AND PURIFICATION TECHNOLOGY	1	9,136	D1	Q1
CHEMOSPHERE	1	8,943	-	Q1
JOURNAL OF ENERGY STORAGE	1	8,907	-	Q1
APPLIED MATERIALS TODAY	1	8,663	-	Q1
MATERIALS SCIENCE & ENGINEERING C-MATERIALS FOR BIOLOGICAL APPLICATIONS	1	8,457	-	Q1
JOURNAL OF CO_2 UTILIZATION	2	8,321	-	Q1
NANOSCALE	1	8,307	-	Q1
JOURNAL OF MATERIALS CHEMISTRY C	1	8,067	-	Q1
FUEL	1	8,035	-	Q1
INTERNATIONAL JOURNAL OF BIOLOGICAL MACROMOLECULES	1	8,025	D1	Q1
JOURNAL OF ENVIRONMENTAL CHEMICAL ENGINEERING	2	7,968	-	Q1
APPLIED SURFACE SCIENCE	2	7,392	D1	Q1

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT	
JOURNAL OF WATER PROCESS ENGINEERING	1	7,340	D1	Q1
ELECTROCHIMICA ACTA	1	7,336	-	Q1
SOLAR ENERGY MATERIALS AND SOLAR CELLS	1	7,305	-	Q1
JOURNAL OF BUILDING ENGINEERING	1	7,144	-	Q1
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	1	7,139	-	Q2
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	3	6,888	-	Q1
FRONTIERS IN PLANT SCIENCE	1	6,627	-	Q1
CATALYSIS TODAY	1	6,562	-	Q1
ADVANCED MATERIALS INTERFACES	1	6,389	-	Q2
JOURNAL OF ALLOYS AND COMPOUNDS	3	6,371	D1	Q1
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	1	6,364	D1	Q1
SCRIPTA MATERIALIA	1	6,302	D1	Q1
JOURNAL OF MATERIALS RESEARCH AND TECHNOLOGY	1	6,267	-	Q1
ASTRONOMY & ASTROPHYSICS	1	6,240	-	Q1
ACS APPLIED NANO MATERIALS	1	6,140	-	Q2
APPLIED CLAY SCIENCE	1	5,907	D1	Q1
MICROPOROUS AND MESOPOROUS MATERIALS	2	5,876	-	Q1
APPLIED CATALYSIS A-GENERAL	3	5,723	-	Q2
NANOMATERIALS	6	5,719	-	Q1
NANOSCALE ADVANCES	1	5,598	-	Q2
FRONTIERS IN CHEMISTRY	3	5,545	-	Q2
CERAMICS INTERNATIONAL	4	5,532	-	Q1
CHEMCATCHEM	2	5,497	-	Q2
INORGANIC CHEMISTRY	1	5,436	-	Q1
POLYMER DEGRADATION AND STABILITY	1	5,204	-	Q1
REACTION CHEMISTRY & ENGINEERING	2	5,200	-	Q2
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	2	5,141	-	Q2
EUROPEAN JOURNAL OF PHARMACEUTICAL SCIENCES	1	5,112	-	Q2
POLYMERS	1	4,967	-	Q1
MOLECULES	1	4,927	-	Q2
SURFACE & COATINGS TECHNOLOGY	4	4,865	-	Q2
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	1	4,804		Q1

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT	
INTERNATIONAL JOURNAL OF ENERGY RESEARCH	1	4,672	D1	Q1
DALTON TRANSACTIONS	1	4,569	-	Q1
CATALYSTS	3	4,501	-	Q2
CHEMICAL ENGINEERING RESEARCH & DESIGN	1	4,119	-	Q2
RSC ADVANCES	1	4,036	-	Q2
CRYSTAL GROWTH & DESIGN	1	4,010	-	Q1
KONA POWDER AND PARTICLE JOURNAL	1	3,919	-	Q2
PLASMA PROCESSES AND POLYMERS	2	3,877	-	Q1
OPTICAL MATERIALS	2	3,754	-	Q2
MATERIALS	4	3,748	-	Q1
BIOMIMETICS	1	3,743	-	Q2
JOURNAL OF MATERIALS IN CIVIL ENGINEERING	1	3,651	-	Q2
MATERIALS LETTERS	2	3,574	-	Q2
WATER	2	3,530	-	Q2
BOLETIN DE LA SOCIEDAD ESPAÑOLA DE CERAMICA Y VIDRIO	2	3,483	-	Q1
JOURNAL OF PHYSICS D-APPLIED PHYSICS	1	3,409	-	Q2
PROCESSES	1	3,352	-	Q2
INORGANICS	1	3,149	-	Q2
CATALYSIS LETTERS	1	2,936	-	Q3
JOURNAL OF APPLIED PHYSICS	2	2,877	-	Q2
HERITAGE SCIENCE	1	2,843	-	Q2
MINERALS	1	2,818	-	Q2
JOURNAL OF MATERIALS SCIENCE-MATERIALS IN ELECTRONICS	1	2,779	-	Q2
METALS	1	2,695	-	Q2
CRYSTALS	1	2,670	-	Q2
EUROPEAN PHYSICAL JOURNAL-APPLIED PHYSICS	1	1,168	-	Q4
ACS APPLIED BIO MATERIALS	1	-	-	-
Total	**135**	**6,730**	**18**	**Q1=64%**

(*) Factor de Impacto correspondiente al año 2021
Journal Citation Reports of 2021

COMPOSICIÓN Y ESTRUCTURA

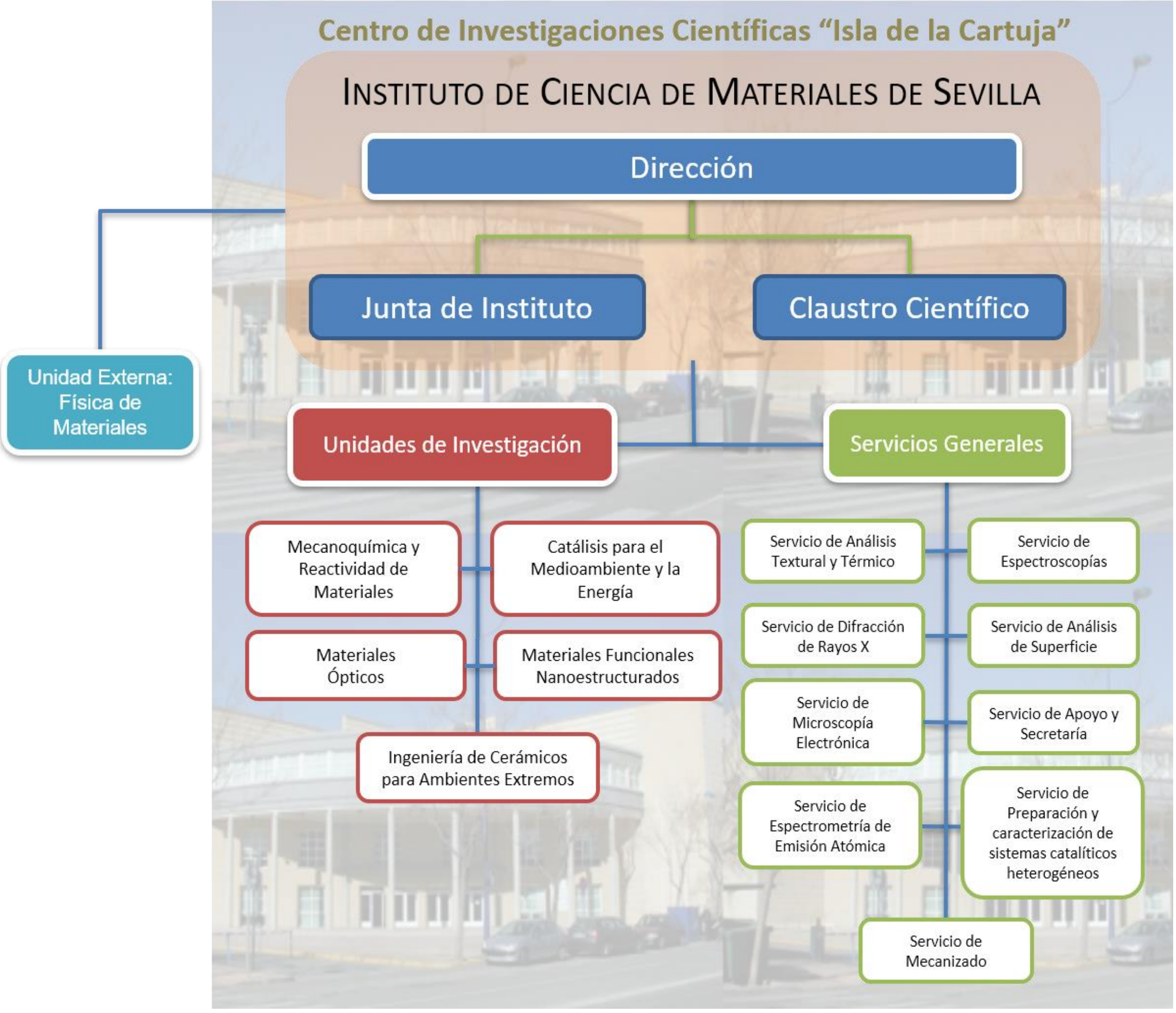
STRUCTURE AND ORGANISATION

EL INSTITUTO / THE INSTITUTE

El Instituto de Ciencia de Materiales de Sevilla (ICMS) fue creado en 1.986. En 1996 se trasladó a unos nuevos locales en la Isla de la Cartuja, integrándose, junto con otros dos Institutos Mixtos, en el Centro de Investigaciones Científicas Isla de la Cartuja (Junta de Andalucía - Consejo Superior de Investigaciones Científicas - Universidad de Sevilla). El ICMS se estructura en cinco Unidades de Investigación ubicadas en el edificio de la Isla de la Cartuja y una Unidad Externa en el campus de Reina Mercedes, Facultad de Física. En la actualidad, el Instituto está formado por grupos de investigación del CSIC y de la Universidad de Sevilla. Estos grupos tratan de aunar sus esfuerzos en diversas áreas de la física y química del estado sólido, físico-química de superficies y otras disciplinas relacionadas con la Ciencia de Materiales. Esta actividad persigue contribuir al desarrollo científico dentro de los Planes de Investigación tanto Autonómicos como Nacionales en el área de la Ciencia y Tecnología de Materiales, así como de las equivalentes de la UE.

The Institute of Materials Science of Seville (ICMS) was created in 1986. In 1996, it moved to new premises at the Isla de la Cartuja, combining with two other Mixed Institutes to make up the Isla de la Cartuja Scientific Research Centre (Junta de Andalucía - Consejo Superior de Investigaciones Científicas - Universidad de Sevilla). The Institute comprises three research units housed in the building at the Isla de la Cartuja and an external unit at the Physics Faculty on the Reina Mercedes campus. Today it incorporates research groups of CSIC and the University of Seville. The aim of these groups is to unite efforts in various areas of solid-state physics and chemistry, physical chemistry of surfaces, and other related disciplines of materials science. Such activity seeks to contribute to scientific development within the Research Plans of both regional and state authorities, and their EU equivalents, in the field of materials science and technology.

ORGANIGRAMA / ORGANIZATION CHART



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Borrás Martos, Ana Isabel
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Budagosky Marcilla, Jorge A.

C

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González Mancebo, Daniel
Gotor Martínez, Francisco José

H

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I

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J

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L

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López Santos, Carmen
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M

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Martínez Blanes, José María
Martínez Fernández, Julián
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Míguez García, Hernán Ruy
Moreno García, Virginia
Muñoz Bernábé, Antonio

N

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Núñez Álvarez, Nuria Ofelia

O

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Odriozola Gordón, José Antonio
Oliva Ramírez, Manuel

P

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Palmero Acebedo, Alberto
Penkova, Anna Dimitrova
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Pereñíguez Rodríguez, Rosa
Pérez Maqueda, Luis Allan
Poyato Galán, Rosalía

R

Ramírez Rico, Joaquín
Ramírez Reina, Tomás
Real Pérez, Concepción
Rico Gavira, J. Víctor
R. González-Elipe, Agustín
Rojas Ruiz, T. Cristina
Romero Sarria, Francisca
Ruiz López, Estela

S

Sánchez Jiménez, Pedro E.
Sánchez López, Juan Carlos
Sánchez Soto, Pedro José
Sánchez Valencia, Juan Ramón
Sayagués De Vega, M. Jesús

T

Thi Tuyen, Ngo

V

Vattier Lagarrigue, Florencia

Y

Yubero Valencia, Francisco

DIRECTORIO / DIRECTORY

UNIDADES DE INVESTIGACIÓN / RESEARCH UNITS

CATÁLISIS PARA EL MEDIOAMBIENTE Y LA ENERGÍA / CATALYSIS FOR ENVIROMENT AND ENERGY

Catedráticos	Dr. Alfonso Caballero Martínez
	Dr. José Antonio Odriozola Gordón
Investigadores Científicos	Dr. Miguel Ángel Centeno Gallego
	Dr. Gerardo Colón Ibáñez
Científicos Titulares	Dra. M. Carmen Hidalgo López
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Profesores Titulares	Dr. José Manuel Córdoba Gallego
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	Dra. Svetlana Lyubomirova Ivanova
	Dra. Leidy Marcela Martínez Tejada
	Dra. Anna Dimitrova Penkova
	Dra. Rosa Pereñíguez Rodríguez
	Dra. Francisca Romero Sarria
Profesores Eméritos	Dr. José Antonio Navío Santos
Profesores Contratados Doctores	Dr. Luis Bobadilla Baladrón
Contratados Ramón y Cajal	Dr. Tomás Ramírez Reina
Contratados Acceso Proyectos JIN	Dra. Laura Pastor Pérez
Doctores Contratados	Dra. Estela Ruiz López
Personal Investigador en Formación	Gda. Débora Álvarez Hernández
	Gdo. Sergio Carrasco Ruiz
	Gda. Ligia Amelia Luque Álvarez
	Lda. Ángeles María López Martín
	Gdo. Juan Luis Martín Espejo
	Ldo. Francisco Jesús Platero Moreno
	Ldo. Felipe Rubén Puga Martínez
	Gda. María Ribota Peláez
	Gdo. Guillermo Torres Sempere

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS
ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

Catedráticos	Dr. Miguel Ángel Castro Arroyo
	Dr. Manuel Jiménez Melendo
	Dra. Pilar Malet Maenner
	Dr. Julián Martínez Fernández
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Científicos Titulares	Dr. José Jesús Benítez Jiménez
Profesores Titulares	Dr. Alfonso Bravo León
	Dr. Joaquín Ramírez Rico
Doctores Contratados	Dra. Esperanza Pavón González
Personal Investigador en Formación	Gdo. Javier Ramiro Chaparro Barajas
	Gda. Sol Fernández Muñoz

MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES
MECHANOCHEMISTRY AND REACTIVITY OF MATERIALS

Profesores de Investigación	Dr. Luis Allan Pérez Maqueda
Catedráticos	Dr. Diego Gómez García
Investigadores Científicos	Dr. Francisco José Gotor Martínez
	Dra. Concepción Real Pérez
	Dr. Pedro José Sánchez Soto
Científicos Titulares	Dra. Rosalía Poyato Galán
	Dra. María Jesús Sayagués de Vega
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CATALYSIS FOR ENVIRONMENT AND ENERGY

GRUPOS DE INVESTIGACIÓN

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Heterogeneus Photocatalysis: Applications [642005]
http://materphotocat.ciccartuja.es

Materiales y Procesos Catalíticos de Interés Ambiental y Energético
Materials and catalytic processes for environment and energy [642004]
http://matproner.icms.us-csic.es

Química de Superficies y Catálisis
Surface Chemistry and Catalysis [642006]
http://surfcat.icms.us-csic.es

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	Dr. Gerardo Colón Ibáñez
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	Dr. Juan Pedro Holgado Vázquez
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	Dra. Svetlana Lyubomirova Ivanova
	Dra. Leidy Marcela Martínez Tejada
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PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Acido fórmico como vector de energía: de la biomasa al hidrógeno verde	**Formic acid as energetic vector: from biomass to green hydrogen**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2020-113809RB-C32
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	263.780 €
Investigador responsable/Research head:	Miguel Angel Centeno Gallego y Svetlana Ivanova
Componentes/Research group:	Leidy Marcela Martínez Tejada, María Isabel Domínguez Leal

RESUMEN / ABSTRACT

El presente proyecto forma parte del proyecto coordinado ENERCATH2 que pretende integrar una estrategia que involucra múltiples reacciones para la producción y uso de hidrógeno verde a partir de la biomasa. El objetivo último es contribuir al desarrollo de tecnologías energéticas sostenibles que sustituyan a las actuales, derivadas de las fuentes fósiles. Específicamente, el proyecto del ICMS se centra en el uso del ácido fórmico como vector energético de hidrógeno, dado que es un compuesto químico líquido con una alta densidad gravimétrica de energía, que puede ser almacenado, transportado y manipulado de manera segura usando la infraestructura existente de distribución de hidrocarburos.

El objetivo principal del proyecto es la generación de ácido fórmico a partir de biomasa lignocelulósica y la posterior obtención de corrientes de hidrógeno a partir de éste. Para este fin, se pretenden desarrollar catalizadores novedosos, preferiblemente basados en carbones derivados de la biomasa y/o en metales de transición, no nobles, (V, Ni, Cu, Co, etc.), activos, selectivos y estables, para: i) la oxidación directa y selectiva de la biomasa lignocelulósica, e.g. glucosa, bien hacia la producción masiva de ácido fórmico, bien hacia la producción de una mezcla de ácido fórmico con otros co-productos, tales como el ácido levulínico, que pueden servir como punto de partida para la generación de productos plataforma de interés industrial, intermedios en la producción de combustibles y ii) la deshidrogenación de ácido fórmico, tanto en fase líquida como gaseosa, para la producción de corrientes de hidrógeno libres de CO.

Los catalizadores preparados serán caracterizados estructural y químicamente por una gran variedad de técnicas (DRX, XPS, SEM, HRTEM, Raman, DRIFTS, TPR/TPD, UV-Vis, Análisis textural), tanto pre- como post-reacción, para evaluar las posibles modificaciones ocurridas en el transcurso de la misma. Igualmente, se realizarán estudios en condiciones de reacción (in-situ y operando) por espectroscopias DRIFTS y ATR, lo que, junto con los resultados de actividad y de caracterización, permitirá analizar el mecanismo de las reacciones y así poder establecer la relación estructura-actividad en cada caso. El conocimiento de esta relación permitirá optimizar el catalizador diseñado y, en última instancia, cada proceso catalítico de producción de vectores sostenibles de energía propuesto en el proyecto.

This project is part of the ENERCATH2 coordinated project that aims to integrate a multi reaction catalytic strategy for green-hydrogen and energy related vectors production and use from biomass in order to contribute to the development of sustainable energy technologies that replace current ones derived from fossil sources. Specifically, ICMS project focuses on the production of formic acid as hydrogen related vector Formic acid is a liquid chemical compound with a high gravimetric energy density, which can be safely stored, transported and manipulated using existing hydrocarbon distribution infrastructure.

The main objective of the project is formic acid generation from lignocellulosic biomass and its subsequent dehydrogenation to green hydrogen. For this purpose, it will be intended to develop a series of novel catalysts, preferably based on biomass-derived carbons and/or on non-noble transition metals (V, Ni, Cu, Co etc), active, selective and stable for i) direct and selective oxidation of lignocellulosic biomass, using glucose as representing molecule, either towards the massive production of formic acid, or towards the production of a mixture of formic and co-product levulinic acid, which serves as a starting point for the generation of intermediate platform products and commodities of industrial interest in the production of fuels and polymers and for ii) the dehydrogenation of formic acid, both in liquid and gas phase, for the production of CO-free hydrogen streams.

After the stages of preparation-functionalization and reaction, the catalysts will be structurally and chemically characterized using a wide variety of techniques available by the whole consortium (XRD, XPS, SEM, HRTEM, Raman, DRIFTS, TPR/TPD, TGA, UV-Vis, Textural Analysis). These results, in addition to the in-situ/operando DRIFTS and ATR spectroscopic ones will give us fundamental information of the reaction mechanisms, allowing to establish structure-activity relationships for the studied reactions. The knowledge of these relationships will contribute to the understanding and optimization of the designed catalysts, and the catalytic process involved on the production of sustainable energy vectors proposed in the project.

Avanzando hacia la economía circular: Biocombustibles para el transporte pesado, a partir del reciclado de residuos (NICER BIOFUELS)	**steppiNg towards Circular EConomy: REcycling bio-waste into heavy tRansport BIOFUELS (NICER-BIOFUELS)**
MINISTERIO DE CIENCIA E INNOVACIÓN Financiado por la Unión Europea NextGenerationEU Plan de Recuperación, Transformación y Resiliencia AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PLEC2021-008086
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	278.900 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón y Tomás Ramírez Reina
Componentes/Research group:	María Isabel Domínguez Leal, Laura Pastor Pérez

RESUMEN / ABSTRACT

Financiado por el programa RETOS-COLABORACION PUBLICO-PRIVADA del Ministerio de Ciencia e Innovación con fondos EU bajo el marco Next Generation Europe, NICER BIOFUELS es fruto de la colaboración entre las Universidades de Zaragoza y Sevilla y la multinacional URBASER. En el contexto de la economía circular y el desarrollo de combustibles sostenibles que permitan descarbonizar el transporte y avanzar hacia una sociedad libre de emisiones, NICER-BIOFUELS representa un paso adelante para combatir el cambio climático combinando ciencia fundamental e ingeniería aplicada.

NICER-BIOFUELS aims to create a unique knowledge infrastructure that supports the decentralised, sustainable and cost-efficient conversion of biowastes and textile residues to sustainable Heavy Transport Biofuels (HTB) to contribute towards full transport system decarbonisation. The project targets the development of disruptive technologies that overcome critical technological barriers, increase process efficiency and reduce marginal costs in the bio-waste to HTB conversion process. Following the spirit of circular economy, the overriding idea of NICER-BIOFUELS is to combine CO_2 emissions with bio-waste as a carbon pool to produce the next generation of HTB. Such an ambitious goal will be achieved by integrating advanced gasification strategies, unique catalytic technologies and digital tools to deliver fuel processors which are adaptable to feedstock input and HTB demands.

Conversión Avanzada de Biogas a Ácido Acético: Soluciones Catalíticas para una Sociedad con Bajas Emisiones de Carbono	**ADVanced convErsioN of biogas To acetic acid: catalytic solUtions for a low caRbon sociEty (ADVENTURE)**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PID2019-108502RJ-100
Periodo/Period:	01-10-2020 / 30-09-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	179.080 €
Investigador responsable/Research head:	Laura Pastor Pérez

RESUMEN / ABSTRACT

En ADVENTURE se presenta un nuevo concepto para convertir biogás, procedente de desechos orgánicos, en productos químicos de alto valor industrial, como es el ácido acético (AA), de una manera tanto amigable con el medio ambiente como viable económicamente. El AA se emplea como precursor de muchos productos procedentes de la química fina, con numerosas aplicaciones, como son la fabricación de pinturas y recubrimientos, la producción de plásticos y adhesivos basados en agua, entre muchos otros, siendo una molécula plataforma muy versátil para la industria química. Tradicionalmente el AA se produce a escala comercial a través de una ruta indirecta produciendo una considerable huella global de CO_2. Por ello, el objetivo principal de ADVENTURE es rediseñar el proceso de producción de AA introduciendo biogás como principal materia prima - un enfoque completamente nuevo que provoca una sinergia entre la utilización de CO_2 y la síntesis de química fina.

En este contexto AVENTURE abordará tres desafíos principales: (i) un desafío global: las preocupaciones ambientales asociadas con la emisión de gases de efecto invernadero; (ii) una oportunidad industrial: abordará el problema de la sostenibilidad económica de la industria del biogás ofreciendo alternativas viables para la conversión de materia prima de bajo valor en bioquímicos de alto valor añadido a escala industrial; y (iii) un desafío a escala científica fundamental: se presentan dos propuestas, la intensificación de una ruta indirecta usando reactores de microcanales y una ruta directa llevada a cabo con catálisis por plasma. Para lograr estos ambiciosos objetivos, se diseñará una nueva generación de catalizadores avanzados multifuncionales capaces de proporcionar los productos específicos deseados con alta actividad, selectividad y durabilidad a largo plazo para garantizar el éxito de ADVENTURE.

ADVENTURE represents a new concept to convert biogas from organic waste into high-value industrial chemicals such as acetic acid (AA) in an environmentally and economically viable manner. AA is a precursor for many fine chemical compounds with a

wide range of applications including paints and coatings manufacturing, plastics and water-based adhesives production among many others, representing a very versatile platform molecule for the chemical industry. Traditionally, AA is produced at a commercial scale through an indirect route with a considerable global CO_2 footprint. In this regard, the main target of ADVENTURE is to re-design the AA production route introducing biogas as initial feedstock - a completely new approach that synergises CO_2 utilisation with fine chemicals synthesis.

In this context, ADVENTURE will tackle three main challenges: (i) A global challenge the environmental concerns associated with the emission of Greenhouse Gases (GHG); (ii) An industrial opportunity the problem of economic sustainability of the biogas industry by offering viable pathways for conversion of low-value feedstock into added-value biochemicals at industrial scale; and (iii) A fundamental scientific challenge the inexistence of AA production from biogas, by introducing two new revolutionary routes for AA production: an intensified indirect route using microchannel reactors and a direct route enabled by plasma catalysis. In order to accomplish these ambitious goals, a new generation of advanced multifunctional catalysts able to deliver the targeted products with high activity, selectivity and long-term durability will be designed to guarantee the success ADVENTURE.

Procesos Power-to-X para la Valorización de CO_2 en Reactores Catalíticos Estructurados (CO_2-Ptx)	**Power-to-X processes for CO_2 valorization in structured catalytic reactors (CO_2-Ptx)**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	RTI2018-096294-B-C33
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	260.150 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón y Francisca Romero Sarria
Componentes/Research group:	Luis F. Bobadilla Baladrón, María Isabel Domínguez Leal, Anna Dimitrova Penkova, Lola de las Aguas Azancot Luque, Marta Romero Espinosa, Juan Carlos Navarro De Miguel

RESUMEN / ABSTRACT

La tecnología Power-to-X (PTX) tiene como objetivo el almacenamiento de energía (preferentemente renovable) en productos químicos. Dichos productos pueden usarse luego como combustibles o como moléculas plataforma para otras síntesis químicas. Por tanto, esta tecnología juega un papel fundamental incrementando la fracción renovable del mix energético en línea con los objetivos de la UE para la reducción de emisiones de gases con efecto invernadero.

La producción de H_2 por electrólisis de agua para PTX es una tecnología madura disponible comercialmente que puede ser usada durante los periodos valle de consumo de energía renovables.

Por otro lado, el CO_2 es una fuente de carbono desaprovechada por lo que el uso combinado de H_2 renovable y CO_2 añade un importante plus al proceso PTX ya que el CO_2 asociado a las emisiones de gases de efecto invernadero es reintegrado contribuyendo a la economía circular y la descarbonización. Esta es la idea central que guía la presente propuesta. En particular, se trata de llevar a cabo las siguientes reacciones: hidrogenación de CO_2 a metano (también llamada metanación de CO_2 o reacción de Sabatier), la reacción reversa Water-Gas-Shift (activación del CO_2 y ajuste de la relación H_2/CO), síntesis de biocombustibles (dimetil éter y SFT) y producción de ácido acético. Estas reacciones ofrecen notables retos químico-ingenieriles en aspectos como: i) desarrollo de catalizadores multifuncionales adecuados; ii) gestión térmica de reacciones fuertemente exotérmicas; iii) control de la selectividad en reacciones múltiples en serie por acción conjunta de la temperatura, el tiempo de residencia, la formulación del catalizador y el diseño del reactor. El conocimiento adquirido por el consorcio en los proyectos previos (MAT2006-12386, ENE2009-14522, ENE2012-37431 y ENE2015-66975) nos permite proponer de una manera sólida y fundamentada el uso de catalizadores y reactores estructurados para superar estos retos.

Por tanto, el objetivo fundamental de esta propuesta es el estudio de sistemas catalíticos estructurados para reacciones relevantes del proceso Power-To-X con CO_2 (CO_2-PTX). Por otro lado, esperamos que la intensificación que aportan los sistemas estructurados sobre metales y los patrones de flujo desarrollados en sistemas como espumas de poro abierto jueguen papeles determinantes en el control de la temperatura y la selectividad de la reacción. En este sentido se estudiarán diferentes arquitecturas de sustrato junto a las variables principales como la densidad de celda o poro, el espesor de película catalítica o la aleación metálica del sustrato. Finalmente, para aproximarnos a la aplicación industrial de estos sistemas CO_2-PtX se considerará la valorización de CO_2 presente en corrientes diluidas como los gases de combustión. Esto supone nuevos retos debido a la baja concentración de CO_2, altos caudales volumétricos y efectos negativos de otros componentes (H_2O, SOx, etc.) en la actividad y estabilidad de los catalizadores. Se investigarán nuevas formulaciones de catalizadores junto con estrategias avanzadas de adsorción-desorción-reacción de CO_2 sobre los sustratos estructurados estudiados.

Globalmente, el proyecto se estructurará en forma matricial con tareas transversales de cada grupo basadas en sus líneas de especialización (modelado, estructuración y caracterización avanzada) junto a reacciones concretas de cada laboratorio que conformarán las tareas longitudinales del proyecto.

The main idea underlying the term "Power-to-X" is the storage of energy (preferably renewable) in the form of chemical products.

Thereafter, these products may be employed in energy-related applications or as platform chemicals. As a result, the Power-to-X (PTX) processes play a key role in increasing the penetration rate of renewables in the energy mix in line with European Union long-term objective of reducing greenhouse gas (GHG) emissions by 80-95 % by 2050 when compared to 1990 levels. Production of hydrogen by water electrolysis is a mature and commercially available technology that can be used during periods of low demand for renewable energy.

On the other hand, CO_2 is the only abundant carbon source within the EU and the combined use of renewable hydrogen and CO_2 remarkably results in additional benefits in the PTX concept since CO_2-associated GHG emissions is reintegrated in the value chain contributing to circular economy and decarbonization. This main idea drives CO_2-PTX proposal. Specifically, our proposal aims to carry out the following reactions in structured catalytic reactors: CO_2 hydrogenation to methane (also called methanation or Sabatier reaction), the reverse Water-Gas Shift reaction (CO_2 activation and adjustment of the H_2/CO ratio) and the direct synthesis of biofuels (dimethylether and FTS) and acetic acid. This set of reactions provides remarkable challenges in key catalytic engineering aspects such as: i) development of suitable multifunctional structured catalysts; ii) management of the thermal effect of highly exothermic reactions; iii) control of the selectivity of multiple reactions in series through the joint action of the reaction temperature, the residence time and suitable catalyst formulation and reactor configuration. The know-how acquired by the consortium during previous projects (MAT2006-12386, ENE2009- 14522, ENE2012-37431 and ENE2015-66975) allows us to propose the use of structured catalysts and reactors as a very convenient way of addressing that challenges. Heat and mass transfer rates intensification provided by metallic substrates-based structured systems as well as the flow patterns characteristic of open-cell foams are expected to play a determinant role in temperature and selectivity control. In this regard, several catalytic-wall reactor configurations as parallel-channels monoliths and open-cell foams will be considered, as well as other characteristics that directly affect the transport properties of the structured systems (monolith cell density, pore density of foams, metal alloy used as substrate and catalyst layer thickness).

To be closed to practical applications it will be also considered within the CO_2-PTX project the valorization of CO_2 present in dilute streams, typically flue gases. This entails additional challenges arising from the low concentration of CO_2, high volumetric flow rates and negative effects of other components (H_2O, SOx, etc.) on the catalytic activity and stability. Improved catalyst formulations as well as sorption-enhanced CO_2 conversion strategies in structured reactors will be investigated.

Overall, the project is organized as a series of transversal tasks for which each group contributes with his main field of specialization and vertical tasks associated to a more intense dedication of each group to one or more of the processes investigated.

Aprovechamiento de biomasa y producción sostenible de energía mediante (foto)catalizadores y reactores estructurados basados en materiales carbonosos	**Biomass valorization and sustainable energy production over (photo)catalysts and structured reactors based on carbonaceous materials**
Código/Code:	ENE2017-82451-C3-3-R
Periodo/Period:	01-01-2018 / 30-09-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Miguel Ángel Centeno Gallego y Svetlana Ivanova
Componentes/Research group:	Leidy Marcela Martínez Tejada, María Isabel Domínguez Leal, Regla Ayala Espinar, Carlos López Cartes

RESUMEN / ABSTRACT

El objetivo principal del presente proyecto coordinado entre la U. de Zaragoza, el ICMS y la U. de Cádiz, es el desarrollo de catalizadores multifuncionales y estructurados basados en materiales catalíticos carbonosos, tanto de carácter biomórfico, como grafénico-grafítico. Estos materiales catalíticos han de ser activos, selectivos y estables en reacciones directamente relacionadas con el aprovechamiento de la biomasa lignocelulósica (producción de 5-HMF, ácido levulínico, FDCA, o γ-valerolactona) y la producción sostenible de energía (producción de H_2), así como la valorización química y fotoquímica de CO_2 (hidrogenación de CO_2, descomposición de biogás, foto-reformado de bio-alcoholes), usando H_2 de origen renovable ("water splitting"). Este proyecto trata de mejorar procesos actualmente implementados que están relacionados con la producción de energía, y otros más novedosos, como el aprovechamiento de la luz solar, que sin lugar a dudas están llamados a tener un papel importante en este campo. De hecho, la utilización de la energía solar haría más viable energéticamente, por ejemplo, la reacción de metanación de CO_2 al usar H_2 de origen (foto)renovable producido por "water splitting". Se busca también la generación de productos de alto valor añadido por procesos de biorefinería, que sustituyan los obtenidos actualmente a partir de fuentes fósiles. Se pretende conseguir un conjunto de sólidos carbonosos con propiedades estructurales (porosidad jerárquica meso/micro), hidrofilicidad-hidrofobicidad, funcionalidades químicas, composición superficial etc. diseñados ad hoc para cada una de las reacciones consideradas por los distintos subproyectos, incluyendo la implementación de procesos en continuo mediante la

utilización de reactores estructurados a partir de los catalizadores más eficientes. El desarrollo y utilización de sistemas catalíticos estructurados aumenta la viabilidad e intensificación de los procesos y por tanto la eficiencia energética y medioambiental. La complementariedad de los tres grupos proponentes abre la posibilidad de abordar en un solo proyecto todos estos objetivos, permitiendo aplicar distintas metodologías emergentes para la síntesis de nuevos materiales carbonosos, como son la mineralización biomórfica, la expansión/funcionalización de compuestos intercalados de grafito, grafitos especiales (e.g. "graphite nanolayers" o "nanoflakes"), uso de plantillas inorgánicas para generación de carbones mesoporosos, su funcionalización avanzada y su aplicación en procesos de alto impacto en el área de la energía, tecnología química y tecnologías ambientales.

The main goal of ENERCARB, project coordinated among the U. of Zaragoza, the ICMS and the U. of Cádiz, is the development of multifunctional and structured catalysts based on carbonaceous catalytic materials of biomorphic and/or graphenic-graphitic character. These materials must be active, selective and stable in catalytic reactions related to i) the production and use of chemicals derived from lignocellulosic biomass, i.e. 5-HMF, levulinic acid, FDCA and g-valerolactone; ii) to sustainable energy vector production (H_2), and iii) to chemical and photochemical utilization of CO_2 (CO_2 hydrogenation), biogas decomposition, photo-reforming of bio-alcohols) using H_2 of renewable origin ("water splitting"). This project tries to improve currently implemented processes for energy production, and to propose other more innovative processes, such as use of sunlight, undoubtedly called to play an important role in this field. In fact, the use of solar energy would make more energy-efficient, the CO_2 methanation reaction by using H_2 of (photo)renewable origin produced by "water splitting". ENERCARB also intends to generate high added value products by bio-refinery processes, as alternative to currently obtained chemicals from fossil sources. A set of carbonaceous solids with tuned structural properties (meso/micro hierarchical porosity), hydrophilicity-hydrophobicity, chemical functionalities, surface composition, etc., will be designed ad hoc for each of the reactions considered by the different subprojects. The implementation of continuous processes through the use of structured reactors is the next logical step to increase the efficiency of the proposed processes. The development and use of structured catalytic systems increases the viability and intensifies the processes, and therefore leads to higher energy and environmental efficiency. The complimentary nature of the three participating groups opens the possibility of addressing all these objectives in one single project. It will allow the application of different emerging methodologies for the synthesis of new carbonaceous materials, such as biomorphic mineralization, the expansion-functionalization of graphite intercalation compounds, special graphites (e.g. graphite nanolayers or nanoflakes), use of inorganic templates for the generation of mesoporous carbons, and also its advanced functionalization and its application in processes of high impact in the area of energy, chemical and environmental technologies.

Valorización de CO_2 mediante procesos catalíticos y termofotocatalíticos: reducción de emisiones y obtención de metano y otros hidrocarburos ligeros	**CO_2 recovery though catalytic and thermophotocatalytic processes: reduction of emissions and obtaining methane and other light hydrocarbons (CO2MET)**
MINISTERIO DE CIENCIA E INNOVACIÓN AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PID2020-119946RB-I00
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Alfonso Caballero Martínez y Gerardo Colón Ibáñez
Componentes/Research group:	Juan Pedro Holgado Vázquez y Rosa María Pereñíguez Rodríguez

RESUMEN / ABSTRACT

En este proyecto se llevarán a cabo diversos estudios y desarrollos relacionados con la reacción de hidrogenación de CO_2 para la producción de Gas Natural Sintético (GNS) e hidrocarburos ligeros. Así, la metanación y las denominadas reacciones modificadas de Fischer-Tropsch a olefinas (FTO) se están convirtiendo en procesos muy interesantes desde el punto de vista económico, energético y medioambiental. Por otra parte, el uso de hidrógeno verde como agente reductor, obtenido a su vez a partir de fuentes renovables, representa, además de la reducción de las emisiones de gases de efecto invernadero, una forma de almacenar la energía procedente de fuentes renovables, muchas de las cuales son intermitentes y, por tanto, difíciles de ajustar a las necesidades de consumo.

Con todo ello, este proyecto persigue un enfoque multicatalítico que comprende la termocatálisis y la fotocatálisis térmica con el fin de conseguir altos rendimientos, alta sostenibilidad y con los menores costes de producción, orientados en todo caso a una aplicación industrial final. Por otro lado, el desarrollo y optimización de los materiales catalíticos, considerando nuevos sistemas catalíticos heterogéneos basados en Ni, Fe, Co, Ru, Au, Pd entre otros metales, que han mostrado un gran potencial para estas reacciones de hidrogenación en los últimos años. En cuanto a los materiales catalíticos, se seleccionarán soportes micro y mesoporosos de composición variable (zeolitas, SBA-15, etc.), así como otros basados en óxidos y perovskitas ABO_3. Para ello se utilizarán una serie de técnicas de preparación recientemente descritas (cristalización por microondas, proceso de autocombustión, mesoestructuración por nanocasting y porosidad jerárquica) que permiten obtener sistemas de alta superficie específica y nanoestructura

controlada. La combinación de diferentes elementos en las posiciones A y B de la estructura de la perovskita, que actúan tanto como promotores de sistemas catalíticos como precursores de aleaciones metálicas en sistemas catalíticos reducidos, permitirá obtener materiales con propiedades catalíticas sintonizables, muy variadas y versátiles.

This project will carry out various studies and developments related to the CO_2 hydrogenation reaction for Synthetic Natural Gas (SNG) and light hydrocarbons production. Thus, methanation and the so-called modified Fischer-Tropsch to olefins (FTO) reactions are becoming very interesting processes under an economic, energy and environmental point of view. Furthermore, the use of green hydrogen as a reducing agent, obtained in turn from renewable sources, represents, in addition to the reduction of greenhouse gas emissions, a way of storing energy from renewable sources, many of which are intermittent and therefore difficult to match with consumption needs.

With all this in mind, this project pursues a multi-catalytic approach comprising thermal-catalysis and thermal photocatalysis in order to achieve high performances, high sustainability and with the lowest costs of production, oriented in all case to a final industrial application. On the other hand, development and optimization of the catalytic materials, considering new heterogeneous catalytic systems based on Ni, Fe, Co, Ru, Au, Pd among other metals, which have shown great potential for this hydrogenation reactions in recent years. Regarding to the catalytic materials, micro and mesoporous supports of variable composition (zeolites, SBA-15, etc.) will be selected, as well as others based on oxides and ABO_3 perovskites. For this purpose, a series of recently described preparation techniques will be used (microwave crystallization, autocombustion process, mesostructuring by nanocasting and hierarchical porosity) that allow to obtain high specific surface systems and controlled nanostructure. The combination of different elements in positions A and B of the perovskite structure, which act both as promoters of catalytic systems and as precursors of metal alloys in reduced catalytic systems, will make it possible to obtain materials with tunable, highly varied and versatile catalytic properties.

Valorización de CO_2: obtención de hidrocarburos mediante procesos catalíticos de hidrogenación	**CO_2 valorization: obtaining hydrocarbons through catalytic hydrogenation processes**
Código/Code:	US-1263455
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	80.000 €
Investigador responsable/Research head:	Alfonso Caballero Martínez y Juan Pedro Holgado Vázquez
Componentes/Research group:	Gerardo Colón Ibáñez, Rosa Pereñíguez Rodríguez, Andrew M. Beale (UCL), Ángeles M. López Martín, Francisco Jesús Platero Moreno

RESUMEN / ABSTRACT

En el presente proyecto se llevarán a cabo diversos estudios y desarrollos relacionados con la reducción de CO_2 a productos de alto valor añadido, como metano, olefinas ligeras, gasolinas y otros hidrocarburos funcionalizados, de gran interés económico, energético y medioambiental. El uso de hidrógeno como agente reductor, obtenido éste a su vez de fuentes renovables supone, además de la reducción de las emisiones de gases de efecto invernadero, una vía para el almacenamiento de la energía procedente de fuentes renovables, muchas de ellas de carácter intermitente y por tanto difícilmente acoplable a las necesidades de consumo.

Con todo ello en este proyecto se propone el desarrollo de nuevos sistemas catalíticos heterogéneos basados en Ni, Fe, Co, Ru e In, entre otros metales, los cuales han mostrado en los últimos años un gran potencial para esta reacción de hidrogenación. Dado el carácter bifuncional de los mecanismos de reacción involucrados en estas reacciones, se seleccionarán soportes micro y mesoporosos de composición variable (zeolitas, SBA-15, etc.), así como otros basados en estructura perovskita ABO_3. Para ello se emplearán una serie de técnicas de preparación recientemente descritas (Cristalización por Microondas, Proceso de Autocombustión, Mesoestructuración por Nanocasting y Porosidad Jerarquizada) que permiten obtener sistemas de alta superficie específica y nanoestructura controlada. La combinación de diferentes elementos en las posiciones A y B de la estructura perovskita, que actúen tanto como agentes promotores de los sistemas catalíticos como de precursores de aleaciones metálicas en los sistemas catalíticos reducidos, permitirá obtener materiales con propiedades catalíticas modulables, muy variadas y versátiles.

This project will carry out several studies and developments related to the reduction of CO_2 to valuable products, such as methane, light olefins, gasolines and other functionalized hydrocarbons, of economic, energetic and environmental interest. The use of hydrogen as a reducing agent, obtained from renewable sources, in addition to the reduction of greenhouse gas emissions, is a way to store energy from renewable sources, many of which are intermittent and therefore difficult to match with consumption needs.

Therefore, this project proposes the development of new heterogeneous catalytic systems based on Ni, Fe, Co, Ru and In, among other metals, which have shown in recent years a great potential for this hydrogenation reaction. Given the bifunctional character of the reaction mechanisms involved in these reactions, micro and mesoporous supports of variable composition (zeolites, SBA-15, etc.) will be selected, as well as others based on ABO_3 perovskite structure. For this purpose, a series of recently described preparation techniques (Microwave Crystallization, Self-Combustion Process, Mesostructuring by Nanocasting and Hierarchical Porosity) will be used to obtain systems with high specific surface area and controlled nanostructure. The combination of different elements in the A and B positions of the perovskite structure, acting both as promoting agents of the catalytic systems and as precursors of metallic alloys in the reduced catalytic systems, will allow obtaining materials with modular, varied and versatile catalytic properties.

Diseño de fotocatalizadores altamente eficientes mediante control de la nanoescala para la producción de H_2 NanoLight2H2	**Design of highly efficient photocatalysts by nanoscale control for H_2 production NanoLight2H2**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P20-00156 - PAIDI 2020
Periodo/Period:	05-10-2021 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	85.000 €
Investigador responsable/Research head:	Gerardo Colón Ibáñez
Componentes/Research group:	Alfonso Caballero Martínez, Rosa Pereñiguez Rodríguez, Juan Pedro Holgado Vázquez

RESUMEN / ABSTRACT

El objetivo principal de este proyecto es el desarrollo de catalizadores heteroestructurados basados en óxidos semiconductores altamente eficientes (Nb_2O_5, WO_3, TiO_2 y Fe_2O_3) y g-C_3N_4, con control a nivel de la nanoescala, y potencial aplicación en la reacción de fotoreformado de alcoholes para la producción de H_2. Así mismo, se pretende estudiar la optimización del proceso catalítico mediante una aproximación multi-catalítica, mediante la combinación de termocatálisis y fotocatálisis. La producción fotocatalítica de H_2 una reacción de gran interés desde el punto de vista energético mediante el uso de una tecnología limpia y sostenible como la fotocatálisis. En este proyecto se pretende el desarrollo de sistemas altamente eficientes para la producción de hidrógeno. Se prestará especial atención al diseño de heteroestructuras que permitan la optimización del proceso fotoinducido. De igual modo se incidirá en el uso de co-catalizadores alternativos a los tradicionales metales nobles; sistemas basados en metales de transición (Cu, Co, Ni), así como estructuras bimetálicas con metales nobles formando aleaciones o core-shell. Junto al proceso fotocatalítico en fase líquida, se estudiará la viabilidad de un proceso de fotoreformado en fase gas, basándonos en recientes estudios que ponen de manifiesto el efecto sinérgico de una aproximación foto-termo catalítica en estos procesos. De esta forma esta propuesta pretende abordar de forma ambiciosa el aumento de la eficiencia del proceso fotocatalítico a fin de poder plantear esta tecnología a mayor escala. En este sentido, además de los estudios de optimización de los catalizadores y del proceso fotocatalítico, se afrontará como algo primordial su escalado a planta solar piloto.

The main objective of this project is the development of heterostructured catalysts based on highly efficient semiconducting oxides (Nb_2O_5, WO_3, TiO_2 and Fe_2O_3) and g-C_3N_4, with control at the nanoscale level, and potential application in the photoreforming reaction of alcohols for the production of H_2. Furthermore, the aim of this project is to study the optimisation of the catalytic process by means of a multi-catalytic approach, combining thermocatalysis and photocatalysis. The photocatalytic production of H_2 is a reaction of great interest from an energetic point of view through the use of a clean and sustainable technology such as photocatalysis. We will try to develop highly efficient systems for hydrogen production. Special attention will be paid to the design of heterostructures that allow the optimisation of the photoinduced process. Likewise, emphasis will be placed on the use of alternative co-catalysts to the traditional noble metals; systems based on transition metals (Cu, Co, Ni), as well as bimetallic structures with noble metals formed into alloys or core-shell. Together with the liquid phase photocatalytic process, the feasibility of a gas phase photoreforming process will be studied, based on recent studies that show the synergistic effect of a photo-thermo-catalytic approach in these processes. In this way, this proposal aims to ambitiously address the increase in efficiency of the photocatalytic process in order to be able to consider this technology on a larger scale. In this sense, in addition to the optimisation studies of the catalysts and the photocatalytic process, its scaling up to a pilot solar plant will be considered as essential.

Diseño de catalizadores avanzados para procesos de HDO: una apuesta revolucionaria para la conversión de biomasa: CLEVER-BIO	**Design of advanced CataLyst for H_2-free hydrodeoxygenation - a rEVolutionary approach Enabling pRactical BIOmass upgrading: CLEVER-BIO**
Junta de Andalucía	Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa
Código/Code:	P20_00667
Periodo/Period:	05-10-2021 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	49.612 €
Investigador responsable/Research head:	Tomás Ramírez Reina
Componentes/Research group:	Luis Francisco Bobadilla Baladrón, José Antonio Odriozola Gordón, Laura Pastor Pérez, Anna Dimitrova Penkova

RESUMEN / ABSTRACT

CLEVER-BIO propone un concepto revolucionario para la producción de biocombustibles limitando la emisión de gases de efecto invernadero sembrando las bases de una tecnología verde: conversión de residuos a combustibles y productos de alto valor. La idea central de CLEVER-BIO es el desarrollo de catalizadores avanzados para llevar a cabo la reacción de HDO de bio-aceites derivados de lignina. El proyecto se llevará a cabo en 24 meses y comprende un programa intenso de investigación multidisciplinar con fuerte participación de instituciones internacionales.

CLEVER-BIO proposes a revolutionary approach to synergise bio-oil upgrading and Green House Gases (GHG) emissions abatement, setting the grounds for a sustainable chemical technology: waste to fuels/chemicals. We aim to develop novel biomass-derived routes to produce deoxygenated aromatic hydrocarbons – highly important chemical compounds in the biofuels and biochemical industries – from lignin-derived bio-oil via designing of advanced catalysts for the H_2-free hydrodeoxygenation (HDO) process. The urgent problem of global warming and the need to decarbonise the transportation and chemical industry in a circular economy context place CLEVER-BIO in a privileged position to become a pioneering approach to contribute towards the development of sustainable societies. CLEVER-BIO will be delivered in 24 months under a comprehensive research program with strong international cooperation and social-scientific impact.

Integración de Energía y Gasificación para procesos sostenibles (GENIUS)	**Gasification and ENergy Integration for User Sustainability (GENIUS)**
Junta de Andalucía	Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa
Código/Code:	P20_00594
Periodo/Period:	05-10-2021 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	49.500 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	Luis F. Bobadilla Baladrón, Laura Pastor Pérez, Anna Dimitrova Penkova, Tomás Ramírez Reina

RESUMEN / ABSTRACT

GENIUS representa una propuesta innovadora para la conversión de bio-residuos en vectores energéticos sostenibles. El proyecto propone la combinación de tecnologías maduras como la gasificación y reformado acuoso para aportar soluciones catalíticas al proceso de conversión de bioresiduos. GENIUS desarrollará reactores de microcanales que permiten el diseño de plantas compactas para el procesado de residuos lo que facilita su implementación en aplicaciones deslocalizadas como por ejemplo explotaciones agrícolas donde los residuos pueden convertirse en productos de valor añadido.

GENIUS proposes an innovative approach to transform biogenic residues into a valuable bioenergy carrier. The proposal is based on the combination of modified mature technologies, e.g. gasification, with first-time approached solutions as the continuous aqueous-phase reforming of tars that compromises downstream processes, usually the bottlenecks for upgrading catalytic processes.

The combination of microchannel reactor technologies with state-of-the-art multifunctional catalysts will provide a path to increase the wealth of rural communities on proposing a decentralized approach allowing territory-based solutions for agricultural residues or marginal lands production.

GENIUS focus in the system perspective demanded in HORIZON EUROPE keeping in mind the Objectives for Sustainable Development and industry decarbonisation. GENIUS will be delivered in 24 months under a comprehensive research program with strong international cooperation and social-scientific impact.

Ácido fórmico como vector energético: viabilidad de los ciclos de carga y descarga de hidrógeno	**Formic acid as energy vector: feasability of hydrogen charge/discharge cycles**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P18-RT-3405
Periodo/Period:	01-01-2020 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	102.268 €
Investigador responsable/Research head:	Svetlana Ivanova y Miguel Angel Centeno
Componentes/Research group:	María Isabel Domínguez Leal, Leidy Marcela Martínez Tejada

RESUMEN / ABSTRACT

El presente proyecto se encuadra en la actual tendencia a nivel mundial de búsqueda de tecnologías para la captura y uso del dióxido de carbono (Carbon dioxide Capture and Utilization CCU). Su interés radica en la utilización directa del CO_2 atmosférico para almacenar hidrógeno verde, esto es, producido con la ayuda de energías renovables, en forma de ácido fórmico, usado como vector energético. Desde el punto de vista medioambiental, el desarrollo de esta tecnología permitiría preservar la huella de CO_2 durante el ciclo completo de generación, almacenamiento y liberación de energía, sin generar más gases de efecto invernadero. La posibilidad de almacenar hidrógeno de esta forma facilitaría su transporte y su uso en aplicaciones deslocalizadas diversas, tanto móviles como estacionarias. Indirectamente, esta tecnología racionalizaría el almacenamiento de las energías renovables, haciéndolas independientes de las condiciones climáticas. Este proyecto pretende estudiar la viabilidad de la tecnología basándose en el desarrollo de un único catalizador, estable y selectivo para los ciclos de carga y descarga de hidrógeno (CO_2/HCOOH).

This project is part of the current trend for future technologies of Carbon dioxide Capture and Utilization (CCU). His interest lies in a direct use of atmospheric CO_2 to store green hydrogen (produced with the help of renewable energies) as formic acid directly used as an energy vector. From an environmental point of view, the development of this technology would make possible the preservation of the CO_2 footprint during the complete cycle of energy generation, storage and release, without generating more greenhouse gases. The possibility of storing hydrogen in this way would facilitate its transport and its use in diverse applications, both mobile and stationary. Indirectly, this technology would rationalize the storage of renewable energies, making them independent of climatic conditions. This project aims to study the feasibility of the technology based on the development of one unique stable and selective catalyst for both, hydrogen charge and discharge cycles (CO_2 / HCOOH).

CO_2 como fuente de carbono para la producción de compuestos químicos de alto valor añadido

Junta de Andalucía

Unión Europea
Fondo Europeo de Desarrollo Regional

Andalucía se mueve con Europa

Código/Code:	US-1263288
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón y Svetlana Ivanova
Componentes/Research group:	Anna Dimitrova Penkova

RESUMEN / ABSTRACT

El principal reto científico de este proyecto es el diseño de un dispositivo catalítico activo y selectivo en la reducción catalítica de CO_2 a CO. El proyecto pretende resolver dos problemas interrelacionados, el diseño del catalizador, que pasa por una comprensión a nivel molecular de la reacción estudiada, y el diseño de un reactor de microcanales que permita realizar la reacción en régimen isotermo y tiempos de residencia muy cortos. Para ello se propone la síntesis de catalizadores constituidos por metales nobles o de transición soportados en óxidos reducibles para llevar a cabo el proceso catalítico analizando los factores determinantes de la reacción: tamaño de partícula de la fase metálica, reducibilidad del soporte, interacciones metal-soporte y resistencia de la desactivación. Las características de la reacción exigen el desarrollo de reactores de microcanales que permitan disminuir las pérdidas de carga y los tiempos de residencia manteniéndose isotermos. El estudio, por tanto, busca diseñar un dispositivo catalítico activo, selectivo y estable que trabaje en régimen isotermo y permita tiempos de residencia inferiores a 100 ms.

The main scientific challenge of this project is active and selective catalytic device design for the catalytic reduction of CO_2 to CO. The project aims to solve two related problems, the design of the catalyst, which requires a deep understanding at the molecular level, and the design of a microchannel reactor that allows reaction in isothermal regime and with very short residence times. For this, the use of noble and/or transition metals supported on reducible oxides catalysts are proposed in this project to

carry out the indicated process analyzing some reaction determining factors: metal phase particle size, support reducibility, metal-support interactions, and deactivation behavior. The characteristics of the reaction require the development of microchannel reactors that allow the reduction of the charge loss and residence time while maintaining the reaction isothermal. This project, therefore, is dedicated to design an active, selective and stable catalytic device that works in an isothermal regime and allows residence times of less than 100 ms.

OTROS PROYECTOS / OTHER PROJECTS

Kit divulgativo: preparación y demostración de superficies autolimpiables

Código/Code:	FGCCLG-2021-0026
Periodo/Period:	01-01-2021 / 31-12-2021
Organismo Financiador/Financial source:	Fundación General CSIC
Investigador responsable/Research head:	María del Carmen Hidalgo López

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Desarrollo de catalizadores para la producción de alcoholes superiores

Periodo/Period:	01-05-2021 / 30-11-2021
Organismo Financiador/Financial source:	ASOCIACIÓN DE INVESTIGACIÓN Y COOPERACIÓN INDUSTRIAL DE ANDALUCÍA
Investigador responsable/Research head:	Tomás Ramírez Reina

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Biorefinería: Aprovechamiento de biomasa residual para la obtención de biocombustibles y compuestos de alto valor añadido (EQC2019-005458-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 196.856,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Química de Superficies y Catálisis"

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Functionalized biochars as supports for Pd/C catalysts for efficient hydrogen production from formic acid

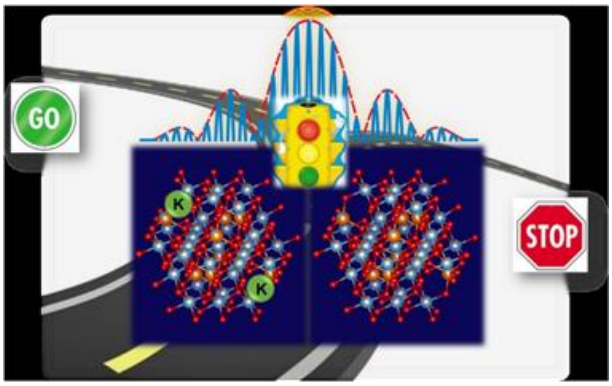
Santos, JL; Megías-Sayago, C; Ivanova, S; Centeno, MA; Odriozola, JA
Applied Catalysis B-Environmental, **282** (2021) 119615
Marzo 2021 | **DOI:** 10.1016/j.apcatb.2020.119615

Biomass waste product was used to generate biochars as catalytic supports for selective hydrogen production from formic acid. The supports were obtained after pyrolysis in CO_2 atmosphere of non-pretreated and che-mically $ZnCl_2$ activated raw materials (vine shoot and crystalline cellulose). The support series includes materials with different textural properties and surface chemistry. The support nature and especially textural properties firstly affects significantly Pd size and dispersion and its interaction with the support and secondly influence in a great extent the catalytic behavior of the final material. The presence of prevailing mesoporous character appeared to be the most important parameter influencing formic acid dehydrogenation and overall hydrogen production.

IR spectroscopic insights into the coking-resistance effect of potassium on nickel-based catalyst during dry reforming of methane

Azancot, L; Bobadilla, LF; Centeno, MA; Odriozola, JA
Applied Catalysis B-Environmental, **285** (2021) 119822
Mayo 2021 | **DOI:** 10.1016/j.apcatb.2020.119822

Dry reforming of methane (DRM) is an effective catalytic route for transforming CO_2 and CH_4 into valuable syngas and thus potentially attractive for mitigating the emission of environmental harmful gases. Therefore, it is crucial to develop rationally Ni-based catalysts highly resistant to coking and sintering. In this scenario, the addition of small amounts of potassium to nickel catalyst increases their resistance to coking during dry reforming of methane. Nonetheless, the specific role of potassium in these catalysts not have been fully understood and there are still important discrepancies between the different reported studies. This work provides a new approach on the anticoking nature of a K-promoted Ni catalyst by means of a combined IR spectroscopic study of in situ characterization by CO adsorption under static conditions and operando DRIFTS measurements under dynamic conditions of DRM reaction. The involved surface species formed during the reaction were elucidated by transient and steady-state operando DRIFTS studies. It was revealed that the existence of Ni-K interfacial sites favours the gasification of carbonaceous deposits towards reverse Boudouard reaction and reduces the sticking probability of CO_2 dissociative adsorption. Moreover, the presence of strongly Mg-O-K basic sites leads to the formation of carbonate intermediates that are subsequently reduced into CO gaseous towards the associative mechanism by RWGS reaction. These results provide a fundamental understanding of the relevant anticoking effect of potassium on Ni-based catalysts.



Dehydration of glucose to 5-Hydroxymethlyfurfural on bifunctional carbon catalysts

Bounoukta, CE; Megías-Sayago, C; Ammari, F; Ivanova, S; Monzón, A; Centeno, MA; Odriozola, JA

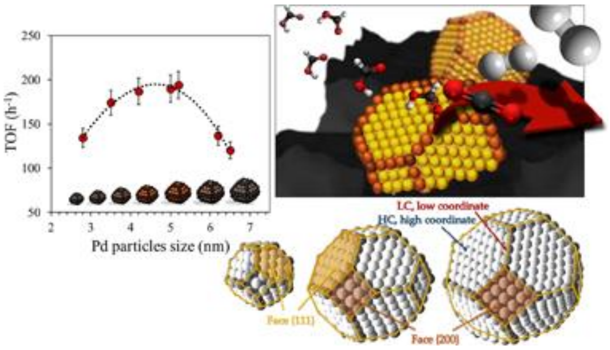
Applied Catalysis B-Environmental, **286** (2021) 119938

Junio 2021 | **DOI:** 10.1016/j.apcatb.2021.119938

The proposed study tries to reply on one important question concerning glucose dehydration: What is the role of bare or tandem Lewis/Bronsted acid sites in the reaction and which are better? A series of mono and bifunctional catalyst are designed and screened for the glucose dehydration reaction. The results clearly reveal that catalyst activity is a function of catalyst composition. The presence of Lewis sites the reaction toward first step isomerization, while the Brunsted acid dehydrate directly glucose to HMF via levoglucosane intermediate. This study proposed also a kinetic modelling of the included reactions and their contrast with the empirical observations.

Structure-sensitivity of formic acid dehydrogenation reaction over additive-free Pd NPs supported on activated carbon

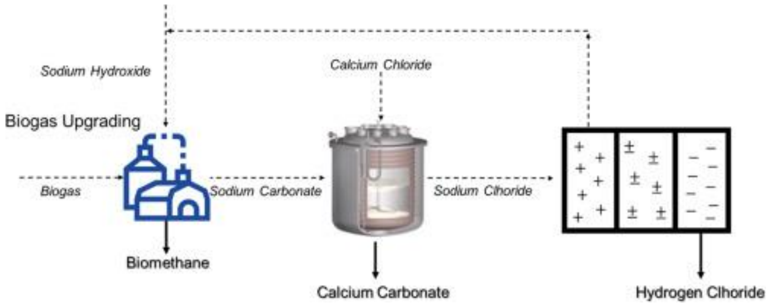
Santos, JL; Megías-Sayago, C; Ivanova, S; Centeno, MA; Odriozola, JA
Chemical Engineering Journal, **420** (2021) 127641
Septiembre 2021 | **DOI:** 10.1016/j.cej.2020.127641



In this study the size-activity dependence of palladium based catalysts in formic acid dehydrogenation reaction was investigated and evaluated. A wide range of particle sizes was considered and the catalyst series were prepared upon variation of some synthetic parameters, precursor and solvent nature in particular. Synthesis method variations affect significantly Pd particle size and results in diverse activity toward hydrogen production. An optimal size was observed and explained by the diverse proportion of low and high coordinated Pd states available for different samples within the series. The evaluation of particles much bigger than 6 nm changes importantly the fraction of high and low coordination atoms and allows the clear confirmation of the importance of the presence of low coordination atoms on the surface of catalyst.

Synergizing carbon capture and utilization in a biogas upgrading plant based on calcium chloride: Scaling-up and profitability analysis

Baena-Moreno, FM; Reina, TR; Rodríguez-Galán, M; Navarrete, B; Vilches, LF
Science of The Total Environment, **758** (2021) 143645
Marzo 2021 | **DOI:** 10.1016/j.scitotenv.2020.143645

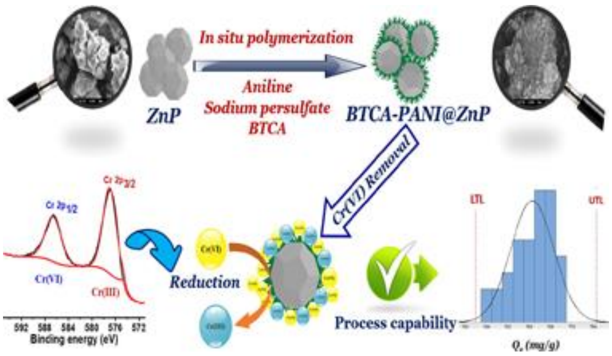


Herein we analyze the profitability of a novel regenerative process to synergize biogas upgrading and carbon dioxide utilization. Our proposal is a promising alternative which allows to obtain calcium carbonate as added value product while going beyond traditional biogas upgrading methods with high thermal energy consumption. Recently we have demonstrated the experimental viability of this route. In this work, both the scale-up and the profitability of the process are presented. Furthermore, we analyze three representative scenarios to undertake a techno-economic study of the proposed circular economy process. The scale-up results demonstrate the technical viability of our proposal. The precipitation efficiency and the product quality are still remarkable with the increase of the reactor size. The techno-economic analysis reveals that the implementation of this circular economy strategy is unprofitable without subsidies. Nonetheless, the results are somehow encouraging as the subsides needed to reach profitability are lower than in other biogas upgrading and carbon dioxide utilization proposals. Indeed, for the best-case scenario, a feed-in tariff incentive of 4.3 (sic)/MWh makes the approach profitable. A sensitivity study through tornado analysis is also presented, revealing the importance of reducing bipolar membrane electrodialysis energy consumption. Overall our study envisages the big challenge that the EU faces during the forthcoming years. The evolution towards bio-based and circular economies requires the availability of economic resources and progress on engineering technologies.

Facile synthesis and characterization of a novel 1,2,4,5-benzene tetracarboxylic acid doped polyaniline@zinc phosphate nanocomposite for highly efficient removal of hazardous hexavalent chromium ions from water

Hsini, A; Naciri, Y; Benafqir, M; Ajmal, Z; Aarab, N; Laabd, M; Navío, JA; Puga, F; Boukherroub, R; Bakiz, B; Albourine, A

Journal of Colloid and Interface Science **585** (2021) 560-573
Marzo, 2021 | **DOI:** 10.1016/j.jcis.2020.10.036

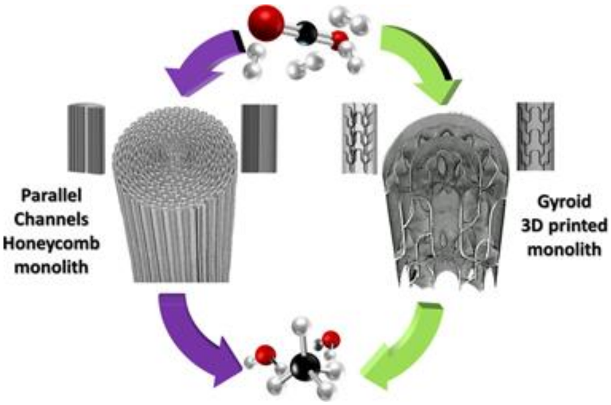


The present study describes the preparation of a novel 1,2,4,5-benzene tetracarboxylic acid doped polyaniline@zinc phosphate (BTCA-PANI@ZnP) nanocomposite via a facile two-step procedure. Thereafter, the as-prepared composite material adsorption characteristics for Cr(VI) ions removal were evaluated under batch adsorption. Kinetic approach studies for Cr(VI) removal, clearly demonstrated that the results of the adsorption process followed the pseudo second order and Langmuir models. The thermodynamic study indicated a spontaneous and endothermic process. Furthermore, higher monolayer adsorption was determined to be 933.88 mg g^{-1}. In addition, the capability study regarding Cr(VI) ions adsorption over BTCA-PANI@ZnP nanocomposite clearly revealed that our method is suitable for large scale application. X-ray photoelectron spectroscopy (XPS) analysis confirmed Cr(VI) adsorption on the BTCA-PANI@ZnP surface, followed by its subsequent reduction to Cr(III). Thus, the occurrence of external mass transfer, electrostatic attraction and reduction phenomenon were considered as main mechanistic pathways of Cr(VI) ions removal. The superior adsorption performance of the material, the multidimensional characteristics of the surface and the involvement of multiple removal mechanisms clearly demonstrated the potential applicability of the BTCA-PANI@ZnP material as an effective alternative for the removal of Cr(VI) ions from wastewater.

Stepping toward Efficient Microreactors for CO_2 Methanation: 3D-Printed Gyroid Geometry

Baena-Moreno, FM; González-Castaño, M; de Miguel, JCN; Miah, KUM; Ossenbrink, R; Odriozola, J.A.

ACS Sustainable Chemistry & Engineering, **9** (2021) 8198-8206
Junio 2021 | **DOI:** 10.1021/acssuschemeng.1c01980

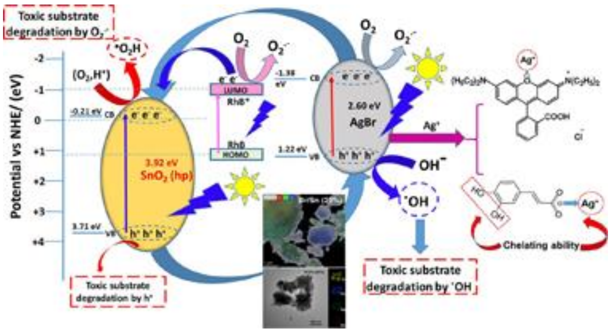


This work presents a comparative study toward the development of efficient microreactors based on three-dimensional (3D)-printed structures. Thus, the study evaluates the influence of the metal substrate geometry on the performance of structured catalysts for the CO_2 methanation reaction. For this purpose, the 0.5%Ru-15%Ni/$MgAl_2O_4$ catalyst is washcoated over two different micromonolithic metal substrates: a conventional parallel channel honeycomb structure and a novel 3D-printed structure with a complex gyroid geometry. The effect of metal substrate geometry is analyzed for several CO_2 sources including ideal flue gas atmospheres and the presence of residual CH_4 and CO in the flue gas, as well as simulated biogas sources. The advantages of the gyroid 3D complex geometries over the honeycomb structures are shown for all evaluated conditions, providing in the best-case scenario a 14% improvement in CO_2 conversion. Moreover, this contribution shows that systematically tailoring geometrical features of structured catalysts becomes an effective strategy to achieve improved catalyst performances independent of the flue gas composition. By enhancing the transport processes and the gas-catalyst interactions, the employed gyroid 3D metal substrates enable boosted CO_2 conversions and greater CH_4 selectivity within diffusion-controlled regimes.

Enhanced UV and visible light photocatalytic properties of synthesized AgBr/SnO_2 composites

Puga, F; Navío, JA; Hidalgo, MC

Separation and Purification Tecnology, **257** (2021) 117948
Febrero 2021 | **DOI:** 10.1016/j.seppur.2020.117948



Composites ($AgBr/SnO_2$) comprised of AgBr and SnO_2 with different molar % of bare SnO_2, have been synthesized by simple precipitation methods; the bare SnO_2 used, was synthesized by hydrothermal procedure. Samples have been characterized by X-ray diffraction (XRD), N_2-adsorption, UV–vis diffuse reflectance spectroscopy (DRS), scanning electron microscopy (SEM), Transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). Photocatalytic activity of the as-prepared photocatalysts was evaluated through photocatalytic degradation of rhodamine B (RhB) and caffeic acid (CAFA) under UV and Visible illumination. In photocatalytic degradation studies, for both substrates, conversion rates of around 95% were found in 45 min of both UV-illumination and 85% under visible lighting. These conversion rates were superior than the conversion rates of pure parental components, AgBr and SnO_2 under the same experimental conditions. At least, for RhB no loss of photocatalytic activity has been observed after five recycles although the mineralization degree progressively diminished along the recycles. The enhanced photocatalytic degradation of $AgBr/SnO_2$ compounds was attributed, in part, to a synergistic increase in adsorption viability, as well as to the effective separation of photoinduced load carriers that resulted from the formation of a heterojunction according to the type II junction. Radical scavengers' experiments indicated that active oxidant species as $O_2^{\cdot -}$, •OH and h^+ all are involved in this photocatalytic system, although it seems that $O_2^{\cdot -}$ played the major role in the photocatalytic degrading of RhB by $AgBr/SnO_2$ composites. In summary, coupling AgBr with SnO_2 remarkably improves the photocatalytic activity under both UV and visible-illumination with respect to the parental components. These features open the route to future applications of this material in the field of environmental remediation.

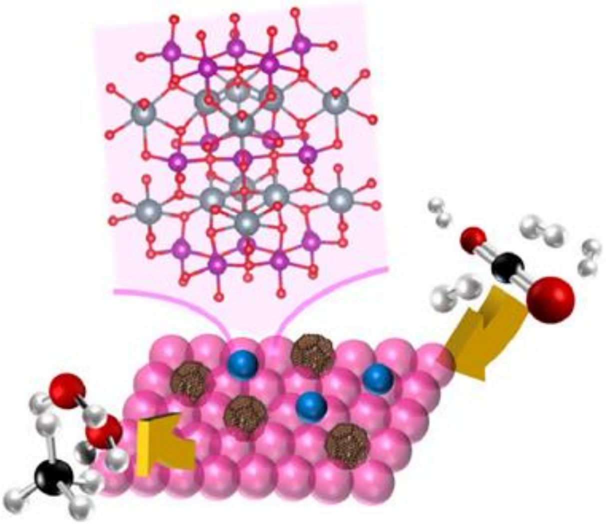
Ni/$YMnO_3$ perovskite catalyst for CO_2 methanation

González-Castaño, M; de Miguel, JCN; Penkova, A; Centeno, MA; Odriozola, JA; Arellano-García, H

Applied Materials Today, **23** (2021) 101055

Junio 2021 | **DOI:** 10.1016/j.apmt.2021.101055

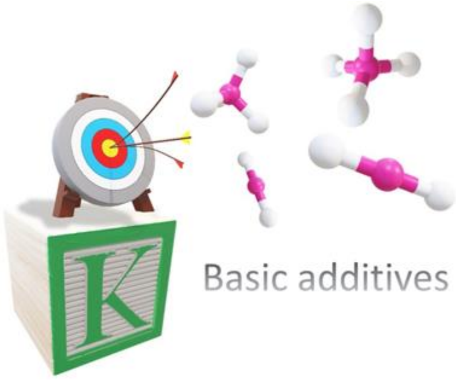
This work proposes an innovative Ni catalyst supported over $YMnO_3$ perovskite as a promising catalytic system for CO_2 methanation reaction. Under reductive conditions, the attendance of Mn redox couples within the layered perovskite structure promotes the constitution of sub-stoichiometric $YMnO_{3-x}$ units which, by means of the flexible $YMnO_{3-x}$ reorganization capacity,



results in boosted anionic mobility's. The competitive turnover frequencies (20.1 and 17.0 s^{-1} at 400 °C under dry- and steamed- CO_2 methanation conditions) displayed by $Ni/YMnO_3$ system were related to the synergism between strongly interacting Ni particles with partially reduced $YMnO_{3-x}$ perovskites. The optimal Ni dispersions, for which no relevant signs of sintering issues were discerned, combined to effective role of oxygen vacancies towards the dissociative activation of CO_2 molecules enabled highly active and stable catalytic behaviours with no evidence of cooking phenomena. On evaluating the water presence within CO_2 methanation feedstock's, the deprived catalytic behaviour was fundamentally associated to depleted oxygen vacancies concentrations and promoted WGS side reactions.

Effect of potassium loading on basic properties of $Ni/MgAl_2O_4$ catalyst for CO_2 reforming of methane

Azancot, L; Bobadilla, LF; Centeno, MA; Odriozola, JA
Journal of CO_2 Utilization, **52** (2021) 101681
Octubre 2021 | **DOI:** 10.1016/j.jcou.2021.101681



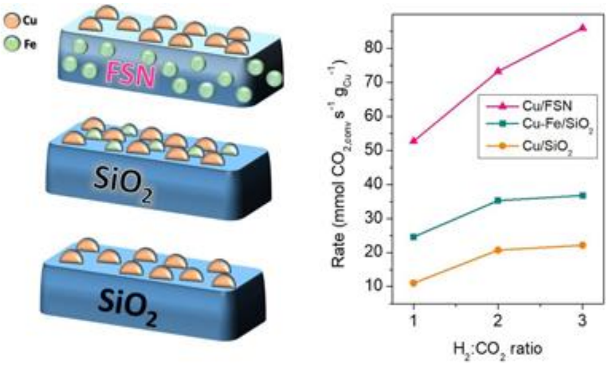
Coke deposition is one of the key issues in the dry reforming of methane on Ni catalysts. In the present work, we investigate the effect of potassium addition for suppressing carbon deposition in the Dry Reforming of Methane. The results obtained demonstrated that potassium contents above 3 wt% promote carbon gasification, favouring both Reverse Water Gas Shift and

Boudouard reaction. Strong basic Mg-O-K sites are responsible for these reactions allowing the suppression of carbon deposits and allowing the stability of the catalyst.

Cu supported Fe-SiO_2 nanocomposites for reverse water gas shift reaction

González-Castaño, M; de Miguel, JCN; Sinha, F; Wabo, SG; Klepel, O; Arellano-García, H

Journal of CO_2 Utilization, **46** (2021) 101493
Abril 2021 | **DOI:** 10.1016/j.jcou.2021.101493



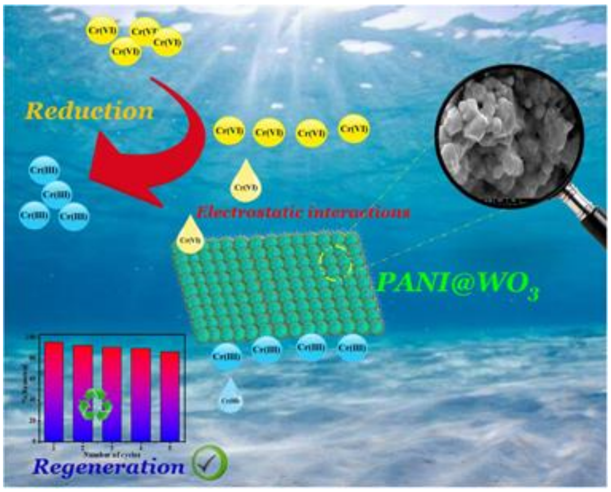
This work analyses the catalytic activity displayed by Cu/SiO_2, Cu-Fe/SiO_2 and Cu/FSN (Fe-SiO_2 nanocomposite) catalysts for the Reverse Water Gas Shift reaction. Compared to Cu/SiO_2 catalyst, the presence of Fe resulted on higher CO's selectivity and boosted resistances against the constitution of the deactivation carbonaceous species. Regarding the catalytic performance however, the extent of improvement attained through incorporation Fe species strongly relied on the catalysts' configuration. At 30 L/gh and H_2:CO_2 ratios = 3, the performance of the catalysts' series increased according to the sequence: Cu/SiO_2 < Cu-Fe/SiO_2 << Cu/FSN. The remarkable catalytic enhancements provided by Fe-SiO_2 nanocomposites under different RWGS reaction atmospheres were associated to enhanced catalyst surface basicity's and stronger Cu-support interactions. The catalytic promotion achieved by Fe-SiO_2 nanocomposites argue an optimistic prospective for nanocomposite catalysts within future CO_2-valorising technologies.

Development of a novel PANI@WO_3 hybrid composite and its application as a promising adsorbent for Cr(VI) ions removal

Hsini, A; Naciri, Y; Laabd, M; Bouziani, A; Navío, JA; Puga, F; Boukherroub, R; Lakhmiri, R; Albourine, A

Journal of Environmental Chemical Engineering, **9** (2021) 105885
Octubre 2021 | **DOI:** 10.1016/j.jece.2021.105885

In the current study, an in-situ oxidative polymerization method was used to synthesize polyaniline-coated tungsten trioxide biphasic composite (PANI@WO_3). The as-developed composite material properties were elucidated using different characterization tools such as X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR), N_2 sorption-desorption isotherm, and X-ray photoelectron spectroscopy (XPS). The PANI@WO_3 was further applied to remove hexavalent chromium (Cr(VI)) from



aqueous solutions. The results demonstrated that the optimal removal efficacy was achieved at pH 2. Meanwhile, the pseudo-second-order kinetic and isotherm of the Langmuir model were fitted for Cr(VI) adsorption. Cr(VI) amount of 549.37 $mg{\cdot}g^{-1}$ was the maximum capacity of adsorption attained for PANI@WO$_3$, which is significantly higher than that of existing adsorbents. From a thermodynamic point of view, the Cr(VI) adsorption process occurred spontaneously and endothermically. Importantly, PANI@WO$_3$ still exhibited an excellent adsorption capability after five regeneration cycles, indicating the potential reusability of the PANI@WO$_3$ composite. XPS analysis of PANI@WO$_3$ surface after adsorption of Cr(VI) confirmed its adsorption and concomitant reduction into Cr(III) ions. The transfer of mass phenomenon, electrostatic attraction, and reduction reaction were the primary processes for Cr(VI) ions elimination. These findings revealed that the synthesized PANI@WO$_3$ exhibited a high potential for wastewater treatment containing Cr(VI).

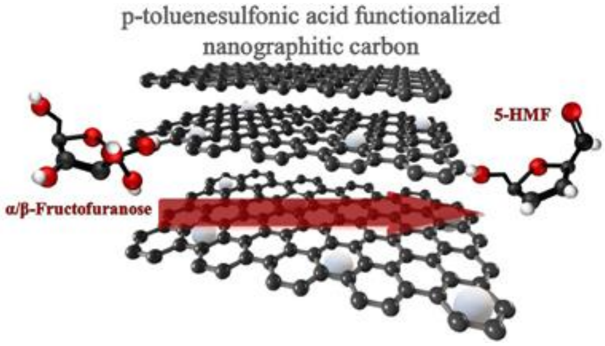
Photocatalytic production of hydrogen and methane from glycerol reforming over Pt/TiO$_2$–Nb$_2$O$_5$

Iervolino, G; Vaiano, V; Murcia, JJ; Lara, AE; Hernández, JS; Rojas, H; Navío, JA; Hidalgo, MC
International Journal of Hydrogen Energy, **46** (2021) 38678-38691
Septiembre, 2021 | **DOI:** 10.1016/j.ijhydene.2021.09.111

In this study, platinized mixed oxides (TiO$_2$–Nb$_2$O$_5$) were tested on photocatalytic hydrogen production from a glycerol solution under UV light. Different samples with different Ti:Nb ratios were prepared by using a simple method that simultaneously combined a physical mixture and a platinum photochemical reduction. This method led to improved physicochemical properties such as low band gap, better Pt nanoparticle distribution on the surface, and the formation of different Pt species. Niobia content was also found to be an important factor in determining the overall efficiency of the Pt–TiO$_2$–Nb$_2$O$_5$ photocatalyst in the glycerol reforming reaction. The photocatalytic results showed that Pt on TiO$_2$–Nb$_2$O$_5$ enhanced hydrogen production from the aqueous glycerol solution at a 5 wt% initial glycerol concentration. The influence of different operating conditions such as the catalyst dosage and initial glycerol concentration was also evaluated. The results indicated that the best hydrogen and methane production was equal to 6657 μmol/L and 194 μmol/L, respectively after 4 h of UV radiation using Pt/Ti:Nb (1:2) sample and with 3 g/L of catalyst dosage. Moreover, the role of water in photocatalytic hydrogen production was studied through photocatalytic activity tests in the presence of D_2O. The obtained results confirmed the role of water molecules on the photocatalytic production of hydrogen in an aqueous glycerol solution.

Fructose dehydration reaction over functionalized nanographitic catalysts in MIBK/H_2O biphasic system

Martín, GD; Bounoukta, CE; Ammari, F; Domínguez, MI; Monzón, A; Ivanova, S; Centeno, MA
Catalysis Today, **366** (2021) 68-76
Abril 2021 | **DOI:** 10.1016/j.cattod.2020.03.016

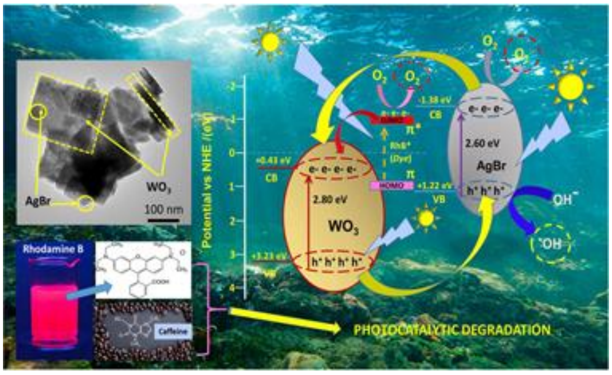


A series of functionalized nanographitic carbons is prepared, characterized and tested in fructose dehydration reaction to 5-hydroxymethylfurfural. The functionalization treatment was selected to introduce various Brönsted acid sites and to modify the textural and catalytic properties of the initial carbon material. Within the series, the sulfonated carbons present the most interesting catalytic behavior resulting in important selectivity to the desired product once the reaction variables were properly adjusted.

Features of coupled AgBr/WO_3 materials as potential photocatalysts

Puga, F; Navío, JA; Hidalgo, MC
Journal of Alloys and Compounds, **367** (2021) 159191
Junio 2021 | **DOI:** 10.1016/j.jallcom.2021.159191

AgBr/WO_3 composite photocatalysts with different selected molar AgBr/WO_3 ratios were prepared and widely characterized by XRD, N_2-adsorption, SEM, TEM, UV–visible/DRS and XPS techniques. The samples were tested using rhodamine B (RhB) or caffeine, under two illumination conditions (UV and visible light). Although AgBr and WO_3 pristine materials have relatively low band gap values (2.6 eV and 2.8 eV, respectively), they exhibit low or no photocatalytic activity under visible light, at least for caffeine degradation. This fact may be mainly related to a high recombination rate of photogenerated charge carriers in these samples. However, the coupling of both leads to a substantial improvement in the degradation of caffeine and RhB under both UV and visible lighting conditions. The increased photocatalytic activity found in the coupled systems with respect to the pristine materials can be attributed to the formation of a type II heterostructure in the coupled AgBr/WO_3 samples. Our results show that for AgBr/WO_3 coupled systems, kinetic degradation profiles have clear dependence on the molar percentages of the coupled pristine materials, as well as on the nature (sensitizing or not sensitizing effect) of the



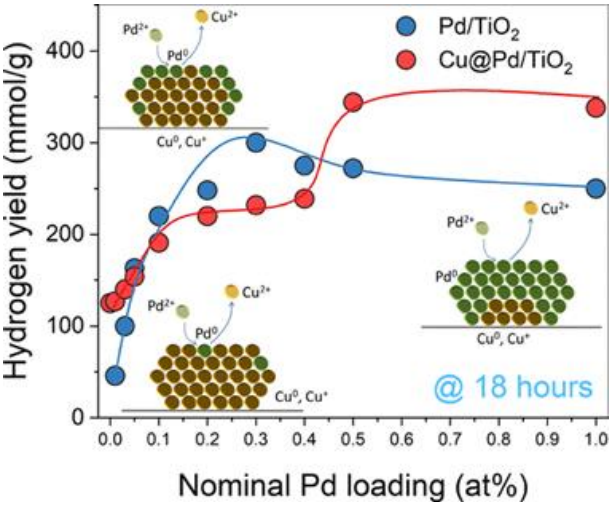
substrate. For caffeine photodegradation, the best performance was obtained when AgBr/WO$_3$(10–15%) catalysts were used. The AgBr/WO$_3$(20%) sample showed the best photocatalytic activity for rhodamine B degradation, exhibiting also excellent dark adsorption capacity (40–45%). Additionally, studies of activity in five consecutive tests showed a good RhB degradation during the successive reuses being involving a N-de-ethylation mechanism with the main O2• – radicals participation; relatively low mineralization percentages were observed, both under UV and visible light conditions. In these successive runs, no silver leaching to the medium was observed but a change from AgBr towards Ag_2CO_3 and/or Ag_xO was produced at the catalyst surface. These features should be known in the use of these systems as potential photocatalysts for practical applications.

Overcoming Pd-TiO$_2$ Deactivation during H$_2$ Production from Photoreforming Using Cu@Pd Nanoparticles Supported on TiO$_2$

Platero, F; López-Martín, A; Caballero, A; Rojas, TC; Nolan, M; Colón, G

ACS Applied Nano Materials, **4** (2021) 3204-3219

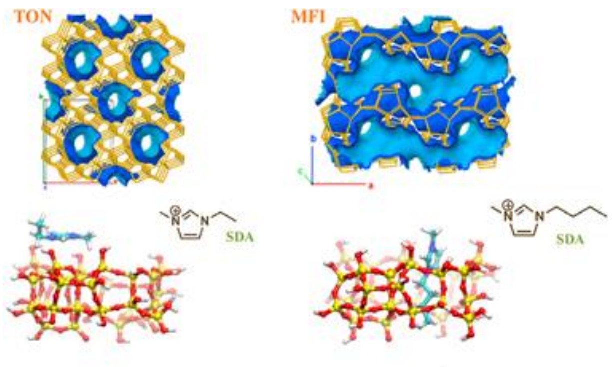
Marzo, 2021 | **DOI:** 10.1021/acsanm.1c00345



Different Cu@Pd-TiO$_2$ systems have been prepared by a two-step synthesis to obtain a bimetallic co-catalyst for the H$_2$ photoreforming reaction. We find that the tailored deposition of Pd covering the Cu nanoclusters by a galvanic replacement process results in the formation of a core@shell structure. The photocatalytic H_2 production after 18 h is 350 mmol/g on the $Cu@Pd_{1.0}$-TiO_2 bimetallic system, which is higher than that on the monometallic ones with a H_2 production of 250 mmol/g on Pd-supported TiO_2. Surface characterization by high-angle annular dark-field scanning transmission electron microscopy, H_2-temperatureprogramed reduction, CO-FTIR spectroscopy, and XPS gives clear evidence of the formation of a core@shell structure. With a Pd loading of 0.2-0.3 at. %, we propose a full coverage of the Cu nanoparticles with Pd. Long-time photoreforming runs show the enhanced performance of supported Cu@Pd with respect to bare palladium leading to a more stable catalyst and ultimately higher H_2 production.

How a small modification in the imidazolium-based SDA can determine the zeolite structure? MFI vs. TON

Megías-Sayago, C; Blanes, JMM; Szyja, BM; Odriozola, JA; Ivanova, S
Microporous and Mesoporous Materials, **322** (2021) 111160
Julio 2021 | **DOI:** 10.1016/j.micromeso.2021.111160

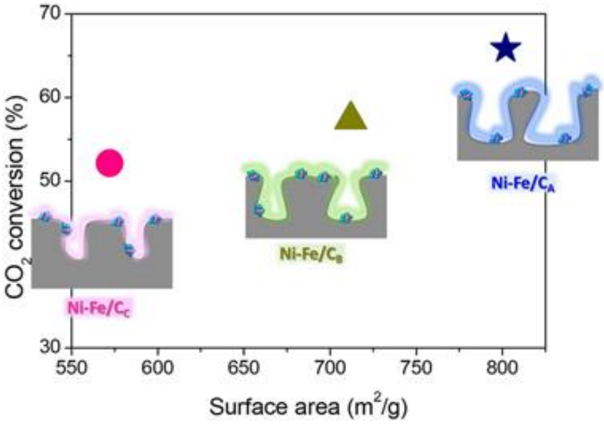


The present study proposes an important contribution to the understanding of ionic liquid role as structure directing agent for zeolite synthesis. A series of imidazolium based ionic liquids are used for this purpose. While the anionic counterpart influences the micellar organization during the synthesis, the imidazolium cation clearly directs the structure to one or another zeolite family as a function of its substituents and their interaction with the zeolite framework. The experimental observations are contrasted with molecular modeling explaining the distinct zeolite families obtained on the basis of different preferential orientation of the ionic liquids to the Si_{33} precursor.

Assessing the impact of textural properties in Ni-Fe catalysts for CO_2 methanation performance

González-Castaño, M; de Miguel, JCN; Boelte, JH; Centeno, MA; Klepel, O; Arellano-García, H
Microporous and Mesoporous Materials, **327** (2021) 111405
Noviembre 2021 | **DOI:** 10.1016/j.micromeso.2021.111405

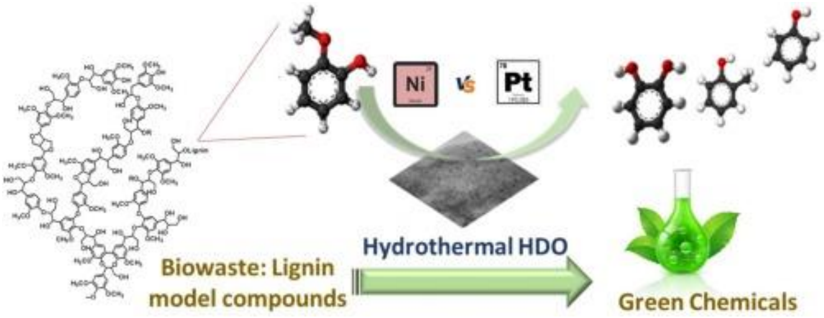
In heterogeneous catalysis, the benefits of employing adequate textural properties on the catalytic performances are usually stated. Nevertheless, the quantification of the extent of improvement is not an easy task since variations on the catalysts' specific areas and pore structures might involve modifications on a number of other surface catalytic features. This study establishes the impact



of the catalyst textural properties on the CO_2 methanation performance by investigating bimetallic Ni–Fe catalysts supported over carbon supports with different textural properties regarding surface area and pore structure. The comparable metal loading and dispersions attained for all systems enabled establishing forthright relationships between the catalyst textural properties and CO_2 methanation rate. Once the influence of the external mass diffusions on the catalysts' performance was experimentally discarded, the estimated Thiele modulus and internal effectiveness (ϕ and η_{Eff}) values showed that the catalyst performance was majorly governed by the surface reaction rate whilst the pore size affected in no significant manner within the examined range (D_{pore} = 10.2 to 5.8 nm). Therefore, the rapport between the catalyst performance and surface area was quantified for the CO_2 methanation reaction over Ni–Fe catalysts: increasing the surface area from 572 to 802 m^2/g permit obtaining ca. 10% higher CO_2 conversions.

Guaiacol hydrodeoxygenation in hydrothermal conditions using N-doped reduced graphene oxide (RGO) supported Pt and Ni catalysts: Seeking for economically viable biomass upgrading alternatives

Parrilla-Lahoz, S; Jin, W; Pastor-Pérez, L; Carrales-Alvarado, D; Odriozola, JA; Dongil, AB; Reina, TR
Applied Catalysis A-General, **611** (2021) 117977
Febrero 2021 | **DOI:** 10.1016/j.apcata.2020.117977

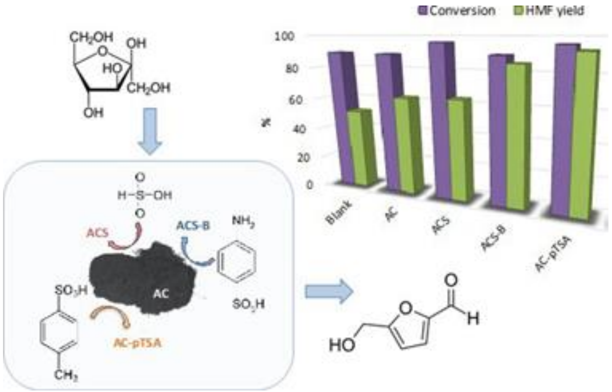


Herein we present an innovative route for model biomass compounds upgrading via "H_2-free" hydrodeoxygenation (HDO) reactions. The underlaying idea is to implement a multifunctional catalyst able to activate water and subsequently use in-situ generated hydrogen for the HDO process. In this sense we have developed a series of effective Ni and Pt based catalysts supported on N-promoted graphene decorated with ceria. The catalyst reached commendable conversion levels and selectivity to mono-oxygenated compounds considering the very challenging reaction conditions. Pt outperforms Ni when the samples are tested as-prepared. However, Ni performance is remarkably boosted upon applying a pre-conditioning reductive treatment. Indeed, our $NiCeO_2$/GOr-N present the best activity/selectivity balance and it is deemed as a promising catalyst to conduct the H_2-free HDO reaction. Overall, this "proof-concept" showcases an economically appealing route for bio-compounds upgrading evidencing the key role of advanced catalysts for a low carbon future.

Effect of the sulphonating agent on the catalytic behavior of activated carbons in the dehydration reaction of fructose in DMSO

Bounoukta, CE; Megías-Sayago, C; Ivanova, S; Penkova, A; Ammari, F; Centeno, MA; Odriozola, JA

Applied Catalysis A-General, **611** (2021) 118108
Mayo 2021 | **DOI:** 10.1016/j.apcata.2021.118108



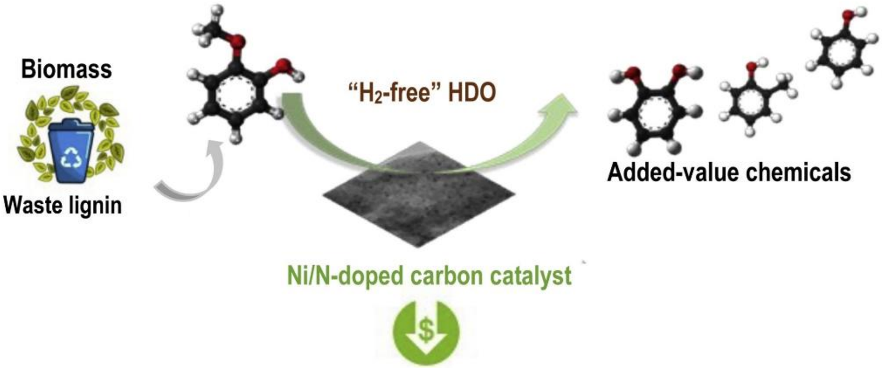
A series of -SO_3R functionalized activated carbons (R=H, O, aryl) were prepared and applied in fructose dehydration reaction to 5-hydroxymethylfurfural. Different sulphonating methods introduce groups on catalyst surface with distinct donor-acceptor and hydrophilic properties. Their nature influences significantly not only activated carbon's textural and chemical properties but also the product yields and selectivity in fructose dehydration reaction. The viability of the solvent free reaction was also investigated and compared to the performance of the catalyst series in presence of DMSO, where the best catalytic results were obtained.

In-situ HDO of guaiacol over nitrogen-doped activated carbon supported nickel nanoparticles

Jin, W; Pastor-Pérez, L; Villora-Pico, JJ; Mercedes Pastor-Blas, M; Odriozola, JA; Sepúlveda-Escribano, A; Ramírez Reina, T

Applied Catalysis A-General, **620** (2021) 118033
Junio 2021 | **DOI:** 10.1016/j.apcata.2021.118033

In-situ hydrodeoxygenation of guaiacol over Ni-based nitrogen-doped activated carbon supported catalysts is presented in this paper as an economically viable route for bio-resources



upgrading. The overriding concept of this paper is to use water as hydrogen donor for the HDO reaction, suppressing the input of external highpressure hydrogen. The effect of nitrogen sources, including polypyrrole (PPy), polyaniline (PANI) and melamine (Mel) on the structural, electronic and ultimately of catalytic features of the designed materials have been addressed. Nitrogen-doped samples are more active than the undoped counterparts in the "H_2-free" HDO process. For instance, the conversion of guaiacol increased by 8 % for Ni/PANI-AC compared to that of Ni/AC catalysts. The superior performance of Ni/NC can be attributed to the acid-base properties and modified electronic properties, which favours the C-O cleavage and water activation as well as enhances dispersion of Ni particles on the catalysts' surface.

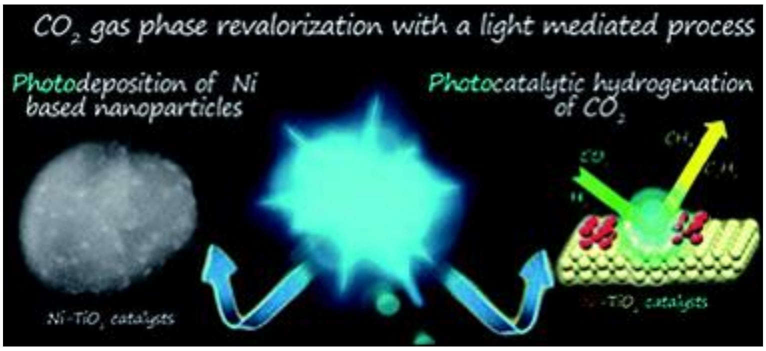
In Situ DRIFTS-MS Methanol Adsorption Study onto Supported NiSn Nanoparticles: Mechanistic Implications in Methanol Steam Reforming

Bobadilla, LF; Azancot, L; Ivanova, S; Delgado, JJ; Romero-Sarria, F; Centeno, MA; Roger, AC
Nanomaterials, **11** (2021) 3234
Diciembre 2021 | **DOI:** 10.3390/nano11123234

Methanol adsorption over both supported NiSn Nps and analogous NiSn catalyst prepared by impregnation was studied by in situ diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) to gain insights into the basis of hydrogen production from methanol steam reforming. Different intermediate species such as methoxides with different geometry (bridge and monodentate) and formate species were identified after methanol adsorption and thermal desorption. It is proposed that these species are the most involved in the methanol steam reforming reaction and the major presence of metal-support interface sites in supported NiSn Nps leads to higher production of hydrogen. On the basis of these results, a plausible reaction mechanism was elucidated through the correlation between the thermal stability of these species and the evolution of the effluent gas released. In addition, it was demonstrated that DME is a secondary product generated by condensation of methoxides over the acid sites of alumina support in an acid-catalyzed reaction.

LED-driven controlled deposition of Ni onto TiO_2 for visible-light expanded conversion of carbon dioxide into C-1-C-2 alkanes

Sanz-Marco, A; Hueso, JL; Sebastián, V; Nielsen, D; Mossin, S; Holgado, JP; Bueno-Alejo, CJ; Balas, F; Santamaría, J
Nanoscale Advances, **3** (2021) 3788-3798
Abril 2021 | **DOI:** 10.1039/d1na00021g



Photocatalytic gas-phase hydrogenation of CO_2 into alkanes was achieved over TiO_2-supported Ni nanoparticles under LED irradiation at 365 nm, 460 nm and white light. The photocatalysts were prepared using photo-assisted deposition of Ni salts under LED irradiation at 365 nm onto TiO_2 P25 nanoparticles in methanol as a hole scavenger. This procedure yielded 2 nm Ni particles decorating the surface of TiO_2 with a nickel mass content of about 2%. Before the photocatalytic runs, Ni/TiO_2 was submitted to thermal reduction at 400 °C in a 10% H_2 atmosphere which induced O-defective TiO_{2-x} substrates. The formation of oxygen vacancies, Ti^{3+} centers and metallic Ni sites upon photocatalytic CO_2 hydrogenation was confirmed by operando EPR analysis. In situ XPS under reaction conditions suggested a strong metal–support interaction and the co-existence of zero and divalent Ni states. These photoactive species enhanced the photo-assisted reduction of CO_2 below 300 °C to yield CO, CH_4 and C_2H_6 as final products.

K-Promoted Ni-Based Catalysts for Gas-Phase CO_2 Conversion: Catalysts Design and Process Modelling Validation

Gandara-Loe, J; Portillo, E; Odriozola, JA; Reina, TR; Pastor-Pérez, L
Frontiers in Chemistry, **9** (2021) 785571
Noviembre 2021 | **DOI:** 10.3389/fchem.2021.785571

The exponential growth of greenhouse gas emissions and their associated climate change problems have motivated the development of strategies to reduce CO_2 levels via CO_2 capture and conversion. Reverse water gas shift (RWGS) reaction has been targeted as a promising pathway to convert CO_2 into syngas which is the primary reactive in several reactions to obtain high-value chemicals. Among the different catalysts reported for RWGS, the nickel-based catalyst has been proposed as an alternative to the expensive noble metal catalyst. However, Ni-based catalysts tend to be less active in RWGS reaction conditions due to preference to CO_2 methanation reaction and to the sintering and coke formation. Due to this, the aim of this work is to study the effect of the potassium (K) in Ni/CeO_2 catalyst seeking the optimal catalyst for low-temperature RWGS reaction. We synthesised Ni-based catalyst with different amounts of K:Ni ratio (0.5:10, 1:10, and 2:10) and fully characterised using different physicochemical techniques where was observed the modification on the surface characteristics as a function of the amount of K. Furthermore, it was observed an improvement in the CO selectivity at a lower temperature as a result of the K-Ni-support interactions but also a decrease on the CO_2 conversion. The 1K catalyst presented the best compromise between CO_2 conversion, suppression of CO_2 methanation and enhancing CO selectivity. Finally, the experimental results were contrasted with the trends obtained from the thermodynamics process modelling observing that the result follows in good agreement with the modelling trends giving evidence of the promising behaviour of the designed catalysts in CO_2 high-scale units.

Biogas Conversion to Syngas Using Advanced Ni-Promoted Pyrochlore Catalysts: Effect of the CH_4/CO_2 Ratio

le Sache, E; Moreno, AA; Reina, TR
Frontiers in Chemistry, **9** (2021) 672419
Abril 2021 | **DOI:** 10.3389/fchem.2021.672419

Biogas is defined as the mixture of CH_4 and CO_2 produced by the anaerobic digestion of biomass. This particular mixture can be transformed in high valuable intermediates such as syngas through a process known as dry reforming (DRM). The reaction involved is highly endothermic, and catalysts capable to endure carbon deposition and metal particle sintering are required. Ni-pyrochlore catalysts have shown outstanding results in the DRM. However, most reported data deals with CH_4/CO_2 stoichiometric ratios resulting is a very narrow picture of the overall biogas upgrading via DRM. Therefore, this study explores the performance of an optimized Ni-doped pyrochlore, and Ni-impregnated pyrochlore catalysts in the dry reforming of methane, under different CH_4/CO_2 ratios, in order to simulate various representatives waste biomass feedstocks. Long-term stability tests showed that the ratio CH_4/CO_2 in the feed gas stream has an important influence in the catalysts' deactivation. Ni doped pyrochlore catalyst, presents less deactivation than the Ni-impregnated pyrochlore. However, biogas mixtures with a CH_4 content higher than 60%, lead to a stronger deactivation in both Ni-catalysts. These results were in agreement with the thermogravimetric analysis (TGA) of the post reacted samples that showed a very limited carbon formation when using biogas mixtures with CH_4 content $<60\%$, but CH_4/CO_2 ratios higher than 1.25 lead to an evident carbon deposition. TGA analysis of the post reacted Ni impregnated pyrochlore, showed the highest amount of carbon deposited, even with lower stoichiometric CH_4/CO_2 ratios. The later result indicates that stabilization of Ni in the pyrochlore structure is vital, in order to enhance the coke resistance of this type of catalysts.

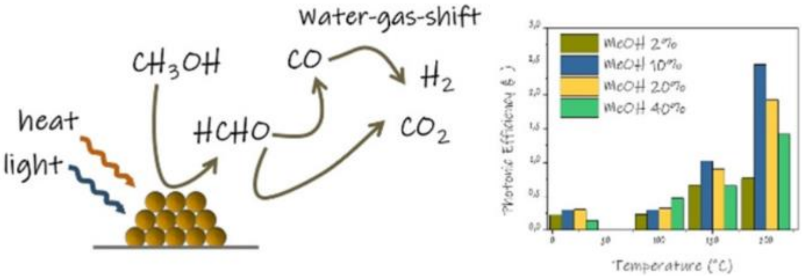
Bimetallic Ni-Ru and Ni-Re Catalysts for Dry Reforming of Methane: Understanding the Synergies of the Selected Promoters

Moreno, AA; Ramírez-Reina, T; Ivanova, S; Roger, AC; Centeno, MA; Odriozola, JA
Frontiers in Chemistry, **9** (2021) 694976
Julio 2021 | **DOI:** 10.3389/fchem.2021.694976

Designing an economically viable catalyst that maintains high catalytic activity and stability is the key to unlock dry reforming of methane (DRM) as a primary strategy for biogas valorization. Ni/Al_2O_3 catalysts have been widely used for this purpose; however, several modifications have been reported in the last years in order to prevent coke deposition and deactivation of the samples. Modification of the acidity of the support and the addition of noble metal promoters are between the most reported strategies. Nevertheless, in the task of designing an active and stable catalyst for DRM, the selection of an appropriate noble metal promoter is turning more challenging owing to the lack of homogeneity of the different studies. Therefore, this research aims to compare Ru (0.50 and 2.0%) and Re (0.50 and 2.0%) as noble metal promoters for a $Ni/MgAl_2O_4$ catalyst under the same synthesis and reaction conditions. Catalysts were characterized by XRF, BET, XRD, TPR, hydrogen chemisorption (H_2-TPD), and dry reforming reaction tests. Results show that both promoters increase Ni reducibility and dispersion. However, Ru seems a better promoter for DRM since 0.50% of Ru increases the catalytic activity in 10% and leads to less coke deposition.

Mechanistic Considerations on the H_2 Production by Methanol Thermal-assisted Photocatalytic Reforming over Cu/TiO_2 Catalyst

Platero, F; López-Martín, A; Caballero, A; Colón, G
CHEMCATCHEM, **13** (2021) 3879-3888
Julio 2021 | **DOI:** 10.1002/cctc.202100680

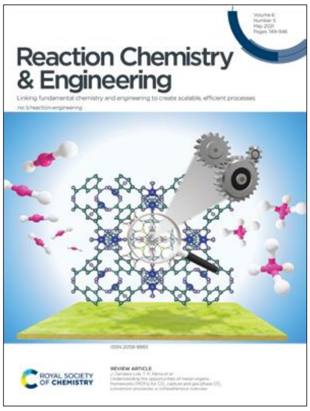


We have studied the gas phase H_2 production by methanol thermo-photoreforming using Cu-modified TiO_2. Metal co-catalyst has been deposited by means of photodeposition method. The concentration of methanol in the steam was also considered. It appears that H_2 production is notably higher as temperature increases. Moreover, the optimum H_2 yield is achieved using methanol concentration of 10 % v/v. CO and CO_2 were monitored as side products of the overall reaction. It has been stated that CO evolution is significant at lower temperatures. As temperature increases, CO evolution is hindered and H_2 appeared boosted. We have demonstrated that other reactions such water-gas-shift or formate dehydration would participate in the overall process. On this basis, optimal operational condition for H_2 production is attained for thermo-photocatalytic reforming of methanol solution 10 % v/v at 200 °C.

Elucidating the Promotional Effect of Cerium in the Dry Reforming of Methane

Rodríguez-Gómez, A; López-Martín, A; Ramírez, A; Gascón, J; Caballero, A
CHEMCATCHEM, **13** (2021) 553-563
Enero 2021 | **DOI:** 10.1002/cctc.202001527

A series of Ni-Ce catalysts supported on SBA-15 has been prepared by co-impregnation, extensively characterized and evaluated in the carbon dioxide reforming of methane (DRM). The characterization by TEM, XRD and TPR has allowed us to determine the effect of metal loading on metal dispersion. Cerium was found to improve nickel location inside the mesopores of SBA-15 and to suppress coke formation during the DRM reaction. The analysis by XPS allowed us to associate the high cerium dispersion with the presence of low-coordinated Ce^{3+} sites, being main responsible for its promotional effect. A combination of XAS and XPS has permitted us to determine the physicochemical properties of metals under reduction conditions. The low nickel coordination number determined by XAS in N-Ce doped systems after reduction suggests the generation of very small nickel particles which showed greater catalytic activity and stability in the reaction, and a remarkable resistance to coke formation.



Understanding the opportunities of metal-organic frameworks (MOFs) for CO_2 capture and gas-phase CO_2 conversion processes: a comprehensive overview

Gandara-Loe, J; Pastor-Pérez, L; Bobadilla, LF; Odriozola, JA; Reina, TR

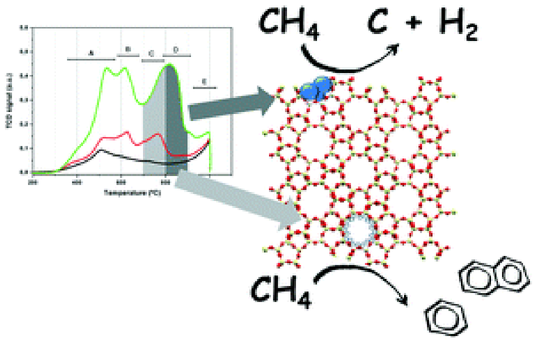
Reaction Chemistry & Engineering, **6** (2021) 787-814
Front Cover | Mayo 2021 | **DOI:** 10.1039/d1re00034a

The rapid increase in the concentration of atmospheric carbon dioxide is one of the most pressing problems facing our planet. This challenge has motivated the development of different strategies not only in the reduction of CO_2 concentrations via green energy alternatives but also in the capture and conversion of CO_2 into value-added products. Metal-organic frameworks (MOFs) are a relatively new class of porous materials with unique structural characteristics such as high surface areas, chemical tunability and stability, and have been extensively studied as promising materials to address this challenge. This comprehensive review identifies the specific structural and chemical properties of MOFs that result in advanced CO_2 capture capacities and fairly encouraging catalytic CO_2 conversion behaviour. More importantly, we describe an interconnection among the unique properties of MOFs and the engineering aspects of these intriguing materials towards CO_2 capture and conversion processes.

Elucidating the nature of Mo species on ZSM-5 and its role in the methane aromatization reaction

López-Martín, A; Platero, F; Colón, G; Caballero, A

Reaction Chemistry & Engineering, **6** (2021) 1265-1276
Abril 2021 | **DOI:** 10.1039/d1re00044f



The valorization of methane is one of the most important goals during the transition period to the general use of renewable energies. Its transformation into a valuable chemical like benzene by direct aromatization of methane (DAM) reaction has been extensively studied in the past years, mainly using Mo/ZSM-5 catalytic systems. Although viable, this DAM reaction poses a number of issues mainly derived from poor conversion and deactivation processes. Therefore, a deeper knowledge of these systems is needed. Herein, by combining chemical (TPR), spectroscopic

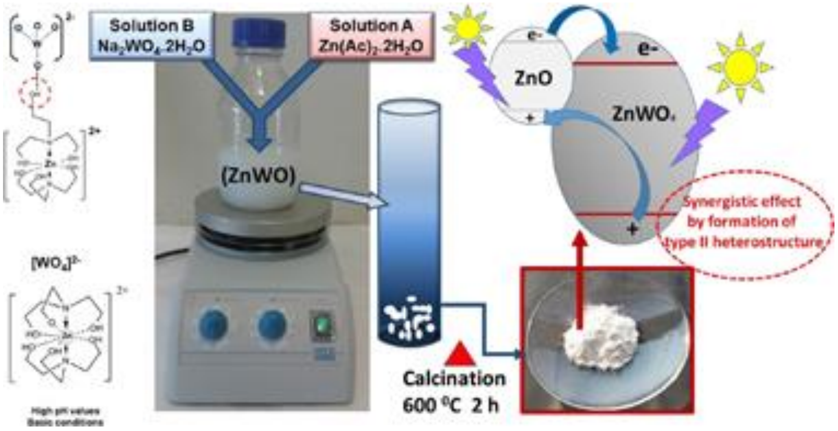
(XPS), HAADF and other techniques, we have identified the different Mo precursors stabilized in the calcined ZSM-5 support, their nature (monomers, dimers and bulk Mo oxides), location in the zeolite framework (external surface or micropores), and the partial segregation of aluminum during the preparation of catalysts. The role of each Mo phase promoting or hindering the transformation of methane in aromatics has been also clarified.

Sol-gel synthesis of $ZnWO_4$-(ZnO) composite materials. Characterization and photocatalytic properties

Jaramillo-Páez, C; Navío, JA; Puga, F; Hidalgo, MC

Journal of Photochemistry & Photobiology, A: Chemistry, **404** (2021) 112962

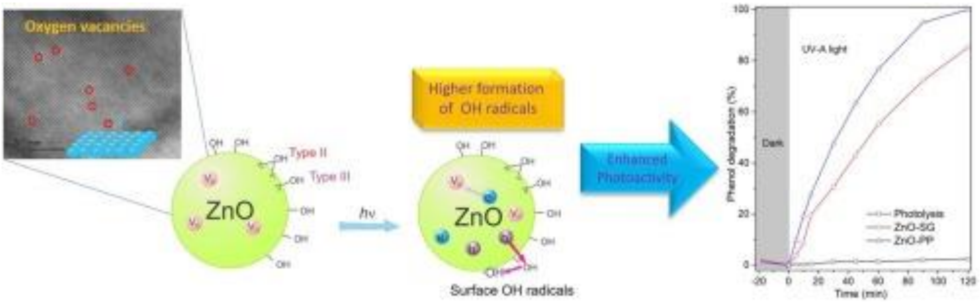
Enero 2021 | **DOI:** 10.1016/j.jphotochem.2020.112962



$ZnWO_4$ based powder photocatalyst have been successfully prepared by calcining a co-precipitated precursor (ZnWO) obtained from aqueous Zn^{2+} and WO_4^{2-} solutions at pH = 7, without surfactants addition. The as-formed sample was characterized by XRD, N_2-absorption, SEM, TEM, DRS and XPS. Both techniques, XRD and XPS results showed that prepared sample corresponds to a crystalline, Zn-enriched composition, $ZnWO_4$ indicating the formation of a $ZnWO_4$-(ZnO) composite, whit ca. 10 wt.-% of ZnO confirmed by XRF analysis. Photocatalytic activities towards degradation of Rhodamine B (RhB), Methyl Orange (MO) and Phenol, under UV-illumination, was investigated not only by monitoring the percentages of conversion of substrates, but also by estimating the corresponding percentages of mineralization that accompany the photocatalytic process. Comparative substrateconversion rates estimated per surface area unit of catalyst, showed that the activity for $ZnWO_4$-(ZnO) composite is similar to that for TiO_2(P25), at least for MO and RhB, and even higher that for TiO_2(P25) in respect to phenol conversion. By adding TEA to the synthesis procedure, a composite named as $ZnWO_4$-ZnO-(pH = 10)-600 is generated, which has a higher proportion of ZnO (ca. 39 %) and superior specific surface area than the so-called $ZnWO_4$-(ZnO) sample. Furthermore, the photocatalytic degradation of MO using the former material indicates that it is superior to $ZnWO_4$-(ZnO) and even that TiO_2(P25) itself under the same operational conditions.

Photocatalytic activity of ZnO nanoparticles and the role of the synthesis method on their physical and chemical properties

Uribe-López, MC; Hidalgo-López, MC; López-González, R; Frías-Márquez, DM; Núñez-Nogueira, G; Hernández-Castillo, D; Álvarez-Lemus, MA
Journal of Photochemistry & Photobiology, A: Chemistry, **404** (2021) 112866
Enero 2021 | **DOI:** 10.1016/j.jphotochem.2020.112866



In the present study, we report on the effect of the synthesis method in the photoactivity of ZnO-NPs. The nanoparticles were prepared by precipitation and sol-gel procedures using zinc nitrate and zinc (II) acetylacetonate as ZnO precursors, respectively. The obtained samples were named as ZnO-PP (precipitation method) and ZnO-SG (sol-gel method). The powders were calcined at 500 °C and further characterized by Fourier Transform Infrared spectroscopy, X-ray Powder Diffraction, N_2 adsorption, thermal analysis, Diffuse Reflectance UV-Vis spectroscopy, and Electron Microscopy. Both methods of synthesis lead to formation of pure ZnO with hexagonal-wurtzite crystalline structures with average crystallite sizes similar to 30 nm. The specific surface area was affected by the synthesis method, since SBET values were 5 m^2/g and 13 m^2/g for sol-gel and precipitation method, respectively. The electron microscopy revealed significant changes in morphology for the obtained nanoparticles, as sol-gel directed the hexagonal rod-like geometries (similar to 50 nm in diameter) while quasi-spherical nanoparticles (similar to 100 nm in diameter) were formed using precipitation method. Photocatalytic activity was estimated by degrading phenol (50 ppm) as probe molecule under UVA irradiation ($\lambda = 356$ nm), the results demonstrated that ZnO-PP reached 100 % of degradation after 120 min and 90 % of the pollutant was mineralized, whereas for ZnO-SG the results were 80 % and 48 % respectively. Fluorescence test using terephthalic acid (TA) demonstrated higher formation of OH• radicals for ZnO synthesized by precipitation method, which could explain the higher photodegradation and mineralization observed. These results support that even slight differences in physical and chemical properties of ZnO, have a significant impact on the photocatalytic performance of such nanoparticles.

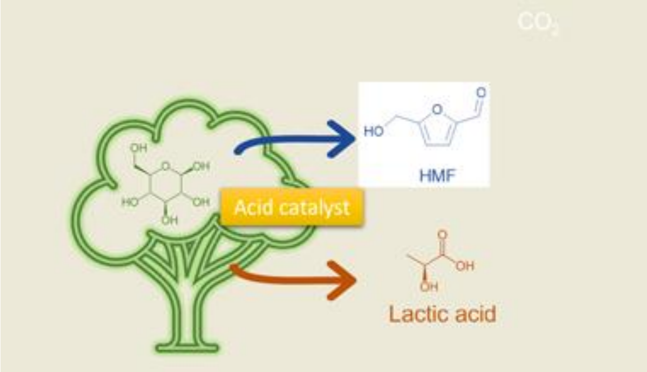
Zr and Fe on Pt/CeO_2-MO_x/Al_2O_3 catalysts for WGS reaction

González-Castaño, M; Ivanova, S; Centeno, MA; Ioanides, T; Arellano-García, H; Odriozola, JA
International Journal of Hydrogen Energy, **45** (2021) 13978-13989
Marzo 2021 | **DOI:** 10.1002/er.6646

By evaluating the functional modifications induced by Zr and Fe as dopants in Pt/CeO_2-MO_x/Al_2O_3 catalysts (M = Fe and Zr), the key features for improving water gas shift (WGS) performance for these systems have been addressed. Pt/ceria intrinsic WGS activity is often related to improved H_2 surface dynamics, H_2O absorption, retentions and dissociation capacities which are influenced greatly by the support nature. Two metals, iron and zirconia, were chosen as ceria dopants in this work, either in separate manner or combined. Iron incorporation resulted in CO-redox properties and oxygen storage capacities (OSC) improvement but the formation of Ce-Fe solid solutions did not offer any catalytic benefit, while the Zr incorporation influenced in a great manner surface electron densities and shows higher catalytic activity. When combined both metals showed an important synergy evidenced by 30% higher CO conversions and attributed to greater surface electron densities population and therefore absorption and activity. This work demonstrates that for Pt/ceria catalysts OSC enhancement does not necessarily imply a catalytic promotion.

Recent Advances in the Bronsted/Lewis Acid Catalyzed Conversion of Glucose to HMF and Lactic Acid: Pathways toward Bio-Based Plastics

Megías-Sayago, C; Navarro-Jaén, S; Drault, F; Ivanova, S
Catalysts, **11** (2021) 1395
Noviembre 2021 | **DOI:** 10.3390/catal11111395

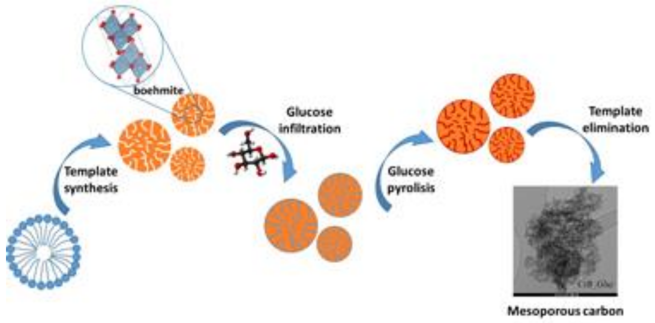


One of the most trending topics in catalysis recently is the use of renewable sources and/or non-waste technologies to generate products with high added value. That is why, the present review resumes the advances in catalyst design for biomass chemical valorization. The variety of involved reactions and functionality of obtained molecules requires the use of multifunctional catalyst able to increase the efficiency and selectivity of the selected process. The use of glucose as platform molecule is proposed here and its use as starting point for biobased plastics production is revised with special attention paid to the proposed tandem Bronsted/Lewis acid catalysts.

Mesoporous Carbon Production by Nanocasting Technique Using Boehmite as a Template

Ortega-Franqueza, M; Ivanova, S; Domínguez, MI; Centeno, MA
Catalysts, **11** (2021) 1132
Septiembre 2021 | **DOI:** 10.3390/catal11091132

A series of mesoporous carbonaceous materials were synthesized by the nanocasting technique using boehmite as a template and glucose as a carbon precursor. After pyrolysis and template removal, the resulting material is a mesoporous carbon that can be additionally doped with N, B and K during prepyrolysis impregnation. In addition, the influence of doping on the morphology,

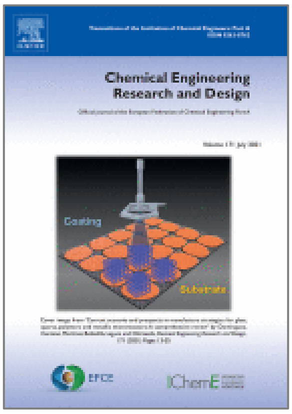


crystallinity and stability of the synthesized carbons was studied using X-ray diffraction, nitrogen physisorption, thermogravimetry, Raman and IR spectroscopy and transmission electron microscopy. While the nanocasting process is effective for the formation of mesopores, KOH and urea do not modify the textural properties of carbon. The use of H_3PO_4 as a dopant, however, led to the formation of an $AlPO_4$ compound and resulted in a solid with a lower specific surface area and higher microporosity. All doped solids present higher thermal stability as a positive effect of the introduction of heteroatoms to the carbon skeleton. The phosphorus-doped sample has better oxidation resistance, with a combustion temperature 120-150 °C higher than those observed for the other materials.

$LaFeO_3$ Modified with Ni for Hydrogen Evolution Via Photocatalytic Glucose Reforming in Liquid Phase

Iervolino, G; Vaiano, V; Sannino, D; Puga, F; Navío, JA; Hidalgo, MC
Catalysts, **11** (2021) 1558
Diciembre 2021 | **DOI:** 10.3390/catal11121558

In this work, the optimization of Ni amount on $LaFeO_3$ photocatalyst was studied in the photocatalytic molecular hydrogen production from glucose aqueous solution under UV light irradiation. $LaFeO_3$ was synthesized via solution combustion synthesis and different amount of Ni were dispersed on $LaFeO_3$ surface through deposition method in aqueous solution and using $NaBH_4$ as reducing agent. The prepared samples were characterized with different techniques: Raman spectroscopy, UltraViolet-Visible Diffuse Reflectance Spettroscopy (UV–Vis-DRS), X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), X-ray Fluorescence (XRF), Transmission Electron microscopy (TEM), and Scanning Electron microscopy (SEM) analyses. For all the investigated photocatalysts, the presence of Ni on perovskite surface resulted in a better activity compared to pure $LaFeO_3$. In particular, it is possible to identify an optimal amount of Ni for which it is possible to obtain the best hydrogen production. Specifically, the results showed that the optimal Ni amount was equal to nominal 0.12 wt% (0.12Ni/$LaFeO_3$), for which the photocatalytic H_2 production was equal to 2574 µmol/L after 4 h of UV irradiation. The influence of different of photocatalyst dosage and initial glucose concentration was also evaluated. The results of the optimization of operating parameters indicated that the highest molecular hydrogen production was achieved on 0.12Ni/$LaFeO_3$ sample with 1.5 g/L of catalyst dosage and 1000 ppm initial glucose concentration. To determine the reactive species that play the most significant role in the photocatalytic hydrogen production, photocatalytic tests in the presence of different radical scavengers were performed. The results showed that •OH radical plays a significant role in the photocatalytic conversion of glucose in H_2. Moreover, photocatalytic tests carried out with D2O instead of H_2O evidenced the role of water molecules in the photocatalytic production of molecular hydrogen in glucose aqueous solution.



Current scenario and prospects in manufacture strategies for glass, quartz, polymers and metallic microreactors: A comprehensive review

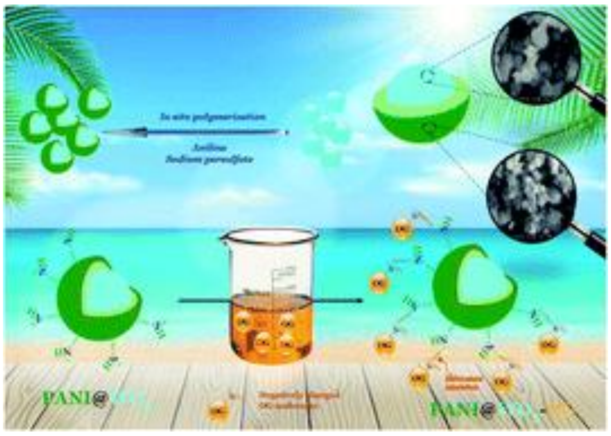
Domínguez, MI; Centeno, MA; Martínez, TM; Bobadilla, LF; Laguna, OH; Odriozola, JA
Chemical Engineering Research & Design, **171** (2021) 13-35
Front Cover | Julio 2021 | **DOI:** 10.1016/j.cherd.2021.05.001

One of the most remarkable benefits of the microreactors is the achievement of more efficient processes by enhancing the heat and mass transfer phenomena, which is the key factor for processes intensification in chemical reactions, resulting in higher conversion, selectivity and yield towards desired products. Currently, the entire scenario of microreaction approach is an emergent technology and further advances are ongoing. Several strategies have been successfully applied for structuring processes that imply the fixation of the catalysts on the microreactors. However, there are features such as the physicochemical stability of the coatings under reaction conditions that must be improved, motivating the search for new protocols. This review provides a general overview of the most important methodologies applied for glass, quartz, polymers and metals microreactors manufacture and for their coating, analyzing the advantages and drawbacks of every procedure. Furthermore, an outline of the novel insights based on additive manufacturing techniques are described.

Polyaniline coated tungsten trioxide as an effective adsorbent for the removal of orange G dye from aqueous media

Hsini, A; Naciri, Y; Bouziani, A; Aarab, N; Essekri, A; Imgharn, A; Laabd. M; Navío, JA; Puga, F; Lakhmirid, R; Albourine, A
RSC Advances, **11** (2021) 31272-31283
Noviembre 2021 | **DOI:** 10.1039/D1RA04135E

In this work, the core–shell PANI@WO_3 composite was obtained from the reaction of aniline monomer polymerization with WO_3 particles; sodium persulfate was used as an oxidant. Various analytical techniques such as scanning electron microscopy (SEM-EDS), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Brunauer–Emmett–Teller (BET), and X-ray photoelectron spectroscopy (XPS) were used to characterize the as-prepared PANI@WO_3 adsorbent, which well confirmed that the WO_3 particles were coated by polyaniline polymer. The PANI@WO_3 composite was tested as an adsorbent to remove reactive orange G (OG) for the first time. pH, adsorbent dose, contact time, initial dye concentration, and temperature were systematically investigated in order to study their effect on the adsorption process. The experimental findings showed that the PANI@WO_3 composite has considerable potential to remove an aqueous OG dye. Langmuir and Freundlich's models were used to analyze the equilibrium isotherms of OG dye adsorption on the PANI@WO3 composite. As a result, the best correlation of the experimental data was provided by the Langmuir model, and the maximum capacity of adsorption was 226.50 mg g^{-1}. From a thermodynamic point of view, the OG dye



adsorption process occurred spontaneously and endothermically. Importantly, PANI@WO_3 still exhibited an excellent adsorption capability after four regeneration cycles, indicating the potential reusability of the PANI@WO_3 composite. These results indicate that the as prepared PANI@WO_3 composite could be employed as an efficient adsorbent and was much better than the parent material adsorption of OG dye.

ZnO/Ag_3PO_4 and ZnO–Malachite as Effective Photocatalysts for the Removal of Enteropathogenic Bacteria, Dyestuffs, and Heavy Metals from Municipal and Industrial Wastewater

Murcia, JJ; Hernández Miño, JS; Rojas, H; Brijaldo, MH; Martín-Gómez, AN; Sánchez-Cid, P; Navío, JA; Hidalgo, MC; Jaramillo-Pérez, C
Water, **13** (2021) 2264
Agosto 2021 | **DOI:** 10.3390/w13162264

Different composites based on ZnO/Ag_3PO_4 and ZnO–malachite ($Cu_2(OH)_2CO_3$) were synthesized in order to determine their effectiveness in the treatment of municipal and industrial wastewaters (mainly polluted by enteropathogenic bacteria, dyes, and heavy metals). The addition of Ag_3PO_4 and malachite did not significantly modify the physicochemical properties of ZnO; however, the optical properties of this oxide were modified as a result of its coupling with the modifiers. The modification of ZnO led to an improvement in its effectiveness in the treatment of municipal and industrial wastewater. In general, the amount of malachite or silver phosphate and the effluent to be treated were the determining factors in the effectiveness of the wastewater treatment. The highest degree of elimination of bacteria from municipal wastewater and discoloration of textile staining wastewater were achieved by using ZnO/Ag_3PO_4 (5%), but an increase in the phosphate content had a detrimental effect on the treatment. Likewise, the highest Fe and Cu photoreduction from coal mining wastewater was observed by using ZnO–malachite (2.5%) and ZnO/Ag_3PO_4 (10%), respectively. Some of the results of this work were presented at the fourth Congreso Colombiano de Procesos Avanzados de Oxidación (4CCPAO$_x$).

Photocatalytic Treatment of Stained Wastewater Coming from Handicraft Factories. A Case Study at the Pilot Plant Level

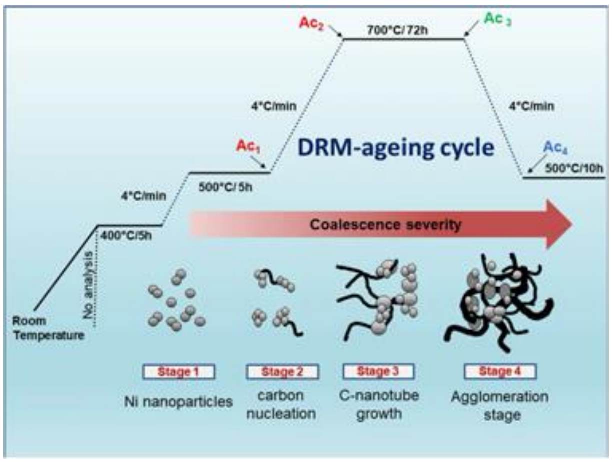
Murcia Mesa, JJ; Hernández Niño, JS; González, W; Rojas, H; Hidalgo, MC; Navío, JA
Water, **13** (2021) 2705
Octubre 2021 | **DOI:** 10.3390/w13192705

UV/H_2O_2 process and TiO_2-based photocatalysis were studied in the present work. The effectiveness of these methods was tested in the treatment of effluents taken from handicraft factories. Microorganisms, dyes, and different organic pollutants were detected in the industrial effluents. The experimental procedure for the wastewater treatment was carried out in a patented sunlight reactor on a pilot plant scale. From this study, UV/H_2O_2 was found to be the best treatment for dye elimination. The optimal peroxide dosage for the degradation of dyes and the elimination of bacteria was 0.07 M. In this case, 70.80% of discoloration was achieved after 7 h of sunlight exposure, under an average sunlight intensity of 3.42 W/m^2. The photocatalytic treatment based on TiO_2 achieved the highest elimination of coliform bacteria and the lowest TOC value; however, the presence of this material in the reactor had a detrimental effect on the overall elimination of dyes. A combination of both UV/H_2O_2 and TiO_2 treatments significantly improves the dyes discoloration, the elimination of bacteria, and the organic compounds degradation. Some of the results of this study were presented at the 4th Congreso Colombiano de Procesos Avanzados de Oxidación, 4CCPAOx.

Examination of the Deactivation Cycle of NiAl- and NiMgAl-Hydrotalcite Derived Catalysts in the Dry Reforming of Methane

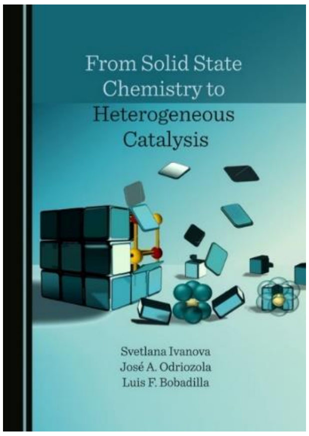
Abdelsadek, Z; Holgado, JP; Halliche, D; Caballero, A; Cherifi, O; González-Cortés, S; Masset, PJ
Catalysis Letters, **151** (2021) 2696-2715
Septiembre 2021 | **OI:** 10.1007/s10562-020-03513-4

The importance of the dry reforming of methane (DRM) lies in its capability to upgrade two greenhouse gases (CH_4 and CO_2) into synthesis gas (CO and H_2), which is one of the main building block for synthesizing hydrocarbons. However, the Ni-based catalysts for DRM reaction usually have a major catalytic stability drawback. This works aims to assess the catalytic activity and stability of two Ni-based catalysts obtained from hydrotalcite (HT) precursors (i.e., NiAl-HT and NiMgAl-HT). The precursors, calcined (-c), reduced (-R) and spent samples were characterized by a series of techniques to gain insight into the influence of MgO over Ni-based catalyst in the



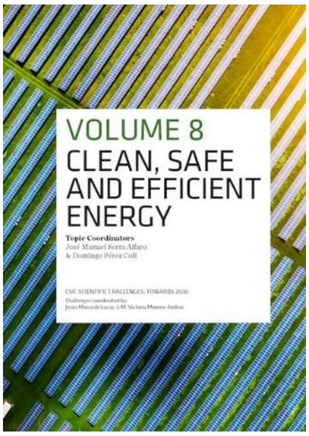
drying reforming of methane. An in-situ ageing cycle process to speed up the deactivation of hydrotalcite-derived catalysts showed that the NiMgAl-HTc-R catalyst displayed a higher activity and resistance to coke formation (stability) than NiAl-HTc-R because of the introduction of Mg into hydrotalcite structure in the catalyst precursor. The presence of this element enhances several factors involved in the stability of Ni-based catalysts for the DRM process such as the reducibility and textural features of the catalysts, size and dispersion of Ni^{o} nanoparticles and also maintains a good compromise between the acid and base properties of the solid catalysts.

Libros y Otras Publicaciones / Books and Other Publications



From Solid State Chemistry to Heterogeneous Catalysis
Svetlana Ivanova, José Antonio Odriozola, Luis F. Bobadilla
Cambridge Scholars Publishing
2021, 330 páginas
ISBN: 1-5275-7210-2
ISBN13: 978-1-5275-7210-2

Heterogeneous catalysis is deeply founded on solid state chemistry, but the relationship between the two often appears to be elusive in many cases. It is generally difficult to relate the allusion of symmetry to the crystal structure and the defect chemistry or acid-base properties to the surface reconstruction and extended defects that in most cases are the basis of physicochemical properties and solids applications. This book provides insights into solid state chemistry in order to widen the vision of heterogeneous catalysis. It covers a broad range of solid state related topics, including symmetry and structure organization, bonding, and methods for structure elucidation, as well as defects formation and their implications in heterogeneous catalysis.



Clean, safe and efficient energy Volumen 8
CSIC Scientific Challenges : Towards 2030
Consejo Superior de Investigaciones Científicas
2021
ISBN Vol. 8: 978-84-00-10752-9

Challengue 7: Catalysis for Industrial Production and of Energy Resources
José Carlos Conesa, Manuel López, Vicente Cortés, Francisco Plou, Miguel Antonio Peña, Juan Manuel Coronado, Ana Iglesias, Pedro Ávila, Miguel Ángel Bañares, Ana Serrano, Agustín Martínez, Mercedes Boronat, Miguel Ángel Centeno, Agustín R. González-Elipe, Jesús Pérez Torrente
páginas 175-205

Ni stabilised on complex structures for dry reforming of methane (DRM): Pyrochlores vs MOF
J.L.M. Espejo, J. Gándara-Loe, J.A. Odriozola, T.R. Reina, L. Pastor-Pérez
Reunión de la Sociedad Española de Catálisis SECAT 2021, pag. 214-215
ISBN: 978-84-09-34568-7

Mechanistic insights for designing optimal catalysts for low-temperature RWGS reaction: a step ahead on gas-phase CO_2 conversion
G.T. Sempere, L.F. Bobadilla, T. R. Reina, L. Pastor-Pérez, J.A. Odriozola
Reunión de la Sociedad Española de Catálisis SECAT 2021, pag. 222-223
ISBN: 978-84-09-34568-7

Hidrodesoxigenación del ácido levulínico a gamma valerolactona con catalizadores de rutenio soportados en materiales carbonosos
Ch. E. Bounoukta, S. Ivanova, F. Ammari, M.A. Centeno, J.A. Odriozola
Reunión de la Sociedad Española de Catálisis SECAT 2021, pag. 300-301
ISBN: 978-84-09-34568-7

Catalizadores de paladio soportados en materiales preparados por descomposición térmica de melamina para las reacciones del uso de ácido fórmico como vector de H_2
L. Lobo, M. Martínez-Tejada, L.F. Bobadilla, S. Ivanova, M.A. Centeno
Reunión de la Sociedad Española de Catálisis SECAT 2021, pag. 377-378
ISBN: 978-84-09-34568-7

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

PARTICIPACIÓN EN LA ORGANIZACIÓN DE CONGRESOS Y REUNIONES / PARTICIPATION IN ORGANISING CONGRESSES AND MEETINGS

XXIX International Materials Research Congress IMRC2021
15 – 20 agosto [Online, Cancún, México]

Tomás Ramírez Reina (Workshop Chairs/Organisers)

COMUNICACIONES / COMMUNICATIONS

Catalysis Conference 2021
22 – 26 febrero [Virtual, San Francisco, Estados Unidos de América]

Multicomponent catalysts for CO2 valorisation
T. Ramírez Reina
Keynote Online

High-Temperature Corrosion and Protection of Materials HTCPM 2021
28 marzo – 2 abril [Toulouse, Francia]

Influence of noble metals in the growth of the oxide scale in a commercial ferritic stainless steel EN 1.4509
P. Navarro, J. Almagro, J.A. Odriozola
Póster

8th International Conference on Engineering for Waste and Biomass Valorisation
31 mayo – 4 junio [Online, Canadá]

Optimization of Visible Photocatalytic Activity of a $Co_{0.2}Zn_{0.8}O$ System Studied in the BB41 Photodegradation via Box-Behnken Design
K. Tanji, M. Zoheir, A. Arrahli, J.A. Navío, M.C. Hidalgo, J. Naja, A. Chaqroune, A. Kherbeche
Comunicación Oral

ALBA II – Workshop on Catalysis: present and future perspective
19 julio [Online]

Potential of infrared synchrotron emission spectroscopy for catalysis research
L.F. Bobadilla Baladrón
Comunicación oral

XXIX International Materials Research Congress IMRC2021
15 – 20 agosto [Online, Cancún, México]

CO_2 valorisation in industrial plants. Methanation of flue gases in a stainless-steel mill plant
J.C. Navarro, F.M. Baena-Moreno, M.A. Centeno, O.H. Laguna, J.A. Odriozola
Keynote Invitada

Role of the Synthesis Method of ZnO Nanoparticles on the Photogeneration of Hydroxyl Radicals in the Photodegradation of Phenol
M. Uribe, M.C. Hidalgo, R. López, D. Frías, G. Núñez, D. Hernández, M. Álvarez-Lemus
Comunicación Oral

Power-to-X catalytic processes for CO_2 valorization
L. Azancot, L.F. Bobadilla, M.A. Centeno, J.A. Odriozola
Comunicación Oral

🟩 CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

PARTICIPACIÓN EN LA ORGANIZACIÓN DE CONGRESOS Y REUNIONES / PARTICIPATION IN ORGANISING CONGRESSES AND MEETINGS

1st Series of Low-Carbon Energy Talks
Programa de Doctorado Ciencias de Materiales, Universidad de Sevilla
[Online, Sevilla, España]

Tomás Ramírez Reina (Workshop Chairs/Organisers)
José Antonio Odriozola Gordón (Workshop Chairs/Organisers)

COMUNICACIONES / COMMUNICATIONS

Reunión de la Sociedad Española de Catálisis SECAT2021
18 – 20 octubre [Valencia, España]

Operando DRIFTS-MS study of methanol carbonylation over CuH-MOR catalysts: Sustainable production of acetic acid
L.A. Luque-Álvarez, L.F. Bobadilla, J.A. Odriozola
Comunicación oral

Catalizadores de paladio soportados en materiales preparados por descomposición térmica de melamina para las reacciones del uso de ácido fórmico como vector de H_2
L. Lobo, M. Martínez-Tejada, L.F. Bobadilla, S. Ivanova, M.A. Centeno
Póster

Mechanistic insights for designing optimal catalysts for low-temperature RWGS reaction: a step ahead on gas-phase CO_2 conversion
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Póster

Ni stabilised on complex structures for dry reforming of methane (DRM): Pyrochlores vs MOF
J.L.M. Espejo, J. Gándara-Loe, J.A. Odriozola, T.R. Reina, L. Pastor-Pérez
Póster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Estudio de la reacción de Sabatier para la valoración de CO_2 a partir de corrientes de flue gas**
Autor: Juan Carlos Navarro de Miguel
Directores: Óscar Hernando Laguna Espitia y José Antonio Odriozola Gordón
Centro: Universidad de Sevilla
Fecha Defensa: 1 de febrero de 2021

Título: **Diseño y preparación de catalizadores soportados en materiales carbonosos estructurados**
Autor: Nicolás Rodríguez Riaño
Directores: Yazmin Yaneth Agamez Pertuz y José Antonio Odriozola Gordón
Centro: Universidad Nacional de Colombia y Universidad de Sevilla
Fecha Defensa: 27 de abril de 2021

Título: **Reforming of biomass-derived stream: Evidence on an active Ni-O-K phase**
Autor: Lola Azancot Luque
Directores: José Antonio Odriozola Gordón y Luis F. Bobadilla Baladrón
Centro: Universidad de Sevilla
Fecha Defensa: 3 de noviembre de 2021

Título: **Estudio de la capa de oxidación formada a alta temperatura en un soporte de acero inoxidable ferrítico EN-14509 mediante microscopía electrónica de barrido**
Autor: Pablo Navarro Vicente
Directores: Juan Francisco Almagro Bello (ACERINOX) y José Antonio Odriozola Gordón
Centro: Universidad de Sevilla
Fecha Defensa: 10 de diciembre de 2021

Título: **Valorization of a platform chemicals issued from sugars, hydrogenation reactions over supported metal catalysts on functionalizaed carbon materials**
Autor: Charf Eddine Bounoukta
Directores: Fátima Ammari y Svetlana Ivanova
Centro: Université Ferhat Abbas – SETIF1 y Universidad de Sevilla
Fecha Defensa: 20 de diciembre de 2021

Formación de Graduados / Master Degree Thesis

Título: **Preparación de xerogeles de carbón conteniendo heteroátomos**
Autor: María Andrades García
Tutores: Leidy Marcela Martínez Tejada, Miguel Angel Centeno
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Fecha Defensa: 11 enero 2021

Título: **Composites de SBA-15/amina para la adsorción de CO_2**
Autor: Jesús Álvarez Yerpes
Tutores: Leidy Marcela Martínez Tejada, José Antonio Odriozola Gordón
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Fecha Defensa: 11 enero 2021

Título: **Ácido fórmico como vector energético: uso de catalizadores de paladio**
Autor: Laura Lobo Andrades
Tutores: Leidy Marcela Martínez Tejada, Miguel Angel Centeno
Grado: Trabajo Fin de Grado de Química
Centro: Universidad de Sevilla
Fecha Defensa: 22 julio 2021

Título: **Design and structuration of catalyst for energetic and environmental applications: Hydrotalcites as potential CO_2 absorbentes & Influence micro-channel configuration on the catalytic performance of a CeO_2/CuO_x catalyst for CO PROX reaction**
Autor: María José Vázquez Bernárdez
Tutores: María Isabel Domínguez Leal, Leidy Marcela Martínez Tejada, Corinne Petit (ECPM)
Grado: Trabajo de Stage 2éme Année, ECPM (Ecole Européenne d'ingénieurs de chimie, polymères et matériaux, Université de Strasbourg)
Centro: Université de Strasbourg, Universidad de Sevilla
Fecha Defensa: 22 julio 2021

Título: **Preparación de catalizadores estructurados activos en reacciones de biorrefinería**
Autor: Luis Alejandro Arriaga Arellano
Tutores: María Isabel Domínguez Leal, Leidy Marcela Martínez Tejada
Grado: Trabajo Fin de Grado de Ingeniería de Materiales
Centro: Universidad de Sevilla
Fecha Defensa: 22 julio 2021

Título: **Mechanistic insights for designing optimal catalysts for low-temperature RWGS reaction**
Autor: Guillermo Torres Sempere
Tutores: Tomás Ramírez Reina, Luis F. Bobadilla Baladrón
Grado: Trabajo Fin del Máster Ciencia y Tecnología de Materiales
Centro: Universidad de Sevilla
Fecha Defensa: 14 julio 2021

Título: **Catalytic Solutions for CO_2 conversion**
Autor: Sergio Carrasco Ruiz
Tutores: Tomás Ramírez Reina, Luis F. Bobadilla Baladrón
Grado: Trabajo Fin de Grado de Química
Centro: Universidad de Sevilla
Fecha Defensa: 22 julio 2021

Título: **Aplicaciones de Catalizadores Avanzados a Procesos de Conversion de CO_2 a fase gas**
Autor: Ignacio Galván Diez
Tutores: Laura Pastor Pérez, Esmeralda Portillo Estevez
Grado: Trabajo Fin de Grado de Ingeniería Química
Centro: Universidad de Sevilla
Fecha Defensa: 22 julio 2021

Título: **Recubrimientos hidrofóbicos para gafas**
Autor: Ignacio Redondo Andrade
Tutores: Leidy Marcela Martínez Tejada
Grado: Trabajo Fin del Doble Grado de Farmacia y Óptica y Optometría
Centro: Universidad de Sevilla
Fecha Defensa: 28 Julio 2021

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales y en titulaciones de Grado y doble Grado de la Universidad de Sevilla (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Molino de bolas de movimiento planetario PM100 de RETSCH
- Espectrofotómetro Infrarrojos: Thermo-Nicolet Nexus FT-IR; Thermo-Nicolet 380 FT-IR
- Accesorio DRIFTS, celdas de alta y baja temperatura
- Sistema de vacío cuarzo/vidrio para adsorción de moléculas sonda seguido por FT-IR
- Espectrómetro de masas: Balzers Thermostar
- Sistema TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas (Balzers) y detector de conductividad térmica. PID Eng&Tech.
- Equipos comerciales de actividad catalítica Microactivity Reference PID Eng&Tech (4)
- Microscopio metalográfico: Leica DMIRM
- Rotavapor: Heidolph Hei-VAP Value
- Equipo de ultra-alto vacío para espectroscopía XPS y Auger equipado con cañón de argón para realizar devastado iónico.
- Estufas: P-Selecta; P-Selecta digiheat
- Ph-metros: Crison pH burette 24; Crison phMeter Basic 20; Crison micropH2000.
- Cromatógrafos (2): Agilent Technologies 7890 A GC System; Agilent Technologies 6890 N Network GC System.
- Micro-cromatógragos, microGC (2): Micro Gas Cromatograph CP-4900 Varian (2)
- HPLC: Varian 356-LC, Solvent Delivery Module Varian ProStar.
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos: P-Selecta Ultrasons Medi-II
- Horno Energon
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis (Varian Cary 100, con esfera integradora para
- muestras sólidas)
- Analizador de Carbón Orgánico (TOC-V CHP Shimadzu 5000ª)
- Cromatógrafo (HPLC Agilent Technologies 1200)
- Espectrómetro IR (Varian 660-IR FTIR Spectrometer)
- Dip-Coater con cámara de temperatura (SS-00 AB Table Dry Oven MTI
- Corporation)
- Espectrofotómetro FTIR con celdas DRIFTS y ATR.
- Sistema de análisis TPR/TPO con detector TCD y espectrómetro de masas.

- 6 reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- 2 reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- 4 reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.
- Espectrofotómetros FTIR con accesorios DRIFTS, ATR y sistema de vacío en cuarzo/vidrio para adsorción de moléculas sonda
- Espectrómetros de masas
- Sistemas TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas y detector de conductividad térmica.
- Microscopio metalográfico
- Rotavapor
- Estufas
- Ph-metros
- Cromatógrafos de Gases
- Micro-cromatógragos, microGC
- Cromatografos HPLC
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos
- Hornos
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis, con esfera integradora para muestras sólidas)
- Analizador de Carbón Orgánico
- Dip-Coater con cámara de temperatura
- Reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- Reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- Reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS
ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

GRUPO DE INVESTIGACIÓN

Materiales de Diseño para la Energía y Medioambiente
Designed Materials for the Energy and Environment [642007]

PERSONAL / PERSONNEL

Catedráticos	Dr. Miguel Ángel Castro Arroyo
	Dr. Manuel Jiménez Melendo
	Dra. Pilar Malet Maenner
	Dr. Julián Martínez Fernández
Investigadores Científicos	Dra. María Dolores Alba Carranza
Científicos Titulares	Dr. José Jesús Benítez Jiménez
Profesores Titulares	Dr. Alfonso Bravo León
	Dr. Joaquín Ramírez Rico
Doctores Contratados	Dra. Esperanza Pavón González
Personal Investigador en Formación	Gdo. Javier Ramiro Chaparro Barajas
	Gda. Sol Fernández Muñoz

Proyectos de Investigación / Research Projects

Cerámicas Conductoras de Protones para Electrolizadores Reversibles de Alta Eficiencia y Aplicaciones Power to X	**Proton conducting ceramics for high efficiency reversible electrolyzers and power to X applications**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PID2019-107019RB-I00 "Retos de la Sociedad"
Periodo/Period:	01-06-2020 / 31-05-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	72.600 €
Investigador responsable/Research head:	Joaquín Ramírez Rico y Ricardo Chacartegui Ramírez
Componentes/Research group:	Alfonso Bravo León, Manuel Jiménez Melendo, Julián Martínez Fernández, Miguel Torres García

RESUMEN / ABSTRACT

PROCEX aborda el Reto social 3 "Energía limpia, eficiente y segura" y pretende abrir camino a una nueva generación de electrolizadores reversibles de alta eficiencia que operan a temperaturas intermedias (sobre 500°C). Su éxito abriría una vía muy prometedora para nuevos sistemas de almacenamiento de energía fotovoltaica y eólica con características sobresalientes, tales como eficiencias de ida y vuelta (superiores al 75%) o tasas de retorno energético (>10). Estos valores son muy superiores a los que se pueden alcanzar con los mejores sistemas de almacenamiento térmico. Además, el uso de electrolizadores de alta eficiencia encontraría un enorme campo de aplicación en la producción de H2 y en la industria química. Para desarrollar estos sistemas, es necesario superar numerosos retos en el ámbito de los materiales cerámicos: en particular, es necesario desarrollar nuevos electrolitos conductores de protones con baja conductividad electrónica.

El objetivo principal del proyecto es identificar, sintetizar y demostrar nuevos materiales cerámicos conductores de protones con bajas pérdidas electrónicas en electrólisis, usando estrategias de dopado en compuestos de circonatos y ceratos de bario. Ponemos énfasis no sólo en mejorar la eficiencia sino también en la durabilidad de estos materiales. El proyecto demostrará el procesado de los electrolitos y su integración en pilas tipo botón a escala laboratorio, y estudiará los principales mecanismos de reacción, desarrollando modelos que permitan predecir su comportamiento a gran escala. Este proyecto parte de resultados publicados muy recientemente en la literatura

y pretende superarlos apoyándose en las capacidades y experiencia previa del equipo investigador. En el proyecto ampliaremos el rango de composiciones y dopantes a estudiar y realizaremos un estudio sistemático que nos permita relacionar la composición y procesado con las propiedades y el rendimiento en condiciones de servicio (i.e. la degradación y el envejecimiento). A partir de la información obtenida pretendemos desarrollar y validar nuevos modelos que permitan evaluar la integración de estos sistemas en distintas aplicaciones. La ambición del este proyecto requiere un tratamiento multidisciplinar fruto de la combinación de dos grupos de investigación, uno de Ciencia de Materiales y otro de Ingeniería Energética, que poseen las capacidades e instalaciones necesarias para llevar a buen término el proyecto: síntesis y procesado de materiales, caracterización física, modelado numérico e integración de sistemas de almacenamiento de energía.

PROCEX is aimed at the Social Challenge 3 "Secure, Clean and Efficient Energy". It aims to open a new pathway for high-efficiency reversible electrolyzers for intermediate temperatures (around 500ºC). Its successful development would open a very promising pathway for energy storage systems in PV and Wind facilities with outstanding characteristics, round-trip efficiencies (75% or higher), and Energy Returned On Investment (>10). These values are much higher than those that can be reached with state of art of thermal energy storage systems. Besides, such a high efficiency concept electrolyzer would have a huge field of application for H2 production and application in the chemical industry. To develop such systems, several materials challenges need to be solved. In particular, novel electrolytes formulations with reduced electronic conductivities are needed.

The project is aimed at the identification and demonstration of new proton conducting ceramic materials that will have reduced electronic leakages in electrolysis operation, based on doping and co-doping strategies in barium cerate and zirconate systems. Emphasis will be placed not only in improving the efficiency but also the durability of such materials. The project will demonstrate the manufacturing of material and electrolyte at laboratory level and it will study the main reaction mechanisms developing models for their understanding and to support the pathways for concept application and scaling up. The project departs from results presented in literature this year that are fully aligned with capacities and previous experience of the participating R&D teams. The project will go further from these results extending the material compositions to develop, tailoring them to specific applications, widening the understanding of the reactions mechanisms and the effects of materials as well to the operation in the materials (i.e. degradation and aging effects). From this approach, within the project new models are expected to be developed and validated and the integration of the concept in different applications will be assessed. The ambition of the project requires a multidisciplinary approach that is developed by two R&D teams, from Material Science and Energy Engineering areas with all the capacities required for the successful development of the project: manufacturing, testing modelling and develop the new concepts and with expertise in materials processing and characterization, electrochemical models, and energy storage systems.

Materiales biomórficos para almacenamiento de energía	**Biomorphic materials for energy storage (BioMatStor)**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P20_011860 - PAIDI 2020
Periodo/Period:	05-10-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	106.550 €
Investigador responsable/Research head:	Joaquín Ramírez Rico
Componentes/Research group:	María Dolores Alba Carranza, Alfonso Bravo León, Manuel Jiménez Melendo, Esperanza Pavón González

RESUMEN / ABSTRACT

El objetivo principal de esta propuesta es desarrollar materiales de carbono a medida para aplicaciones relacionadas con tecnologías energéticas y medioambientales, con un enfoque en tres aplicaciones principales: almacenamiento de energía, soportes de catalizador en pilas de combustible y electrolizadores y el almacenamiento y captura de gas, especialmente hidrógeno y dióxido de carbono. Proponemos producir estos materiales mediante pirólisis de desecho de biomasa y otros residuos orgánicos. El uso de biomasa como precursor en la síntesis de materiales tiene interés dada su abundancia y bajo costo, y presenta una oportunidad para convertir los residuos de la industria agroalimentaria local en un producto de alto valor añadido.

BioMatStor desarrolla I + D en diferentes niveles de aplicación: fundamental para la caracterización y fabricación de la ciencia de los materiales, y ciencia aplicada para el modelado y caracterización de sistemas de almacenamiento de energía. Este proyecto combina ciencia de materiales e ingeniería energética con el objetivo de obtener materiales de alto rendimiento para una amplia gama de aplicaciones en la producción y almacenamiento de energía. Proponemos un enfoque multidisciplinar que tiene su base en la excelencia científica, responde a los desafíos sociales y puede resultar en una transferencia de tecnología significativa a la industria. Este proyecto también aborda los objetivos socio-estratégicos de Horizonte 2020, ya que tiene como objetivo contribuir a la mejora de nuestro entorno a través de la ciencia avanzada y la investigación multidisciplinar, y está totalmente alineado con los objetivos y políticas de la Unión Europea, Horizon2020, SET Plan y los objetivos RIS3 de la región de Andalucía.

Biomass derived carbon materials will play a key role in several energy conversion and storage technologies in the future, with application in supercapacitors and batteries,

power-to-X systems (fuel cells and electrolyzers), CO2 and H2 storage. Large amounts of biomass waste are generated in local agrofood industries. Among these wastes, the overall estimated production of olive stones in Spain is approximately 1,050,000–1,400,000 tons per year (campaign of 2017). The main use of this byproduct has been as solid biofuel for domestic applications, but given its abundance and low cost, this project presents an opportunity to convert what is considered waste into an added value product.

This proposal's main objective is to develop tailored carbon materials for applications related to energy and environmental technologies, with a focus on three main applications: i) electrochemical energy storage; ii) catalyst supports in fuel cells and electrolyzers; iii) and gas storage and capture, with a focus on both hydrogen and carbon dioxide storage and separation processes. The main proposed synthesis approach for these materials will be the pyrolysis of biomass precursors, with a focus on biomass waste products such as grain husks, peels, pits and stones and other organic waste. A first objective will be to perform a survey of readily available biomass waste materials from regional agrofood industries. A second objective will be the investigation and optimization of pyrolysis and activation routes to obtain carbon materials with tailored properties for each of the applications targeted in this project. Lastly, a third objective is to assess the applicability and the potential for the application of these materials at commercial scale.

Extensive physical and chemical characterization of the obtained carbon materials will be performed and testing of the resulting materials for the targeted applications will allow us to tailor the processing parameters. A scale-up analysis, with definition of materials integration and systems configurations will be performed by means of simulations, as well as technological and industrial applicability evaluation and assessment of the feasibility of the proposed approach in the large scale. BioMatStor develops R&D at different levels of application: fundamental for materials science characterization and manufacturing, and applied science for energy storage systems modeling and characterization. This Project combines Materials Science and Energy Engineering with the goal of obtaining highly performing materials for a wide range of applications in energy production and storage. Such a proposal requires a multidisciplinary approach, as evidenced in the research team and collaborators. We propose a multidisciplinary approach which has its foundation in scientific excellence, responds to societal challenges and may result in a significant technology transfer to the industry. This project also addresses the socio-strategic goals of Horizon 2020 as it aims to contribute to the improvement of our environment through advanced science and multidisciplinary research. It is fully aligned with the objectives and policies of European Union, the Energy Union Energy, H2020, SET Plan and Andalucía region RIS3 objectives.

Otros Proyectos / Other Projects

Aspectos genéticos y biofísicos de la formación de la cutícula del fruto de tomate

Código/Code:	RTI2018-094277-B-C22
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovacion y Universidades
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Eva María Domínguez Carmona (IHSM) y Rafael Fernández Muñoz (IHSM)
Participante del ICMS como investigador:	José Jesús Benítez

Convenios y Contratos / Contracts and Agreements

Development of a coating for fruits and vegetables derived from aleuritic acid

Periodo/Period:	26-12-2019 / 26-05-2021
Organismo Financiador/Financial source:	DECCO WORLDWIDE POST
Importe total/Total amount:	28.798 €
Investigador responsable/Research head:	José Jesús Benítez

Patentes / Patents

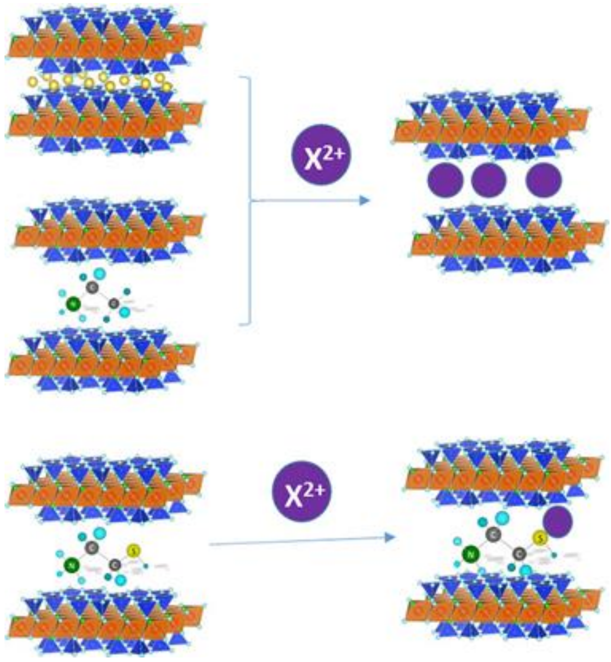
Sensor luminiscente para la monitorización de residuos radioactivos y uso del mismo

Inventores: Esperanza Pavón González, María Dolores Alba Carranza, María José García Jiménez
Tipo de Patente: Internacional
Número de Solicitud: PCT/ES21/070313
Fecha Solicitud: 6 de mayo de 2021
Entidades Titulares: Universidad de Cantabria, Consejo Superior de Investigaciones Científicas

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Pb^{2+}, Cd^{2+} and Hg^{2+} removal by designed functionalized swelling high-charged micas

Osuna, FJ; Pavón, E; Alba, MD
Science of The Total Environment, **764** (2021) 142811
Abril 2021 | **DOI:** 10.1016/j.scitotenv.2020.142811

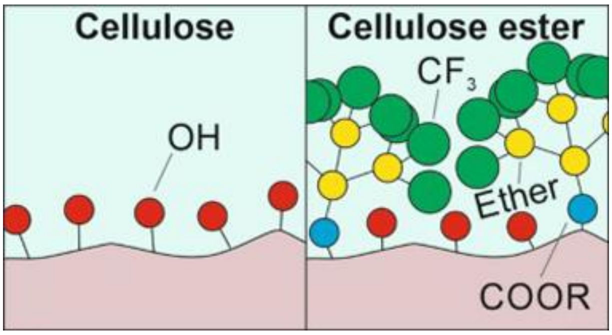


The increasing accumulation of toxic heavy metals in the environment has generated the need of efficient removal systems, being the adsorption method the most popular one applied in aqueous solutions. Of particular concern is the case of Pb^{2+}, Cd^{2+} and Hg^{2+} due to their high potential hazard. In this paper, we describe the feasibility of a new family of nanomaterials, swelling high charge micas, in the removal of these cations from aqueous solutions. Batch adsorption experiments were carried out in the as-made micas, NaM*n*, and after functionalization with ethylammonium, EA-Mn, and mercaptoethylammonium, MEA-M*n*. The results have demonstrated that all of them are efficient heavy metal adsorbents, being Na-M2 the best adsorbent for Pb^{2+} and Cd^{2+}, and, MEA-M2 for Hg^{2+}.

Waterproof-breathable films from multi-branched fluorinated cellulose esters

Tedeschi, G; Guzmán-Puyol, S; Ceseracciu, L; Benítez, JJ; Goldoni, L; Koschella, A; Heinze, T; Cavallo, G; Dichiarante, V; Terraneo, G; Athanassiou, A; Metrangolo, P; Heredia-Guerrero, JA
Carbohydrate Polymers, **271** (2021) 118031
Noviembre 2021 | **DOI:** 10.1016/j.carbpol.2021.118031

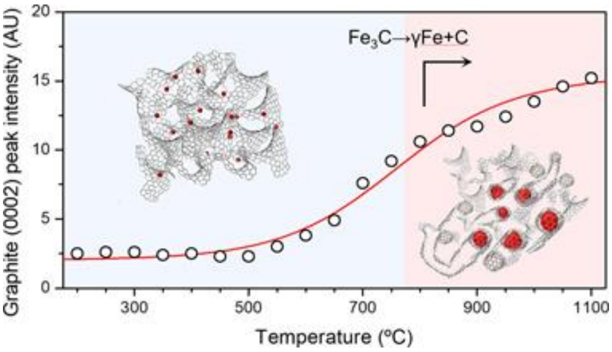
Cellulose ester films were prepared by esterification of cellulose with a multibranched fluorinated carboxylic acid, "BRFA" (BRanched Fluorinated Acid), at different anhydroglucose unit:BRFA



molar ratios (i.e., 1:0, 10:1, 5:1, and 1:1). Morphological and optical analyses showed that cellulose-BRFA materials at molar ratios 10:1 and 5:1 formed flat and transparent films, while the one at 1:1 M ratio formed rough and translucent films. Degrees of substitution (DS) of 0.06, 0.09, and 0.23 were calculated by NMR for the samples at molar ratios 10:1, 5:1, and 1:1, respectively. ATR-FTIR spectroscopy confirmed the esterification. DSC thermograms showed a single glass transition, typical of amorphous polymers, at -11 degrees C. The presence of BRFA groups shifted the mechanical behavior from rigid to ductile and soft with increasing DS. Wettability was similar to standard fluoropolymers such as PTFE and PVDF. Finally, breathability and water uptake were characterized and found comparable to materials typically used in textiles.

Structural Evolution in Iron-Catalyzed Graphitization of Hard Carbons

Gómez-Martín, A; Schnepp, Z; Ramírez-Rico, J
Chemistry of Materials, **33** (2021) 3087-3097
Mayo 2021 | **DOI:** 10.1021/acs.chemmater.0c04385

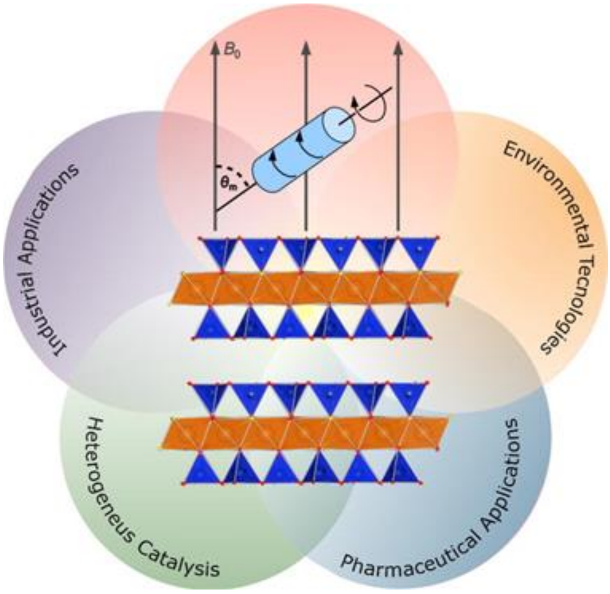


Despite the recent interest in catalytic graphitization to obtain graphite-like materials from hard-carbon sources, many aspects of its mechanism are still poorly unknown. We performed a series of in situ experiments to study phase transformations during graphitization of a hard-carbon precursor using an iron catalyst at temperatures up to 1100 °C and ex situ total scattering experiments up to 2000 °C to study the structural evolution of the resulting graphitized carbon. Our results show that upon heating and cooling, iron undergoes a series of reductions to form hematite, magnetite, and wustite before forming a carbide that later decomposes into metallic iron and additional graphite and that the graphitization fraction increases with increasing peak temperature. Structural development with temperature results in decreasing sheet curvature and increased stacking, along with a decrease in turbostratic disorder up to 1600 °C. Higher graphitization temperatures result in larger graphitic domains without further ordering of the graphene sheets. Our results have implications for the synthesis of novel biomass-derived carbon materials with enhanced crystallinity.

Swelling layered minerals applications: A solid state NMR overview

Pavón, E; Alba, MD

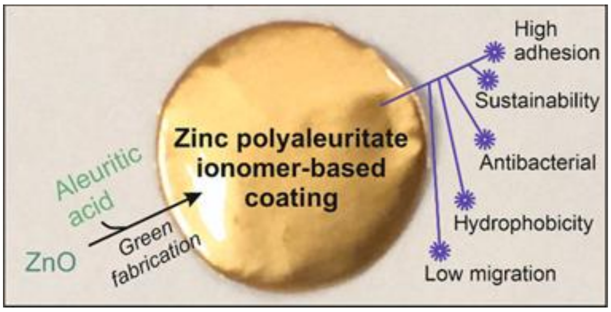
Progress in Nuclear Magnetic Resonance Spectroscopy, **124** (2021) 99-128
Junio 2021 | **DOI:** 10.1016/j.pnmrs.2021.04.001



Swelling layered clay minerals form an important sub-group of the phyllosilicate family. They are characterized by their ability to expand or contract in the presence or absence of water. This property makes them useful for a variety of applications, ranging from environmental technologies to heteroge-neous catalysis, and including pharmaceutical and industrial applications. Solid State Nuclear Magnetic Resonance (SS-NMR) has been extensively applied in the characterization of these materials, providing useful information on their dynamics and structure that is inaccessible using other characterization methods such as X-ray diffraction. In this review, we present the key contributions of SS-NMR to the understanding of the mechanisms that govern some of the main applications associated to swelling clay minerals. The article is divided in two parts. The first part presents SS-NMR conventional applications to layered clay minerals, while the second part comprises an in-depth review of the information that SS-NMR can provide about the different properties of swelling layered clay minerals.

Zinc Polyaleuritate Ionomer Coatings as a Sustainable, Alternative Technology for Bisphenol A-Free Metal Packaging

Morselli, D; Cataldi, P; Paul, UC; Ceseracciu, L; Benítez, JJ; Scarpellini, A; Guzmán-Puyol, S; Heredia, A; Valentini, P; Pompa, PP; Marrero-López, D; Athanassiou, A; Heredia-Guerrero, A
ACS Sustainable Chemistry & Engineering, **9** (2021) 15484-15495
Noviembre 2021 | **DOI:** 10.1021/acssuschemeng.1c04815

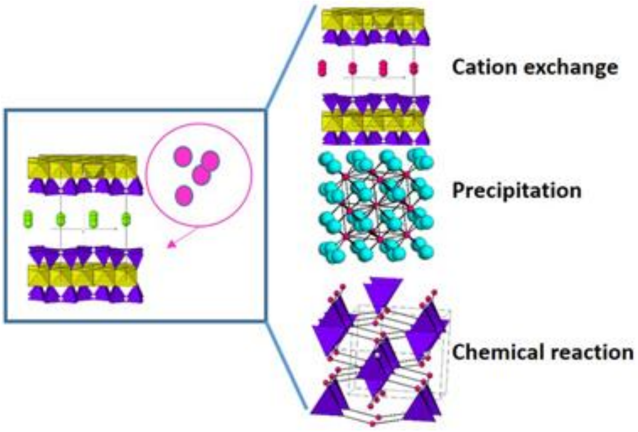


Sustainable coatings for metal food packaging were prepared from ZnO nanoparticles (obtained by the thermal decomposition of zinc acetate) and a naturally occurring polyhydroxylated fatty acid named aleuritic (or 9,10,16-trihydroxy-hexadecanoic) acid. Both components reacted, originating under specific conditions zinc polyaleuritate ionomers. The polymerization of aleuritic acid into polyaleuritate by a solvent-free, melt polycondensation reaction was investigated at different times (15, 30, 45, and 60 min), temperatures (140, 160, 180, and 200 °C), and proportions of zinc oxide and aleuritic acid (0:100, 5:95, 10:90, and 50:50, w/w). Kinetic rate constants calculated by infrared spectroscopy decreased with the amount of Zn due to the consumption of reactive carboxyl groups, while the activation energy of the polymerization decreased as a consequence of the catalyst effect of the metal. The adhesion and hardness of coatings were determined from scratch tests, obtaining values similar to robust polymers with high adherence. Water contact angles were typical of hydrophobic materials with values $\geq 94°$. Both mechanical properties and wettability were better than those of bisphenol A (BPA)-based resins and most likely are related to the low migration values determined using a hydrophilic food simulant. The presence of zinc provided a certain degree of antibacterial properties. The performance of the coatings against corrosion was studied by electrochemical impedance spectroscopy at different immersion times in an aqueous solution of NaCl. Considering the features of these biobased lacquers, they can be potential materials for bisphenol A-free metal packaging.

Zirconium retention for minimizing environmental risk: Role of counterion and clay mineral

Montes, L; Pavón, E; Cota, A; Alba
Chemosphere, **267** (2021) 128914
Marzo 2021 | **DOI:** 10.1016/j.chemosphere.2020.128914

Zr(IV) together with U(IV) are the major components of high-level radionuclide waste (HLRW) and spent nuclear fuel (SNF) from nuclear power plants. Thus, their retention in the waste disposal is of great importance for the environmental risk control. Here, the influence of clay



minerals on the retention of Zr(IV), as component of the nuclear waste and as chemical analogues of U(IV), has been evaluated. Three clay minerals, two bentonites and one saponite, were hydrothermally treated with three zirconium salts. A structural study at long-range order by X-ray diffraction and short-range order by NMR was performed to evaluate the generation of new zirconium phases and degradation of the clay minerals. Three immobilization mechanisms were observed: i) cation exchange of ZrO^{2+} or Zr^{4+} by clay minerals, ii) the precipitation/crystallization of ZrO_2, and, iii) the chemical interaction of zirconium with the clay minerals, with the formation of zirconium silicates.

Impact of flame confinement with inert ceramic foams on the particulate emissions of domestic heating systems

Ciria, D; Orihuela, MP; Becerra, JA; Chacartegui, R.; Ramirez-Ríco, J
Fuel, **304** (2021) 121264
Noviembre 2021 | **DOI:** 10.1016/j.fuel.2021.121264

Small solid biomass combustion systems are among the main contributors to the global particulate emissions share, and cheap, efficient abatement systems are not yet available for them. The placement of inert porous material to confine the combustion region is being recently explored as a possible mitigation system for this kind of pollution. However, given the complexity of biomass thermochemical decomposition processes, it is challenging to justify the performance of these systems on the basis of a physicochemical understanding. A foundational experiment-based study is carried out in this work to understand how combustion confinement affects the particulate emissions production mechanisms. A combustion unit was designed and built to systematically test ceramic foams with different porosities: keeping constant airflow and fuel feed rates. A comprehensive characterisation study was carried out on the solid biomass fuel, the temperature profile, the particulate emissions, and the remaining solid residue. The results evidenced that the use of foams has a substantial impact on the temperature distribution in the combustion chamber. The higher the cell density of the foam, the higher and more homogeneous are the temperatures reached in the combustion bed. This fact improved the thermal decomposition process of the pellets due to a better air-fuel mixture, leading to a reduction of the solid particulate matter emissions by more than 70%. These findings suggest that the use of an inert porous material above the combustion region might be a feasible solution for particulate emission control in small-size biomass combustion technology.

Pectin-cellulose nanocrystal biocomposites: Tuning of physical properties and biodegradability

Moreno, AG; Guzmán-Puyol, S; Domínguez, E; Benítez, JJ; Segado, P; Lauciello, S; Ceseracciu, L; Porrás-Vázquez, JM; León-Reina, L; Heredia, A; Heredia-Guerrero, JA

International Journal of Biological Macromolecules, **180** (2021) 709-717

Junio 2021 | **DOI:** 10.1016/j.ijbiomac.2021.03.126

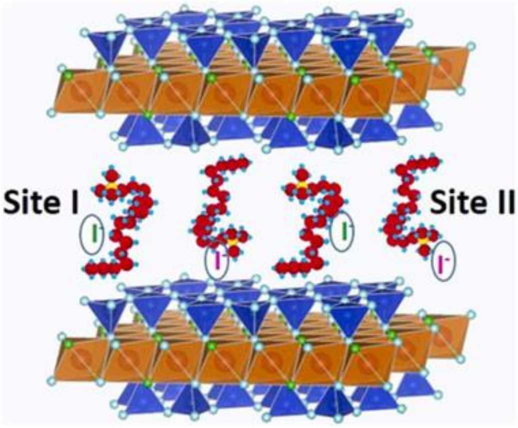
The fabrication of pectin-cellulose nanocrystal (CNC) biocomposites has been systematically investigated by blend-ing both polysaccharides at different relative concentrations. Circular free-standing films with a diameter of 9 cm were prepared by simple solution of these carbohydrates in water followed by drop-casting and solvent evaporation. The addition of pectin allows to finely tune the properties of the biocomposites. Textural characterization by AFM showed fibrous morphology and an increase in fiber diameter with pectin content. XRD analysis demonstrated that pectin incorporation also reduced the degree of crystallinity though no specific interaction between both poly-saccharides was detected, by ATR-FTIR spectroscopy. The optical properties of these biocomposites were character-ized for the first time and it was found that pectin in the blend reduced the reflectance of visible light and increased UV absorbance. Thermal stability, analyzed by TGA, was improved with the incorporation of pectin. Finally, pectin-cellulose nanocrystal biocomposites showed a good biodegradability in seawater, comparable to other common bioplastics such as cellulose and low-molecular weight polylactide, among others.

Designed organomicaceous materials for efficient adsorption of iodine

Osuna, FJ; Pavón, E; Pazos, MC; Alba, MD

Journal of Environmental Chemical Engineering, **9** (2021) 106577

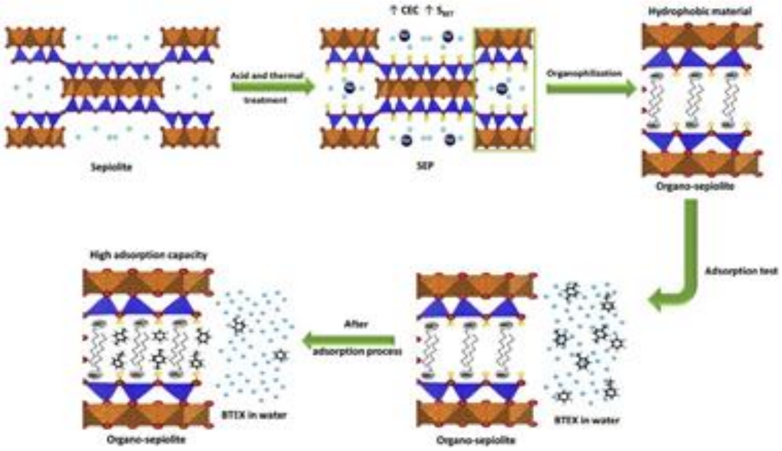
Diciembre 2021 | **DOI:** 10.1016/j.jece.2021.106577



The anionic iodine ^{129}I has a significant contribution to overall long-term dose resulting from the nuclear waste storage and its immobilization by clay barrier is crucial. Organoclays have been tested as ideal adsorption materials, being the clay layer charge and the length and type of organic molecules the most relevant parameters affecting the adsorption. In this work, a family of designed organomicas are explored in term of iodine adsorption capacity. Their adsorption capacities were always higher than that of the traditional clays and organoclays. C_{18}-M4 shows a maximum monolayer adsorption capacity one order of magnitude higher than natural organoclays, with a free energy typical of physical adsorption and adsorption sites of high affinity. However, its surface is not homogeneous in terms of stability constant according to the Scatchard adsorption parameters. Hence, this study can provide a guidance for the design and construction of ultrahigh-capacity iodine adsorbents.

Organophilization of acid and thermal treated sepiolite for its application in BTEX adsorption from aqueous solutions

Varela, CF; Pazos, MC; Alba, MD
Journal of Water Process Engineering, **40** (2021) 101949
Abril 2021 | **DOI:** 10.1016/j.jwpe.2021.101949



Acid and thermal treated sepiolite was organophilized by cationic exchange with several alkylammonium cations (octylammonium, hexadecylammonium, tetradecyltrimethylammonium, and hexadecyltrimethylammonium). The adsorption capacity of BTEX from aqueous solutions was evaluated through the adsorption isotherms performed in batch. The results were analysed using three isotherm models: Freundlich, Langmuir and Dubinin-Radushkevich (D-R model). The behaviour of adsorption isotherm suggested the multilayer coverage on a heterogeneous surface, which is according to the Freundlich isotherm model. The thermodynamic analyse using the D-R model show that physical mechanisms govern the process. The maximum adsorption capacity of BTEX on the obtained materials was in the range values of 81.19 mg g^{-1} - 1448.42 mg g^{-1}, which are higher than those reported up to now. The organo-sepiolite materials exhibit a high potential in the adsorption of BTEX compounds from aqueous solutions.

Mechanical Performances of Isolated Cuticles Along Tomato Fruit Growth and Ripening

Benítez, JJ; Guzmán-Puyol, S; Vilaplana, F; Heredia-Guerrero, JA; Domínguez, E; Heredia, A
Frontiers in Chemistry, **12** (2021) 787839
Diciembre 2021 | **DOI:** 10.3389/fpls.2021.787839

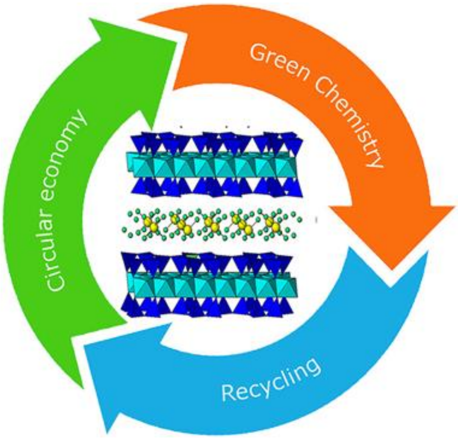
The cuticle is the most external layer that protects fruits from the environment and constitutes the first shield against physical impacts. The preservation of its mechanical integrity is essential to avoid the access to epidermal cell walls and to prevent mass loss and damage that affect the commercial quality of fruits. The rheology of the cuticle is also very important to respond to the size modification along fruit growth and to regulate the diffusion of molecules from and toward the atmosphere. The mechanical performance of cuticles is regulated by the amount and assembly of its components (mainly cutin, polysaccharides, and waxes). In tomato fruit cuticles, phenolics, a minor cuticle component, have been found to have a strong influence on their mechanical behavior. To fully characterize the biomechanics of tomato fruit cuticle, transient creep, uniaxial tests, and multi strain dynamic mechanical analysis (DMA) measurements have been carried out. Two well-differentiated stages have been identified. At early stages of growth, characterized by a low phenolic content, the cuticle displays a soft elastic behavior. Upon increased phenolic accumulation during ripening, a progressive stiffening is observed. The increment of viscoelasticity in ripe fruit cuticles has also been associated with the presence of these compounds. The transition from the soft elastic to the more rigid viscoelastic regime can be explained by the cooperative association of phenolics with both the cutin and the polysaccharide fractions.

By-products revaluation in the production of design micaceous materials

Mouchet, A; Raffin, F; Cota, A; Osuna, FJ; Pavón, E; Alba, MD

Aplied Clay Science, **214** (2021) 106292

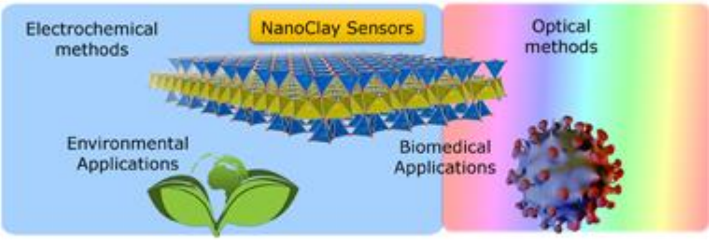
Noviembre 2021 | **DOI:** 10.1016/j.clay.2021.106292



One of the main objectives of a sustainable development and circular economy is the recycling of by-products generated in industrial and agricultural production processes. One of the possible solution is the use of such by-product materials in the synthesis of environmental adsorbents. In the current research, we present the synthesis of a high charge swelling mica with enhance adsorbent properties from blast furnace slag and rice husk ash. Moreover, to ensure the sustainable synthesis a natural bentoniteis used as Si and Al source. Thus, the current study investigated the fabrication of swelling high charged micas, Na-Mn (n (layer charge) = 2 or 4), from FEBEX bentonite, blast furnace slag and rice husk ash thorough the NaCl melt method. The reaction yield, cation framework distribution and structural characteristic of micas have been studied thorough X-ray Diffraction and Solid State Nuclear Magnetic Resonance. The yields of Na-Mn synthesis and degree of purity of the mica depends on the nature of these precursors. Thus, a sustainable, non-expensive and environmental friendly process has been evaluated.

New Trends in Nanoclay-Modified Sensors

Pavón, E; Martín-Rodríguez, R; Perdigón, AC; Alba, MD
Inorganics, **9** (2021) 43
Junio 2021 | **DOI:** 10.3390/inorganics9060043



Nanoclays are widespread materials characterized by a layered structure in the nano-scale range. They have multiple applications in diverse scientific and industrial areas, mainly due to their swelling capacity, cation exchange capacity, and plasticity. Due to the cation exchange capacity, nanoclays can serve as host matrices for the stabilization of several molecules and, thus, they can be used as sensors by incorporating electroactive ions, biomolecules as enzymes, or fluorescence probes. In this review, the most recent applications as bioanalyte sensors are addressed, focusing on two main detection systems: electrochemical and optical methods. Particularly, the application of electrochemical sensors with clay-modified electrodes (CLME) for pesticide detection is described. Moreover, recent advances of both electrochemical and optical sensors based on nanoclays for diverse bioanalytes' detection such as glucose, H_2O_2, organic acids, proteins, or bacteria are also discussed. As it can be seen from this review, nanoclays can become a key factor in sensors' development, creating an emerging technology for the detection of bioanalytes, with application in both environmental and biomedical fields.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

#LatinXChem Twitter Conference 2021
20 septiembre [México]

Obtención y caracterización de formas micro-mesoposas de una clinoptilolita natural y su empleo en la adsorción de colorantes
K. Borrego, L. Carrasco, R.I. Meneau, M.D. Alba, A. R. Ruiz-Salvador, T. Farías
Póster

Zeolita A jerarquizada para la remoción de antibióticos
R.I. Meneau, J.A. Millán, K. Borrego, M.B. Livia, M.D. Alba, T. Farías
Póster

TOPFULE 2021
6-10 junio [Santander, España]

Adsorption of Eu^{3+} in high-charge micas: dual functionality as radioactive waste encapsulation and in-situ luminescent sensor
R. Martín Rodríguez, A. Perdigón, F. Aguado, R. Valiente, M.D. Alba, E. Pavón
Póster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Nanoadsorbentes de diseño para la eliminación de contaminantes en agua / Designed nanoadsorbents for water remediation**
Autor:	Jonatan Guarino Suárez
Tutores:	Esperanza Pavón González, Joaquín Ramírez Rico
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Fecha Defensa:	12 diciembre 2021

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales y en titulaciones de Grado y doble Grado de la Universidad de Sevilla (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Horno tubular hasta 1200 °C
- Horno tubular en atmósfera controlada hasta 1700 °C
- Horno de Cámara hasta 1650 °C
- Horno de Cámara hasta 1200 °C
- Estufa hasta 400 °C

- Estufa hasta 200 °C
- Caja de guantes
- Reactores hidrotermales
- Línea de vacío
- 3 microscopios AFM
- Microscopio STM en aire
- Calorímetro diferencial de barrido (DSC)
- Analizador mecánico dinámico (DMA)
- Máquina de ensayos mecánicos por tracción
- Analizador de ángulo de contacto
- Balanza Langmuir-Blodgett (LB)
- Spin coater

MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES
MECHANOCHEMISTRY AND REACTIVITY OF MATERIALS

GRUPOS DE INVESTIGACIÓN

Materiales Avanzados
Advanced Materials [642010]

Propiedades Mecánicas, Modelización y Caracterización de Cerámicos Avanzados
Mechanical properties, Modelling and Characterization of Advanced Ceramics [642016]

Reactividad de Sólidos
Reactivity of Solids [642008]

PERSONAL / PERSONNEL

Profesores de Investigación	Dr. Luis Allan Pérez Maqueda
Catedráticos	Dr. Diego Gómez García
Investigadores Científicos	Dr. Francisco José Gotor Martínez
	Dra. Concepción Real Pérez
	Dr. Pedro José Sánchez Soto
Científicos Titulares	Dra. Rosalía Poyato Galán
	Dra. María Jesús Sayagués de Vega
Profesores Titulares	Dra. María Dolores Alcalá González
	Dra. María Regla Ayala Espinar
	Dr. Antonio Perejón Pazo
Talentia Senior CCAA	Dr. Pedro E. Sánchez Jiménez
Doctores Contratados	Dr. Juan Jesús Arcenegui Troya
	Dra. Eva Gil González
	Dr. Alejandro F. Manchón Gordón
	Dra. Virginia Moreno García
Personal Investigador en Formación	Ldo. Nabil Mohamed Amghar
	Gda. Sandra Molina Molina
	Gda. Mónica Salazar Lago
Personal Técnico Contratado	Gdo. Ahmed Taibi

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Integración de la tecnología "calcium looping" en plantas solares para el almacenamiento termoquímico de energía	**Solar Calcium-looping integRAtion for Thermo-Chemical Energy Storage**
	SOCRATCES
Código/Code:	727348
Periodo/Period:	01-01-2018 / 31-12-2021
Organismo Financiador/Financial source:	Comisión Europea. EUROPEAID
Importe total/Total amount:	223.500 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda
Componentes/Research group:	María Jesús Diánez Millán, Pedro Enrique Sánchez Jiménez

RESUMEN / ABSTRACT

El almacenamiento de energía es uno de los mayores desafíos para aumentar la viabilidad a corto plazo de las plantas de energía solar concentrada (CSP), que generalmente se caracterizan por su intermitencia en la producción de energía. El proceso Ca-Looping (CaL) basado en la reacción reversible de carbonatación/calcinación del CaO es una de las tecnologías más prometedoras para el almacenamiento termoquímico de energía (TCES). La amplia disponibilidad en la naturaleza de piedra caliza ($CaCO_3$) y su bajo precio (<10 € / ton) son factores clave para el despliegue comercial de la tecnología CaL.

SOCRATCES tiene como objetivo demostrar la viabilidad de la integración CSP-CaL mediante la construcción de una planta a escala piloto que utiliza materiales baratos, abundantes y no tóxicos, así como tecnologías actualmente en uso en la industria, como los reactores de lecho fluidizado y los intercambiadores de calor.

El objetivo global de SOCRATCES es desarrollar un prototipo que reduzca los riesgos a la hora de ampliar la tecnología CaL a escala global y resuelva cuestiones abiertas; comprender mejor y optimizar las eficiencias operativas que podrían obtenerse, con el objeto de habilitar plantas de CSP altamente competitivas y sostenibles.

Energy storage is one of the greatest challenges for a short-term deeper penetration of Concentrating Solar Power (CSP) plants, which are usually characterized by the intermittency of power production. The Ca-Looping (CaL) process based upon the reversible carbonation/calcination of CaO is one of the most promising technologies for thermochemical energy storage (TCES). The wide availability of natural limestone

(almost pure $CaCO_3$) and its low price (<10€/ton) are key factors for the feasibility of the CaL process.

SOCRATCES is aimed at demonstrating the feasibility of the CSP-CaL integration by erecting a pilot-scale plant that uses cheap, abundant and non-toxic materials as well as mature technologies used in the industry, such as fluidized bed reactor, cyclones or gas-solid heat exchangers.

SOCRATCES global objective is to develop a prototype that will reduce the core risks of scaling up the technology and solve challenges; further understanding and optimise the operating efficiencies that could be obtained; with the longer-term goal of enabling highly competitive and sustainable CSP plants.

Demostración en entorno relevante del uso de reacciones de calcinación-solar/carbonatación para el almacenamiento de energía térmica	**Validation in a relevant environment of solar-calcination/carbonation reactions for thermal energy storage**
MINISTERIO DE CIENCIA E INNOVACIÓN Financiado por la Unión Europea NextGenerationEU Plan de Recuperación, Transformación y Resiliencia AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PDC2021-121552-C21
Periodo/Period:	01-12-2021 / 30-11-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	68.770 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda, Pedro Enrique Sánchez Jiménez
Componentes/Research group:	Juan Jesús Arcenegui Troya, Antonio Perejón Pazo

RESUMEN / ABSTRACT

España es uno de los países europeos con mayor irradiación solar media y líder mundial en implantación de Energía Solar Concentrada (CSP). Una ventaja de la tecnología CSP es su capacidad de almacenar energía térmica y usarla cuando no hay irradiación. La plantas CSP de última generación incluyen sistemas de almacenamiento en sales fundidas (calor sensible) que presentan limitaciones: temperatura máxima limitada por degradación térmica, almacenaje a alta temperatura para evitar solidificación, corrosión y coste. En nuestro proyecto CTQ2017 se investigó el almacenamiento termoquímico mediante reacciones de calcinación/carbonatación, proceso calcium-lopping (CaL), usando caliza natural, que es abundante, barata, no corrosiva y permite operar a alta temperatura aumentando la eficiencia de conversión termoeléctrica. Su densidad energética (~1 MWhr/m^3) es superior al de las sales (0.25-0.40 MWhr/m^3). Un

problema del CaL para almacenamiento termoquímico es la desactivación del CaO con el número de ciclos. En nuestro proyecto CTQ2017 se propusieron diversas estrategias de mejora con las que se consiguieron rendimientos muy altos incluso después de muchos ciclos: (i) cambio de condiciones de calcinación-carbonatación (reducción de la temperatura de calcinación e incrementar la de carbonatación para mejorar el rendimiento tanto del proceso como de la planta) y (ii) propuesta de otros carbonatos diferentes de la caliza, uso de aditivos, uso de residuos (escorias) y materiales sintéticos de bajo coste. Estos resultados de laboratorio son de extraordinario interés para su aplicación a plantas de CSP, pero para su transferencia se requiere de validación en entorno relevante. En este proyecto se propone escalar los resultados de laboratorio mediante ensayos en planta piloto, el desarrollo y ensayo de un nuevo calcinador solar, así como la evaluación de la viabilidad técnico-económica de la tecnología a escala industrial. En este proyecto se desarrollará una prueba de concepto de un novedoso reactor/intercambiador de calor de tipo ciclón basado en energía solar. La radiación solar concentrada alcanzará el calcinador solar tipo ciclón mediante un sistema beam-down (concentrador solar secundario) desde el campo solar, formado por 14 heliostatos con una superficie total de 30 m^2 de la planta piloto construida en el marco del proyecto H2020 SOCRATCES, en el que han participado la mayor parte de los miembros del equipo de investigación del proyecto coordinado. El estudio y desarrollo de esta prueba de concepto permitirá establecer la viabilidad del diseño y demostrar su interés a empresas del sector energético y del cemento de cara a una futura integración de energía solar, en busca de una reducción de costes y emisiones de CO_2. Se parte de estudios a nivel de concepto desarrollados en el proyecto CTQ2017 con nivel de madurez tecnológica TRL 4, y se estima que se avanzará hasta niveles TRL 5-6. Se realizará un análisis de la viabilidad económica de la implantación de los nuevos conceptos propuestos en el marco del proyecto CTQ2017 y se elaborará un plan de transferencia. Este plan recogerá las acciones a llevar a cabo para favorecer una transferencia efectiva al sector industrial. Además, dado el potencial de patentabilidad de la tecnología objeto del proyecto, una vez probada en escala relevante (prueba de concepto), se desarrollará un plan de explotación y protección de derechos intelectuales.

Spain is one the European countries with the largest solar irradiation and world leader in concentrated solar power (CSP). A significant advantage of CSP technology is its ability to store thermal energy to be used when there is no irradiation. Last generation CSP plants include a storage system based on molten salts (Sensible Heat Storage) that show certain limitations: maximum temperature limited by thermal degradation, storage at high temperature to prevent solidification, corrosion, costs. In our CTQ2017 project we investigated on thermochemical energy storage by calcination (carbonation reactions, calcium looping (CaL) process, using limestone, which is abundant, cheap, non-corrosive, and allows high temperature operation, increasing the thermoelectric efficiency of the plant. Its energy density (~1 MWhr/m^3) is larger than that of salts (0.25-0.40 MWhr/m^3). A limitation of CaL for energy storage is the deactivation of CaO with the increasing number of cycles. In our project CTQ20, we proposed several improvement strategies for achieving high performance: (i) change of calcination/carbonation conditions (calcination temperature decrease and carbonation temperature increase) and (ii) proposal of other carbonates different from limestone, use of additives, use of waste materials (slags) and low-cost synthetic materials. These lab results are of great interest for its application in CSP, but it requires of validation in a relevant environment. In this project we propose the scale up of the lab results by tests in a pilot plant, the test of a new solar calcinator and the evaluation of the technical-economic feasibility of the technology on an industrial scale. Furthermore, a proof of concept of a novel solar power based cyclone type heat exchanger/reactor will be achieve within the project. The concentrated solar radiation will reach the cyclone-type solar calciner through a beam-down system (secondary solar concentrator) from the solar field, made up of 14 heliostats with a total area of 30 m^2 from the pilot plant built within the framework of the H2020 SOCRATCES project, in which most of the members of the research team of the coordinated project have participated. The study and development of this proof of concept will make it possible to establish the viability of the design and demonstrate their interest to companies in the energy and cement sectors with a view to a future integration of solar energy in search of a reduction in costs and CO_2 emissions. It is based on studies at the concept level developed in the CTQ2017 project with a level of technological maturity TRL 4, and it is estimated that it will advance to levels TRL 5-6. An analysis of the economic viability of the implementation of the new concepts proposed in the framework of the CTQ2017 project will be carried out and a transfer plan will be drawn up. This plan will include the actions to be carried out to favor an effective transfer to the industrial sector. In addition, given the potential for patentability of the technology object of the project, once tested on a relevant scale (proof of concept), a plan for the exploitation and protection of intellectual rights will be developed.

Integración del proceso Ca-looping en centrales de energía solar concentrada para el almacenamiento termo-químico de energía	**Integration of the Ca-looping process in concentrated solar power plants for thermochemical energy storage**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	CTQ2017-83602-C2-1-R
Periodo/Period:	01-01-2018 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	145.200 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda, Pedro Enrique Sánchez Jiménez
Componentes/Research group:	María Jesús Diánez Millán

RESUMEN / ABSTRACT

El proyecto solicitado se enmarca dentro del reto general de encontrar nuevas tecnologías de almacenamiento de energía baratas y no contaminantes que permitan superar una de las limitaciones mayores de las fuentes renovables que es la intermitencia en la generación de electricidad. En particular, en este proyecto se propone realizar una integración de la tecnología de Ca-looping en una planta termosolar de concentración. La tecnología de Ca-looping, originariamente propuesta para procesos de captura de CO_2, se basa en procesos de carbonatación-descarbonatación (o calcinación) de óxido de calcio-carbonato cálcico repetidos de forma cíclica. Nuestro grupo de investigación ha trabajado durante varios años en esta tecnología, con el objeto de comprender los mecanismos de desactivación conforme se incrementa el número de ciclos. Así, hemos estudiado los mecanismos cinéticos de estos procesos y los cambios microestructurales que tienen lugar a medida que se ciclan los compuestos. En un proyecto coordinado que concluye a finales de año (SOLARTEQH, Retos 2014) hemos realizado ya una propuesta de integración de Ca-Looping para almacenamiento de energía solar. Este proyecto ha dado lugar a una propuesta H2020 (SOCRATCES) aprobada y que comenzará a comienzos del año próximo. En el proyecto CALSOLAR que ahora presentamos se pretende avanzar más en esta idea de integración para incrementar los valores de eficiencia termoeléctrica. El subproyecto 1 realizará las tareas de coordinación de todo el proyecto. Además, en el subproyecto 1 se realizará la selección, preparación y caracterización de los compuestos a utilizar en el proyecto. En este sentido, se trabajará con empresas mineras que nos facilitarán distintas materias primas (principalmente calizas y dolomitas) con diverso grado de pureza y cristalinidad. Se prepararán compuestos con sílicas nanoestructuradas obtenidas a partir de cascarilla de arroz (suministradas por arroceras del valle del Guadalquivir). Se investigarán compuestos preparados a partir de escorias de acerías (suministrados por dos empresas del sector afincadas en Andalucía) que son ricos en calcio para su aplicación en ciclos de almacenamiento termoquímico. En el subproyecto, se diseñará y construirá un equipo termogravimétrico que permita realizar experimentos en las condiciones realistas de los ciclos de almacenamiento térmico. Así el equipo permitirá trabajar en condiciones de presión absoluta controlada de CO_2 y en vapor sobrecalentado. En dicho instrumento se realizarán los ciclos de almacenamiento y se estudiarán las condiciones óptimas de dichos ciclos. Se investigarán los mecanismos cinéticos de carbonatación y descarbonatación y se estudiarán los cambios microestructurales durante el ciclado. El equipo de investigación está compuesto por personal con gran experiencia en las tareas propuestas y se cuenta con la participación de investigadores extranjeros con gran experiencia en reacciones sólido-gas y en caracterización microestructural por microscopía de alta resolución. Además, participa una investigadora de la empresa Abengoa con extensa experiencia en almacenamiento termoquímico en plantas solares. Se trabajará en este subproyecto de forma totalmente coordinada con los investigadores del subproyecto 2 con idea de establecer conjuntamente las condiciones de proceso óptimas. Finalmente, los resultados obtenidos del proyecto podrán demostrarse en la planta piloto que se construye en el marco del proyecto H2020 SOCRATCES.

The proposal deals with the general social challenge of finding new cheap and environmentally friendly energy storage technologies to overcome the intermittency of energy generation from renewable sources. Particularly, in this project we propose integrating Ca-looping technology within a thermosolar concentration plant. Ca-Looping technology was originally proposed for CO_2 capture and it is based on cycled carbonation-calcination of calcium oxide-calcium carbonate. Our research group has been working on this technology for several years with the objective of understanding the deactivation mechanisms as the number of cycles increases. Thus, we have studied the kinetic mechanisms of these processes and the microstructural changes that takes place during cycling. In a coordinated project that is about to finish this year (SOLARTEQH, Retos 2014) where we already proposed the integration of Ca-Looping for thermosolar energy storage. This project was the basis of a H2020 proposal (SOCRATCES) that has been recently approved and that will start by the beginning of 2018. The project CALSOLAR is a step forward in the integration to increase the efficiency of the plant. Subproject I will coordinate the new project. Moreover, subproject I will select, prepare and characterize all compounds investigated in the project. We will work with mining companies that will provide the raw materials (mainly limestone and dolomite) with different purities and crystallinity. Composite materials with nanostructured silica obtained from rice husk (provided by rice mills from the Guadalquivir area) will be prepared. Compounds obtained from steel slags (supplied by nearby steel mills) rich in calcium will be prepared. Within subproject I, a new thermogravimetric instrument to perform thermal storage cycles under realistic conditions will be designed and constructed in our laboratories. This instrument should work under different controlled CO_2 pressures and under superheated steam. The kinetic mechanisms of carbonation and decarbonation and the microstructural changes will be investigated during cycling. The working team is experienced in the tasks of the project while some additional external scientists will participate. Thus, two foreign professors with solid backgrounds in solid-gas reactions and high resolution TEM are collaborating with us. Moreover, an industrial scientist from Abengoa with a very broad experience in thermal storage and thermosolar power plants is also included in the team. Both subprojects will work in a coordinated way with the aim of setting the optimum conditions for the final application. Finally, the results of the project will be directly applied to the pilot plant constructed within the H2020 SOCRATCES project.

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales	**Processing and Characterization of ceramic composites with two-dimensional laminar nanomaterials**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PGC2018-101377-B-I00
Periodo/Period:	01-01-2019 / 31-12-2022
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Ángela Gallardo López, Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora

RESUMEN / ABSTRACT

La incorporación de nanomateriales bidimensionales en materiales compuestos de matriz cerámica está aumentando en un esfuerzo para superar la inherente fragilidad de las cerámicas y para conferirles nuevas funcionalidades. Aún existen cuestiones abiertas en este campo en cuanto a resistencia mecánica, tenacidad a la fractura, cinética de crecimiento de fisuras, comportamiento tribológico, papel de fases interfaciales o idoneidad para el electromecanizado, entre otras. Aunque las nanoláminas de grafeno (NLG) se han mostrado como una segunda fase muy adecuada, los nanomateriales inorgánicos análogos al grafeno podrían extender el rango de aplicabilidad de los materiales con grafeno. El uso de nanoláminas de nitruro de boro (NLNB) en estos compuestos es muy prometedor y está prácticamente inexplorado.

Este proyecto propone un estudio sistemático de materiales compuestos con usos en aplicaciones estructurales y funcionales, con dos matrices de circona estabilizada con itria y dos tipos diferentes de nanomateriales 2D -nanoláminas de grafeno o de nitruro de boro- para profundizar en la comprensión de sus comportamientos mecánico y eléctrico. Con este fin, se fabricarán materiales con matrices de circona tetragonal dopada con 3 %mol de itria y circona cúbica dopada con itria, persiguiendo una microestuctura óptima con una distribución homogénea de los nanomateriales 2D en ambas matrices cerámicas. Se investigarán en profundidad materiales con NLG para dar respuesta a cuestiones abiertas en el conocimiento de estos materiales. La distribución, tamaño e integridad estructural de las NLG se caracterizarán mediante difracción de rayos X, microscopía electrónica de barrido y espectroscopía Raman, y las intercaras entre las NLG y la matriz se caracterizarán mediante microscopía electrónica de transmisión. La resistencia mecánica, resistencia a la rotura, mecanismos de refuerzo y cinética de crecimiento de grano en estos materiales se examinará en profundidad, y se

establecerá la mejor combinación de ruta de procesado y contenido de NLG en términos de refuerzo a la matriz. Se realizarán medidas de conductividad eléctrica en materiales con diferentes contenidos de NLG y se evaluará la respuesta al electromecanizado de los materiales eléctricamente conductores. Se realizarán también medidas de conductividad eléctrica en función de la temperatura para describir las posibles variaciones en el tipo de conducción al aumentar el contenido en NLG. Por otra parte, se investigarán materiales con NLNB con el objetivo de obtener una primera aproximación a la comprensión de este sistema. Con este fin, tras la síntesis de las NLNB usando una estrategia de mezcla de disolventes para la exfoliación en fase líquida de nanoláminas a partir de polvo de BN hexagonal, se prepararán polvos con diferentes contenidos de NLNB usando técnicas de procesado de polvo en medio húmedo. La caracterización microestructural de los materiales sinterizados mediante “Spark Plasma Sintering" se realizará mediante microscopía electrónica de barrido y de transmisión, difracción de rayos X y espectroscopía Raman. Se estudiarán propiedades como dureza, resistencia a la flexión y resistencia al desgaste, y se realizarán ensayos mecánicos a alta temperatura. Se analizará la conductividad eléctrica en función de la temperatura para esclarecer el efecto de la incorporación de una segunda fase aislante en las fronteras de grano sobre el comportamiento eléctrico de un conductor iónico.

Two-dimensional nanomaterials are being increasingly used as fillers in ceramic composites in an effort to overcome the inherent fragility of ceramics and to provide them with new functionalities. There are open issues in the field of these composites regarding their strength and fracture toughness mechanisms, crack growth kinetics, tribological behavior, role of interfacial phases or suitability for electrical discharge machining, among others. Although graphene nanosheets (GNS) are excellent fillers, inorganic graphene analogues could extend the range of applicability of graphene ceramic composites. The use of boron nitride nanosheets (BNNS) as fillers in ceramic composites is promising and practically unexplored.

This proposal outlines a systematic study of composites intended for use in structural and functional applications, with two different ceramic matrices from the yttria-stabilized zirconia system incorporating two different 2D laminar nanomaterials -graphene or boron nitride nanosheets-, to deepen in the understanding of their mechanical and electrical behavior. To that end, composites with 3 mol% yttria tetragonal zirconia and 8 mol% yttria cubic zirconia matrices will be fabricated, pursuing an optimum microstructure with a homogeneous distribution of the 2D nanomaterials throughout both ceramic matrices. On the one hand, ceramic composites with graphene nanosheets will be investigated in depth to complete the gaps in the current knowledge of these materials. The distribution, size and structural integrity of the GNS will be characterized by X-ray diffraction, scanning electron microscopy and Raman spectroscopy while the interfaces between the GNS and the matrix will be characterized by transmission electron microscopy. The strength, failure resistance, reinforcement mechanisms and crack growth kinetics of these composites will be thoroughly examined, and the best combination of processing route and GNS content in terms of reinforcement will be established. Electrical conductivity measurements of composites with different GNS contents will be carried out at room temperature and the response to electrical

discharge machining of the electrically conductive composites will be evaluated. Conductivity measurements will be carried out also as a function of temperature in order to describe the possible variations of conduction type when increasing the GNS content. On the other hand, ceramic composites with boron nitride nanosheets will be investigated in order to get a first approach to the understanding of this system. For this purpose, after the synthesis of the BN nanosheets using a mixed-solvent strategy for liquid exfoliation of BNNS from h-BN powder, composites with different contents of BNNS will be prepared using wet powder processing techniques. The microstructural characterization of the spark plasma sintered composites will be carried out by scanning and transmission electron microscopy, X-ray diffraction and Raman spectroscopy. Mechanical properties as hardness, flexural strength and wear resistance will be studied at room temperature, whereas deformation tests at high temperatures will be also performed. The electrical conductivity as a function of temperature will be analyzed in order to clarify the effect of incorporating an insulating second phase at the grain boundaries on the electrical performance of an ionic conductor.

Cerámicas en un Flash: La nueva ruta para un procesado energética y medioambientalmente eficiente	**Ceramics in a FLASH: The new route for environmentally efficient ceramic processing**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P18-FR-1087
Periodo/Period:	01-01-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	99.700 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda
Componentes/Research group:	María Jesús Diánez Millán, Pedro Enrique Sánchez Jiménez

RESUMEN / ABSTRACT

El proyecto CeramFLASH propone la utilización de las novedosas técnicas de Sinterizado Flash (FS) y Sinterizado Flash Reactivo (SFR) para la síntesis y preparación de cerámicas con interés tecnológico tales como electrolitos sólidos, piezoeléctricos o cerámicas duras electromecanizables. Estas técnicas permiten preparar materiales cerámicos en segundos a temperaturas significativamente más bajas que las requeridas por las técnicas de sinterizado convencional simplemente haciendo circular por la pieza una muy pequeña intensidad de corriente eléctrica (de unos pocos miliamperios). Esta

ventaja permite reducir de forma significativa el elevado consumo energético necesario en el procesado de materiales cerámicos.

Adicionalmente, se posibilita la preparación en forma densa y nanoestructurada de cerámicas muy difíciles de preparar mediante métodos convencionales, tales como compuestos de baja estabilidad térmica o compuestos que requieren temperaturas de sinterizado muy elevadas.

Finalmente, CeramFLASH pretende utilizar campos alternos con frecuencia de oscilación variable y métodos de control inteligente basados en la respuesta de la muestra al campo para conseguir un mejor control de las características microestructurales en las cerámicas resultantes. A pesar de que la técnica de FS se propuso por vez primera hace solo 8 años, y la SFR fue introducida en 2018 por nuestro grupo, el interés por este proceso está creciendo de forma importante por su gran potencial científico y tecnológico. CeramFLASH cuenta con la implicación de personal con experiencia en la técnica y la colaboración activa del investigador pionero en su propuesta, por lo que su financiación permitirá establecer una línea de investigación a largo plazo que permita consolidar en Andalucía un grupo de referencia a nivel internacional en este ámbito.

The CeramFLASH project proposes the novel ceramic processing techniques Flash Sintering (FS) and Reaction Flash Sintering (RFS) for the synthesis and preparation of ceramics with technological interest such as solid electrolytes, piezoelectric or hard ceramics. These techniques allow the preparation of ceramic materials in mere seconds at significantly lower temperatures than required by conventional sintering techniques simply by circulating a small electric current under moderate electric fields. This advantage makes it possible to reduce the considerable energy consumption required current ceramic processing techniques. Additionally, it facilitates the preparation of ceramics difficult to obtain in dense and nanostructured form by conventional methods, such as compounds of low thermal stability or compounds that require very high sintering temperatures.

Finally, CeramFLASH aims to use alternating fields with oscillation frequency as well as intelligent control methods based on the sample response to improve the control of microstructural characteristics of resulting ceramics. Although the FS technique was first discovered only 8 years ago, and the RFS was first proposed in 2018 by our group, there is rising interest in this process due to its great scientific and technological potential.

Nuevos materiales para el almacenamiento de Energía Solar Concentrada mediante Calcium-Looping (SOLACAL)	**New materials for energy storage of Concentrated Solar Power using Calcium-Looping (SOLACAL) processes**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	US-1262507
Periodo/Period:	01-02-2020 / 30-04-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	76.700 €
Investigador responsable/Research head:	Antonio Perejón Pazo y José Manuel Valverde Millán (US)
Componentes/Research group:	María Jesús Diánez Millán, Luis A. Pérez Maqueda, Virginia Moreno García

RESUMEN / ABSTRACT

En este proyecto se estudia el comportamiento de nuevos materiales basados en CaO durante ciclos de calcinación/carbonatación (Ca-Looping) en condiciones realistas de almacenamiento de energía en centrales de energía solar concentrada (CSP).

Para simular las condiciones realistas se utilizan equipos termogravimétricos capaces de emplear elevadas velocidades de calentamiento y enfriamiento y diferentes atmósferas de gases. De esta forma, los resultados obtenidos son realmente representativos y podrán ser extrapolados a las condiciones prácticas de operación en plantas CSP.

Se estudia la reactividad multicíclica de muestras de caliza y dolomita, a las que se les realizan tratamientos mecánicos y con ácido acético que pueden mejorar su actividad. Además, se ha demostrado que la presencia de MgO en la dolomita calcinada estabiliza térmicamente el CaO, por lo que se preparan dolomitas sintéticas con distinto contenido en MgO mediante tratamientos mecánicos y coprecipitación con el objeto de encontrar la cantidad óptima de MgO que mejore la actividad multicíclica del CaO. Se estudian asimismo otros materiales con los que se pueda aumentar la temperatura de carbonatación, como el $SrCO_3$ y el $BaCO_3$, lo que permitiría incrementar aún más la eficiencia termoeléctrica de las plantas CSP con almacenamiento termoquímico.

Un aspecto relevante de SOLACAL es que los resultados obtenidos serán transferidos de manera directa a la planta CSP-CaL de demostración que se está construyendo en Sevilla dentro del proyecto H2020 SOCRATCES iniciado en 2018 y coordinado por la Universidad de Sevilla.

This project is focused on the performance of new CaO-based materials during calcination/carbonation cycles (Ca-Looping) under realistic energy storage conditions in concentrated solar power plants (CSP).

In order to simulate realistic conditions, thermogravimetric instruments are used, which are able of employing high heating and cooling rates and different atmospheres of gases. In this way, the results obtained are truly representative and can be extrapolated to practical operating conditions in CSP plants. The multicycle reactivity of limestone and dolomite samples is studied. These samples are modified by mechanical and acetic acid treatments that can improve their reactivity. Moreover, it has been shown that the presence of MgO in calcined dolomite thermally stabilizes CaO. Synthetic dolomites with different MgO content are prepared by mechanical treatments and co-precipitation in order to find the optimal amount of MgO that improves the multicycle activity of CaO. Other materials in which the carbonation temperature can be increased, such as SrCO3 and BaCO3, are also studied, which would further increase the thermoelectric efficiency of CSP plants with thermochemical energy storage.

A relevant aspect of SOLACAL is that the results obtained will be transferred directly to the CSP-CaL demonstration plant that is being built in Seville within the H2020 SOCRATCES project, started in 2018 and coordinated by the University of Seville.

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de Cerámicas Avanzadas con Nanomateriales 2D para su Aplicación en Sistemas de Propulsión y Frenado en la Industria Aeroespacial (AEROCER-2D)

Código/Code:	PY20_01024
Periodo/Period:	05-10-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	60.125 €
Investigador responsable/Research head:	Ángel Gallardo López (US)
Participante del ICMS como investigador:	Rosalía Poyato Galán

Diseño y selección de materiales novedosos para fabricar pilas de combustible de óxido sólido de alto rendimiento

Código/Code:	US-15382
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	Francisco J. García García (US)
Participante del ICMS como investigador:	Francisco J. Gotor Martínez

Fabricación y caracterización de cilindros con gradiente de porosidad longitudinal mediante congelación dirigida, modificación superficial e infiltración con un compuesto de quitosano y bio-vidrios para sustitución de tejido óseo

Código/Code: P20_00671
Periodo/Period: 05-10-2021 / 31-12-2022
Organismo Financiador/Financial source: Junta de Andalucía
Importe total/Total amount: 43.500 €
Investigador responsable/Research head: Yadir Torres Hernández (US)
Participante del ICMS como investigador: Francisco J. Gotor Martínez

Aplicando la economía circular en el desarrollo de nuevos conglomerantes hidráulicos activados alcalinamente de baja huella de carbono para soluciones constructivas (CongActiva)

Código/Code: PID2020-11516RB-I00
Periodo/Period: 01-09-2021 / 31-08-2024
Organismo Financiador/Financial source: Ministerio de Ciencia e Innovación
Importe total/Total amount: 108.900 €
Investigador responsable/Research head: D. Eliche-Quesada (Universidad de Jaén)
Participante del ICMS como investigador: Pedro José Sánchez Soto

A la Búsqueda de Nuevos Cerámicos Ultraduros a Base de Boro para Aplicaciones Estructurales en la Nueva Generación de Aviones y uso Seguro y Eficiente de la Energía

Código/Code: PID2019-103847RJ-I00
Periodo/Period: 01-10-2020 / 30-09-2023
Organismo Financiador/Financial source: Ministerio de Ciencia e Innovación
Investigador responsable/Research head: Bibi Malmal Moshtaghion Entezari
Participante del ICMS como investigador: Diego Gómez García

Sinterización FLASH Reactiva para la preparación de compuestos inestables y resistentes a la densificación

Código/Code: 201960E092
Periodo/Period: 01-10-2019 / 30-09-2022
Organismo Financiador/Financial source: CSIC (Intramural)
Importe total/Total amount: 350.000 €
Investigador responsable/Research head: Luis Allan Pérez Maqueda

Síntesis y procesado de cerámicas avanzadas mediante Sinterizado FLASH
Código/Code: 2020601004
Periodo/Period: 02-10-2020 / 01-10-2021
Organismo Financiador/Financial source: CSIC (Ayudas para la incorporación de personal investigador a las escalas científicas del CSIC)
Importe total/Total amount: 5.000 €
Investigador responsable/Research head: Pedro Enrique Sánchez Jiménez

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Equipo de Análisis Térmico de Altas Prestaciones (EQC2019-005791-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 112.379,78 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Reactividad de Sólidos"

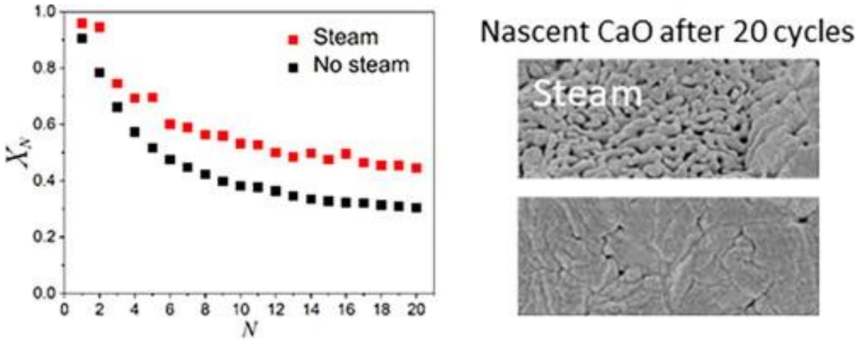
PATENTES / PATENTS

Procedimiento de preparación de materiales cerámicos, biocerámicos y refractarios de carácter básico con alto contenido en forsterita mediante molienda reactiva
Inventores: P.J. Sánchez Soto, L. Pérez Villarejo, D. Eliche Quesada, B. Carrasco Hurtado, S. Martínez Martínez, E. Garzón Garzón
Tipo de Patente: Nacional
Número de Solicitud: 202130248
Fecha Solicitud: 19 de marzo de 2021
Entidad Titular: Universidad de Jaén, Universidad de Almería, Consejo Superior de Investigaciones Científicas

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Kinetics and cyclability of limestone ($CaCO_3$) in presence of steam during calcination in the CaL scheme for thermochemical energy storage

Arcenegui-Troya, J; Sánchez-Jiménez, PE; Perejón, A; Moreno, V; Valverde, JM; Pérez-Maqueda, LA
Chemical Engineering Journal, **417** (2021) 129194
Agosto 2021 | **DOI:** 10.1016/j.cej.2021.129194

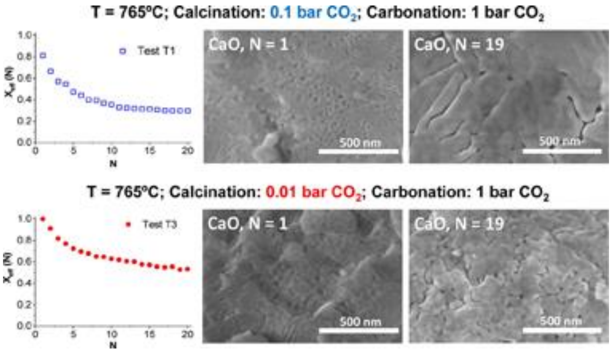


In the present work, we explore the use of steam in the $CaCO_3$ calcination step of the Calcium Looping process devised for thermochemical energy storage (CaL-TCES). Steam produces a double benefit: firstly, it fastens calcination, allowing a reduction of the temperature needed to attain full calcination in short residence times, as those required in practice, resulting in energy savings. This behaviour is justified on the basis of a kinetics study results obtained from a non-parametric kinetic analysis, which demonstrate that the presence of steam during calcination can reduce the apparent activation energy from 175 kJ/mol to 142 kJ/mol with a steam's partial pressure of 29%. In addition, the results obtained for multicycle CaL-TCES tests show that steam alleviates the deactivation of the sorbent, which is one of the main limiting factors of this technology. This behaviour is explained in terms of the effect of steam on the microstructure of the regenerated CaO. Importantly, the values of residual conversion attained by calcining in steam are higher than those without steam.

Calcination under low CO_2 pressure enhances the calcium Looping performance of limestone for thermochemical energy storage

Sarrión, B; Perejón, A; Sánchez-Jiménez, PE; Amghar, N; Chacartegui, R; Valverde, JM; Pérez-Maqueda, LA
Chemical Engineering Journal, **417** (2021) 127922
Agosto 2021 | **DOI:** 10.1016/j.cej.2020.127922

The Calcium Looping performance of limestone for thermochemical energy storage has been investigated under novel favorable conditions, which involve calcination at moderate temperatures under CO_2 at low pressure (0.01 and 0.1 bar) and carbonation at high temperature under CO_2 at atmospheric pressure. Calcining at low CO_2 pressures allows to substantially reduce the temperature to achieve full calcination in short residence times. Moreover, it notably enhances CaO multicycle conversion. The highest values of conversion are obtained for limestone samples calcined under 0.01 bar CO_2 at 765 °C. Under these conditions, the residual conversion



is increased by a factor of 10 as compared to conditions involving calcination under CO_2 at atmospheric pressure. The enhancement of CaO conversion is correlated to the microstructure of the CaO samples obtained after calcination. As seen from SEM, BET surface and XRD analysis, calcination under low CO_2 pressure leads to a remarkable decrease of pore volume and CaO crystallite size. Consequently, CaO surface area available for carbonation in the fast reaction-controlled regime and therefore reactivity in short residence times is promoted.

Advanced parametrisation of phase change materials through kinetic approach

Lizana, J; Perejón, A; Sánchez-Jiménez, PE; Pérez-Maqueda, LA
Journal of Energy Storage, **44** (2021) 103441
Diciembre 2021 | **DOI:** 10.1016/j.est.2021.103441

Phase change materials (PCM) have been widely investigated for heat storage and transfer applications. Numerous numerical simulation approaches have been proposed for modelling their behaviour and predicting their performance in thermal applications. However, simulation approaches do not consider the kinetics of the phase transition processes, compromising the accuracy of their predictions. The phase change is a kinetically driven process in which both the reaction rate and the reaction progress depend on the heating schedule. This work evaluates and parametrises the influence of kinetics in the melting and crystallisation behaviour of a well-known PCM, PEG1500, and compares potential discrepancies with common phase change parametrisation alternatives. The kinetic dependence was experimentally evaluated through differential scanning calorimetry (DSC). The kinetic parameters required for modelling the kinetics of the processes were determined by both model-free and model-fitting procedures following ICTAC (International Confederation for Thermal Analysis and Calorimetry) recommendations. Then, the phase transition was parametrised through a kinetic model and compared with three conventional phase transition models: linear without hysteresis, non-linear without hysteresis, and non-linear with hysteresis. The statistical comparison between models demonstrates the higher accuracy of the kinetic approach to correctly represent the partial enthalpy distribution of latent heat storage materials during alternative phase change rates, obtaining a coefficient of determination (R^2) of 0.80. On the other hand, the accuracy of kinetic-independent models is limited to the range from 0.40 to 0.61. The results highlight the high discrepancies of conventional models compared to the kinetic approach and provide criteria and guidelines for efficient kinetic modelling of phase change in heat transfer evaluations.

Geopolymers made from metakaolin sources, partially replaced by Spanish clays and biomass bottom ash

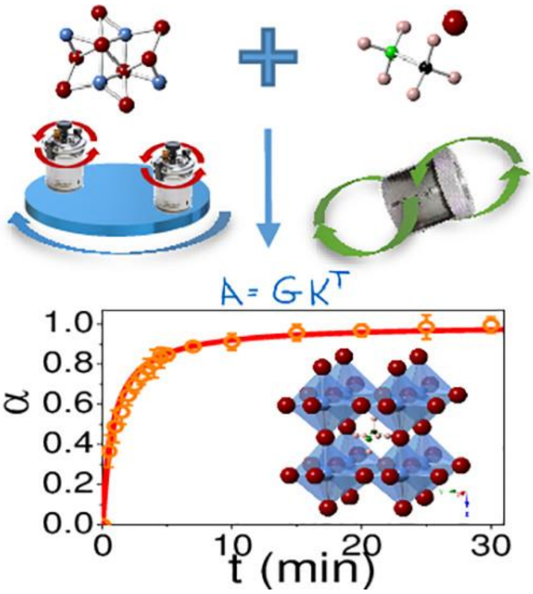
Eliche-Quesada, D; Calero-Rodríguez, A; Bonet-Martínez, E;Pérez-Villarejo, L; Sánchez-Soto, PJ
Journal of Building Engineering, **40** (2021) 102761
Agosto 2021 | **DOI:** 10.1016/j.jobe.2021.102761

The main objective of this investigation is to study the effect of the substitution of metakaolin (MK) (from calcined industrial kaolin) by four different calcined natural Southern Spain clays traditionally used in the brick and tile sector, as well as by the biomass bottom ash residue (BBA) from the combustion of a mix of olive and pine pruning on the synthesis of geopolymer with physical, mechanical and thermal properties comparable to those of classic construction materials. As alkaline activator, a 8 M solution of sodium hydroxide and sodium silicate have been used. Raw materials, metakaolin; Spanish clays: black clay (BC), yellow clay (YC), white clay (WC), red clay (RC) and BBA were characterized by chemical analysis (XRF), mineralogical analysis (XRD), and particle size analysis. Control geopolymers containing only metakaolin, and batch of geopolymers were formulated containing equal proportions of metakaolin, BBA and each of the four types of clay. After the curing period, at 60 degrees C for 1 day geopolymers were demolded and stored 27 days at room temperature. Geopolymers were characterized using Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS), XRD and Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR). Their physical, mechanical and thermal properties have also been studied. The addition of BBA and different types of calcined clays to metakaolin gives rise to geopolymers with higher mechanical properties increasing the compressive strength of the control geopolymer containing only MK (24.9 MPa) by more than 50% for the GMK-BBA-WC geopolymers (38.5 MP a). The clays act as fillers and/or promote the precipitation of calcium-rich phases (Ca)-A-S-H-G gel that coexists with the (Na)-A-S-H gel type. The relevant results of physical, mechanical and thermal properties obtained in this research demonstrate the potential of Spanish clays and BBA as binders and substitutes for metakaolin.

Paving the Way to Establish Protocols: Modeling and Predicting Mechanochemical Reactions

Gil-González, E; Pérez-Maqueda, LA; Sánchez-Jiménez, PE; Perejón, A
Journal of Physical Chemistry Letters, **12** (2021) 5540-5546
Junio 2021 | **DOI:** 10.1021/acs.jpclett.1c01472

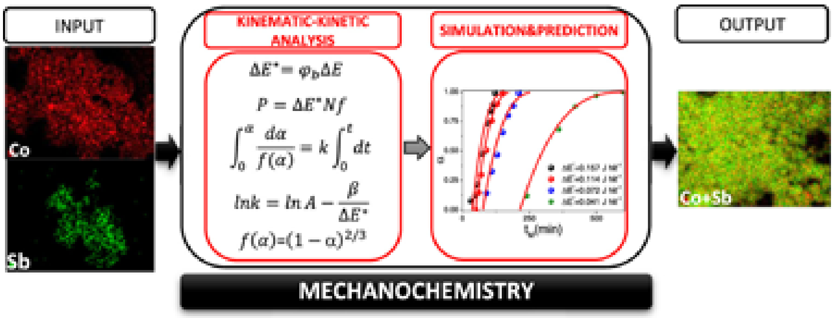
Parametrization of mechanochemical reactions, or relating the evolution of the reaction progress to the supplied input power, is required both to establish protocols and to gain insight into mechanochemical reactions. Thus, results could be compared, replicated, or scaled up even under different milling conditions, enlarging the domains of application of mechanochemistry. Here, we propose a procedure that allows the parametrization of mechanochemical reactions as a function of the supplied input power from the direct analysis of the milling experiments in a model-free approach, where neither the kinetic model function nor the rate constant equation are previously assumed. This procedure has been successfully tested with the mechanochemical reaction of $CH_3NH_3PbCl_3$, enabling the possibility to make predictions regardless of the milling device as well as gaining insight into the reaction dynamic. This methodology can work for any other mechanical reaction and definitely paves the way to establish mechanochemistry as a standard synthetic procedure.



Unveiling mechanochemistry: Kinematic-kinetic approach for the prediction of mechanically induced reactions

Gil-González, E; Rodríguez-Laguna, MdR; Sánchez-Jiménez, PE; Perejón, A; Pérez-Maqueda, LA

Journal of Alloys and Compounds, **866** (2021) 158925
Junio 2021 | **DOI:** 10.1016/j.jallcom.2021.158925



Mechanochemistry has attracted a lot of attention over the last few decades with a rapid growth in the number of publications due to its unique features. However, very little is known about how mechanical energy is converted into chemical energy. Most of the published works using mechanochemistry neglect the required attention to the experimental parameters and their effect over the resulting products, what makes extremely difficult to reproduce the results from lab to lab. Moreover, if it is taken into consideration the broad range of experimental conditions used in different studies, it is quite difficult to compare results and set optimum conditions. As a result, mechanochemistry is generally viewed as a "black box". The aim of this work is to provide some insight into mechanochemistry. Thus, a simple kinematic-kinetic approach that allows the full parametrization of mechanically induced reactions is proposed. In an analogous way to thermally activated process, it is shown that kinetic modeling can serve to parametrize and model mechanically induced reactions as a function of the milling parameters with great reliability, thereby gaining prediction capability. As a way of example, this methodology has been applied for the first time to the mechanochemical reaction of Co and Sb to form $CoSb_3$, a skutterudite-type thermoelectric material. Moreover, the universality of this methodology has also been validated with data from the literature. A key feature of the proposed kinematic-kinetic approach is that it can be extrapolated to other mechanically induced reactions, either inorganic or organic.

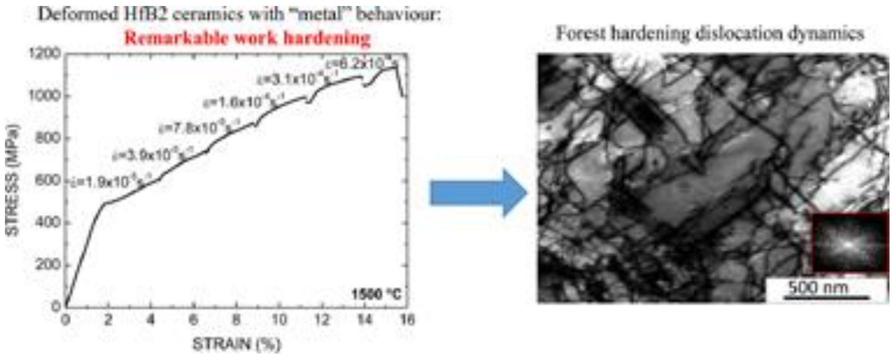
Unravelling the optimization of few-layer graphene crystallinity and electrical conductivity in ceramic composites by Raman spectroscopy

Muñoz-Ferreiro, C; López-Pernía, C; Gallardo-López, A; Poyato, R
Journal of the European Ceramic Society, **41** (2021) 290-298
Diciembre 2021 | **DOI:** 10.1016/j.jeurceramsoc.2021.09.025

Zirconia composites with few-layer graphene (FLG) were prepared by two powder processing routines-ultrasonic agitation or planetary ball milling-and spark plasma sintered at 1250 and 1300 °C. An in-depth study of the crystallinity of FLG, in terms of presence and nature of defects, was performed by Raman spectroscopy, revealing enhanced FLG crystallinity after sintering. This enhancement was more noticeable in the composites sintered at the highest temperature, with lower amount of structural defects and amorphous carbon. However, remaining amorphous carbon was detected in the composites prepared by planetary ball milling even after sintering at the highest temperature, resulting in lower electrical conductivities. Optimum results in terms of electrical conductivity were achieved for the composites prepared by ultrasonic agitation and sintered at 1300 °C, with electrical percolation limit below 2.5 vol% FLG and high electrical conductivity (678 S/m for 5 vol% FLG), as result of the enhanced FLG crystallinity after sintering.

HfB_2 ceramic polycrystals: A low-temperature metal-like ceramic at high temperatures?

Zapata-Solvas, E; Moshtaghioun, BM; Gómez-García, D; Domínguez-Rodríguez, A; Lee, WE
Scripta Materialia, **203** (2021) 114037
Octubre 2021 | **DOI:** 10.1016/j.scriptamat.2021.114037



Hafnium diboride (HfB_2) is a highly refractory (melting above 3000 °C) ceramic with many potential applications at high temperatures. To enable its use at temperature for extended periods its high-temperature plasticity must be known. This paper examines the mechanical response at temperatures between 900 °C and 2000 °C in air and in a reducing atmosphere, interpreting the data in the frame of classical models for the plasticity of compact-packed metals at low temperatures. In particular, the Friedel law and the principle of similitude for dislocation patterning are assessed. This reveals that HfB_2 is a singular example of a ceramic material with "metal" mechanical behaviour.

Synthesis of clay geopolymers using olive pomace fly ash as an alternative activator. Influence of the additional commercial alkaline activator used

Gómez-Casero, MA; Moral-Moral, FJ; Pérez-Villarejo, L; Sánchez-Soto, PJ; Eliche-Quesada, D
Journal of Materials Research and Technology, **12** (2021) 1762-1776
Mayo 2021 | **DOI:** 10.1016/j.jmrt.2021.03.102

In this research, the use of olive pomace fly ash (OPFA) as an alkaline source for the activation of calcined clays (CC) from Bailen (Jaen, Spain) was studied. The optimal composition was obtained for 70 wt % CC and 30 wt % OPFA. The physical, mechanical and thermal properties of control geopolymers that use water as a liquid medium have been studied and compared with geopolymers that use additional activating solutions as sodium or potassium hydroxide solutions (8 M), or a mixture of alkaline hydroxide and alkaline silicate solution ($NaOH$-Na_2SiO_3 or KOH-K_2SiO_3). The results showed that OPFA can be used as an alkaline activator, showing mechanical properties slightly lower than those obtained when additional alkaline hydroxide activating solutions were used. The best compressive strength was obtained for geopolymers that use alkaline silicates as an activating solution. However, the best thermal insulation properties were obtained for control geopolymers. The microstructural characteristics of the geopolymers were evaluated by means of X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM-EDS) that corroborate the formation of geopolymeric gel in all the specimens, being the amount of gel formed greater in samples using commercial potassium activating solutions. These results demonstrate the feasibility of using this type of waste, OPFA, as activating reagents in the manufacture of geopolymers or alkaline activated materials. The manufactured geopolymers can be used as compressed earth blocks for walls and partitions, since the specimens pursue mechanical properties that comply with current regulations, presenting better thermal insulation properties.

A Novel, Simple and Highly Efficient Route to Obtain $PrBaMn_2O_{5+\delta}$ Double Perovskite: Mechanochemical Synthesis

García-García, FJ; Sayagués, MJ; Gotor, FJ
Nanomaterials, **11** (2021) 380
Mayo 2021 | **DOI:** 10.3390/nano11020380

In this work, a mechanochemical route was proposed for the synthesis of the $PrBaMn_2O_{5+\delta}$ (PMBO) double layered perovskite phase. The mechanochemical reaction between Pr_6O_{11}, BaO_2, and MnO powders with cationic stoichiometric ratios of 1/1/2 for Pr/Ba/Mn was performed using high-energy milling conditions in air. After 150 min of milling, a new phase with perovskite structure and cubic symmetry consistent with the A-site disordered $Pr_{0.5}Ba_{0.5}MnO_3$ phase was formed. When this new phase was subsequently annealed at a high temperature in an inert Ar atmosphere, the layered $PrBaMn_2O_{5+\delta}$ phase was obtained without needing to use a reducing atmosphere. At 1100 °C, the fully reduced layered $PrBaMn_2O_5$ phase was achieved. A weight gain was observed in the 200–300 °C temperature range when this fully reduced phase was annealed in air, which was consistent with the transformation into the fully oxidized $PrBaMn_2O_6$ phase.

The microstructural characterization by SEM, TEM, and HRTEM ascertained the formation of the intended $PrBaMn_2O_{5+\delta}$ phase. Electrical characterization shows very high electrical conductivity of layered PBMO in a reducing atmosphere and suitable in an oxidizing atmosphere, becoming, therefore, excellent candidates as solid oxide fuel cell (SOFC electrodes).

Pure perovskite $BiFeO_3$-$BaTiO_3$ ceramics prepared by reaction flash sintering of Bi_2O_3-Fe_2O_3-$BaTiO_3$ mixed powders

Taibi, A; Chaguetmi, S; Sánchez-Jiménez, PE; Perejón, A; García, JE; Satha, H; Pérez-Maqueda, LA

Ceramics International, **47** (2021) 26947-26954

Octubre 2021 | **DOI:** 10.1016/j.ceramint.2021.06.108

In this work, the $0.67BiFeO_3$-$0.33BaTiO_3$ ferroelectric ceramic was prepared by Reaction Flash Sintering (RFS). This preparation technique combines synthesis and sintering in a single Flash experiment. The starting oxides reacted during the flash to produce a stoichiometric well-sintered solid solution at a temperature of 858 °C by applying a modest field of 35 V cm^{-1}. The process takes place in a matter of seconds, which allows obtaining a pure perovskite structure without secondary phases. X-ray diffraction (XRD) results show the mixture of rhombohedral and pseudocubic phases expected for a composition that lies within a morphotropic phase boundary (MPB) region, since a significant splitting is observed in the reflections at 2 theta values of 39 ° and 56.5 °. The microstructure exhibit a peculiar bimodal grain size distribution that determines the electrical properties. As compared with previous results, flash-prepared $0.67BiFeO_3$-$0.33BaTiO_3$ evidences smaller grain size, as well as slightly lower remanent polarization (P_r) and smaller coercive field (E_c) under similar electric fields. It is also demonstrated that the preparation by RFS provides benefits regarding electrical energy consumption.

Enhancing the electrical conductivity of *in-situ* reduced graphene oxide-zirconia composites through the control of the processing routine

López-Pernía, C; Morales-Rodríguez, A; Gallardo-López, A; Poyato, R

Ceramics International, **47** (2021) 9382-9391

Abril 2021 | **DOI:** 10.1016/j.ceramint.2020.12.069

Graphene oxide (GO) was mixed with 3 mol% yttria tetragonal zirconia polycrystal (3YTZP) using two powder processing routines: a colloidal method in an aqueous solution and a combination of ultrasonication with high-energy planetary ball milling in wet conditions. Highly densified 3YTZP composites with reduced GO (rGO) were consolidated by Spark Plasma Sintering. The in-situ reduction of GO was successfully achieved during the high temperature sintering process and a detailed study of the restoration of the graphene structure in the sintered composites has been made by Raman spectroscopy. Although no differences between the composites prepared by the two processing methods were found in the distribution of the rGO throughout the 3YTZP matrix for high rGO contents (i.e. the composites with 5 and 10 vol% rGO), a better distribution of the graphene phase was found in the composites with 1 and 2.5 vol% rGO prepared by planetary ball milling. This result, together with a better reduction of the GO in these composites, led to the obtaining of rGO/3YTZP composites with a better behavior in terms of electrical conductivity: an electrical percolation threshold below 2.5 vol% rGO and a high electrical conductivity value (-610 S/m for 10 vol% rGO).

Cation-driven electrical conductivity in Ta-doped orthorhombic zirconia ceramics

Moshtaghioun, BM; Laguna-Bercero, MA; Pena, JI; Gómez-García, D; Domínguez-Rodríguez, A
Ceramics International, **47** (2021) 7248-7522
Marzo 2021 | **DOI:** 10.1016/j.ceramint.2020.10.227

This paper is devoted to the study of the electrical conductivity of tantalum-doped zirconia ceramics prepared by spark plasma sintering. In this study, the temperature dependence of conductivity in as-prepared specimens and in those previously annealed in air is determined and compared. A semi-empirical model, which is based on the oxidation states of the cations, has been developed and successfully assessed. According to this, the conductivity is basically controlled by the diffusion of tetravalent zirconium cations in both cases, although the concentration of these species varies drastically with the amount of induced oxygen vacancies. This is a quite unexpected fact, since conductivity is normally controlled by anionic diffusion in zirconia ceramics. This option is forbidden here due to the presence of substitutional pentavalent cations. Therefore, conductivity values are much lower than those reported in trivalent or divalent substitutional cation doped zirconia ceramics.

Kinetic study of complex processes composed of non-independent stages: pyrolysis of natural rubber

Perejón, A; Sánchez-Jiménez, PE; García-Garrido, C; Pérez-Maqueda, LA
Polymer Degradation and Stability, **188** (2021) 109590
Junio 2021 | **DOI:** 10.1016/j.polymdegradstab.2021.109590

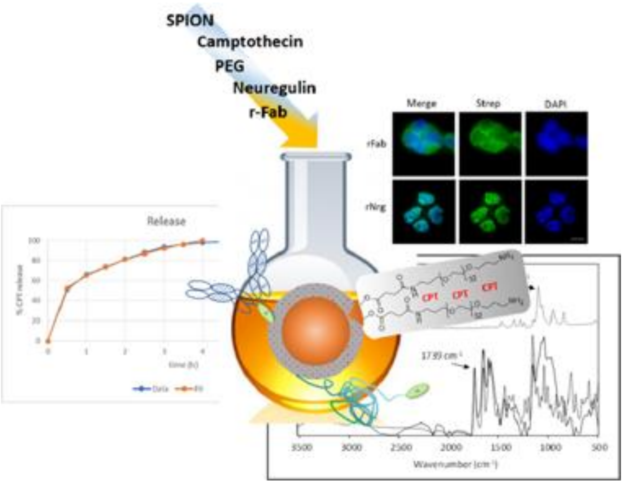
In this work, it is proposed a method for studying kinetics of complex processes composed of non-independent stages. In this method, the variable contribution of the different stages as a function of the heating schedule is taken into account. The method involves the simultaneous kinetic analysis of a set of experimental data registered under linear heating rate conditions, without any previous assumptions regarding the kinetic models followed by the stages or their corresponding activation energies.

The method has been tested with the kinetic analysis of the pyrolysis of natural rubber, since the kinetics of this process is complex and depends on temperature and heating schedule. It is demonstrated that the behavior of the experimental curves can be accurately predicted with the kinetic parameters calculated by the proposed methodology.

The kinetic analysis presented here could be applied to other complex processes as those found in pyrolysis, without the need of using oversimplified kinetic models that could yield significant errors when used in real applications.

EGFR-targeting antitumor therapy: Neuregulins or antibodies?

de Lavera, I; Merkling, PJ; Oliva, JM; Sayagués, MJ; Cotán, D; Sánchez-Alcázar, JA; Infante, JJ; Zaderenko, A.P.
European Journal of Pharmaceutical Sciences, **158** (2021) 105678
Marzo 2021 | **DOI:** 10.1016/j.ejps.2020.105678



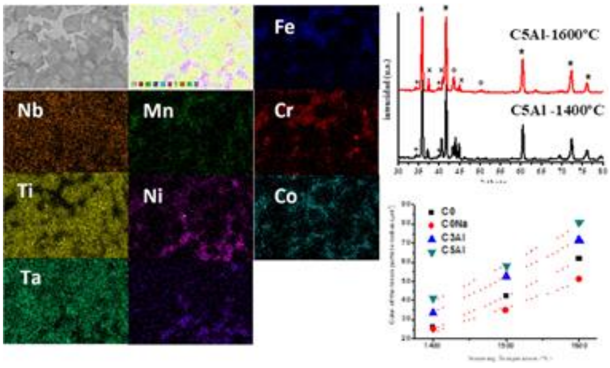
Malignancies such as lung, breast and pancreatic carcinomas are associated with increased expression of the epidermal growth factor receptor, EGFR, and its role in the pathogenesis and progression of tumors has made this receptor a prime target in the development of antitumor therapies. In therapies targeting EGFR, the development of resistance owing to mutations and single nucleotide polymorphisms, and the expression of the receptor ligands themselves are very serious issues. In this work, both the ligand neuregulin and a bispecific antibody fragment to EGFR are conjugated separately or together to the same drug-delivery system to find the most promising candidate. Camptothecin is used as a model chemotherapeutic drug and superparamagnetic iron oxide nanoparticles as a delivery system. Results show that the lowest LD50 is achieved by formulations conjugated to both the antibody and the ligand, demonstrating a synergy. Additionally, the ligand location in the nucleus favors the antitumor activity of Camptothecin. The high loading capacity and efficiency convert these systems into a good alternative for administering Camptothecin, a drug whose use is otherwise severely limited by its chemical instability and poor solubility. Our choice of targeting agents allows treating tumors that express ErbB2 (Her2+ tumors) as well as Her2- tumors expressing EGFR.

Fabrication and characterization of FeCoNiCrMn,(Al) high entropy alloy based (Ti,Ta,Nb)(C,N) cermet

Real, C; Alcalá, MD; Trigo, I; Fombella, I; Córdoba, JM

International Journal of Refractory Metals & Hard Materials, **101** (2021) 105694

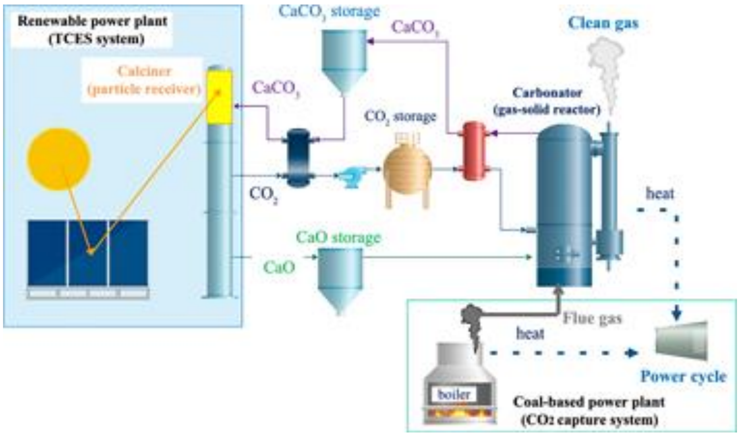
Diciembre 2021 | **DOI:** 10.1016/j.ijrmhm.2021.105694



From nanostructured mechanically synthesized powder a set of FeCoNiCrMn,(Al) based (Ti,Ta,Nb)(C,N) cermets were fabricated and sintered by a pressureless procedure. Highly dense cermets were obtained, and the nature of chemical change, microstructure, mechanical properties and coarsening kinetic of ceramic phase were characterized by image analysis, microindentation, scanning electron microscopy and X-ray diffraction. The design of the material was performed using a set of three different chemical cermet composition and three different sintering temperatures, or comparative purposes.

Scaling-up the Calcium-Looping Process for CO_2 Capture and Energy Storage

Ortiz, C; Valverde, JM; Chacartegui, R; Pérez-Maqueda, LA; Giménez-Gavarrell, P
Kona Powder and Particle Journal, **38** (2021) 189-209
Octubre 2021 | **DOI:** 10.14356/kona.2021005



The Calcium-Looping (CaL) process has emerged in the last years as a promising technology to face two key challenges within the future energy scenario: energy storage in renewable energy-based plants and CO_2 capture from fossil fuel combustion. Based on the multicycle calcination-carbonation reaction of $CaCO_3$ for both thermochemical energy storage and post-combustion CO_2 capture applications, the operating conditions for each application may involve remarkably different characteristics regarding kinetics, heat transfer and material multicycle activity performance. The novelty and urgency of developing these applications demand an important effort to overcome serious issues, most of them related to gas-solids reactions and material handling. This work reviews the latest results from international research projects including a critical assessment of the technology needed to scale up the process. A set of equipment and methods already proved as well as those requiring further demonstration are discussed. An emphasis is put on critical equipment such as gas-solids reactors for both calcination and carbonation, power block integration, gas and solids conveying systems and auxiliary equipment for both energy storage and CO_2 capture CaL applications.

Tuning the excitation wavelength of luminescent Mn^{2+}-doped ZnS_xSe_{1-x} obtained by mechanically induced self-sustaining reaction

Avilés, MA; Gotor, FJ
Optical Materials, **117** (2021) 111121
Julio 2021 | **DOI:** 10.1016/j.optmat.2021.111121

Mn^{2+}-doped ZnS_xSe_{1-x} solid solution samples ($Mn:ZnS_xSe_{1-x}$) were synthesized by the mechanochemical process denoted as mechanically-induced self-sustaining reaction from Mn/Zn/S/Se powder elemental mixtures. The samples were characterized by X-ray diffraction, scanning electron microscopy, diffuse reflectance UV-Vis spectroscopy and emission and excitation photoluminescence measurements. The band-gap energy of samples was controlled by changing the stoichiometry, x, of the solid solution. All samples showed the characteristic Mn^{2+} $^4T_1$-$^6A_1$ emission at -588 nm when exciting the host material, so it was possible to tune the excitation wavelength from 349 nm to 467 nm. However, an efficiency loss was observed with increasing Se content, probably due to the overlap between the absorption and emission spectra that induced self-absorption and emission quenching.

Critical Influence of the Processing Route on the Mechanical Properties of Zirconia Composites with Graphene Nanoplatelets

Gallardo-López, A; Muñoz-Ferreiro, C; López-Pernía, C; Jiménez-Pique, E; Gutiérrez-Mora, F; Morales-Rodríguez, A; Poyato, R
Materials, **14** (2021) 108
Enero 2021 | **DOI:** 10.3390/ma14010108

Graphene-based nanostructures, used as potential reinforcement in ceramic composites, have a great tendency to agglomerate. This requires the use of homogenization techniques during the powder processing, posing the need to evaluate how these techniques affect the microstructure and the mechanical properties of the resulting composites. The influence of the processing route on the properties of 3YTZP (3 mol % yttria tetragonal zirconia polycrystals) ceramic composites with 10 vol % cost-effective GNP (graphene nanoplatelets) has been addressed. Four different powder processing routines combining ultrasonic powder agitation (UA) and planetary ball milling (PBM) in wet and dry media have been used and all the composites were densified by spark plasma sintering (SPS). The mechanical properties at room temperature in the macroscale have been assessed by Vickers indentations, four-point bending tests and the impulse-echo technique, while instrumented indentation was used to measure the hardness and Young's modulus at the nanoscale. The application of dry-PBM enhances greatly the mechanical and electrical isotropy of the composites, slightly increases the hardness and lowers the elastic modulus, independently of the application of UA. The combination of UA and dry-PBM enhances the flexure strength by 50%, which is desirable for structural applications.

The Possible Detriment of Oxygen in Creep of Alumina and Zirconia Ceramic Composites Reinforced with Graphene

Cano-Crespo, R; Rivero-Antúnez, P; Gómez-García, D; Moreno, R; Domínguez-Rodríguez, A
Materials, **14** (2021) 984
Febrero 2021 | **DOI:** 10.3390/ma14040984

This paper aims to give an answer to the following question: is the oxidation of graphene a critical issue for high-temperature plasticity in graphene-reinforced ceramics? To give a convincing reply, we will focus on two very different graphene-based ceramic composites: reduced graphene oxide (rGO)-reinforced alumina (alpha-Al_2O_3) and reduced graphene oxide (rGO)-reinforced yttria tetragonal zirconia (t-ZrO_2). The processing of the powders has been made using a colloidal route, and after that, a spark plasma sintering process was performed in order to densify the samples. Creep tests were performed at temperatures between 1200-1250 °C in an argon atmosphere. The microstructure obtained by SEM of the sintered and tested specimens was characterized quantitatively to elucidate the deformation mechanism. Raman spectroscopy was carried out to check the integrity of the graphene. The average grain size was in the order of 1 μm and the shape factor was 0.7 for all the studied materials. The integrity of the graphene was checked before and after the creep experiments. The careful analysis of the creep tests shows that graphene oxide or its reduced version are not efficient phases for creep resistance improvement in general, contrary to what is reported elsewhere. However, the results permit the suggestion of a creep improvement in nanocomposites at a very high temperature regime due to an enhanced reactivity of oxygen between carbon and alumina interfaces. In the case of zirconia, the results give us the conclusion that the oxidation of graphene is a highly detrimental issue regarding the improvement of high-temperature plasticity.

Synthesis and Characterization of a Nearly Single Bulk Ti_2AlN MAX Phase Obtained from Ti/AlN Powder Mixture through Spark Plasma Sintering

Salvo, C; Chicardi, E; Poyato, R; García-Garrido, C; Jiménez, JA; López-Pernía, C; Tobosque, P; Mangalaraja, RV
Materials, **14** (2021) 2217
Mayo 2021 | **DOI:** 10.3390/ma14092217

MAX phases are an advanced class of ceramics based on ternary carbides or nitrides that combine some of the ceramic and metallic properties, which make them potential candidate materials for many engineering applications under severe conditions. The present work reports the successful synthesis of nearly single bulk Ti_2AlN MAX phase (>98% purity) through solid-state reaction and from a Ti and AlN powder mixture in a molar ratio of 2:1 as starting materials. The mixture of Ti and AlN powders was subjected to reactive spark plasma sintering (SPS) under 30 MPa at 1200 °C and 1300 °C for 10 min in a vacuum atmosphere. It was found that the massive formation of Al_2O_3 particles at the grain boundaries during sintering inhibits the development of the Ti_2AlN MAX phase in the outer zone of the samples. The effect of sintering temperature on the microstructure and mechanical properties of the Ti_2AlN MAX phase was investigated and discussed.

Study of the Influence of Sintering Atmosphere and Mechanical Activation on the Synthesis of Bulk Ti_2AlN MAX Phase Obtained by Spark Plasma Sintering

Salvo, C; Chicardi, E; García-Garrido, C; Poyato, R; Jiménez, JA; Mangalaraja, RV
Materials, **14** (2021) 4574
Agosto 2021 | **DOI:** 10.3390/ma14164574

The influence of the mechanical activation process and sintering atmosphere on the microstructure and mechanical properties of bulk Ti_2AlN has been investigated. The mixture of

Ti and AlN powders was prepared in a 1:2 molar ratio, and a part of this powder mixture was subjected to a mechanical activation process under an argon atmosphere for 10 h using agate jars and balls as milling media. Then, the sintering and production of the Ti_2AlN MAX phase were carried out by Spark Plasma Sintering under 30 MPa with vacuum or nitrogen atmospheres and at 1200 °C for 10 min. The crystal structure and microstructure of consolidated samples were characterized by X-ray Diffraction, Scanning Electron Microscopy, and Energy Dispersive X-ray Spectroscopy. The X-ray diffraction patterns were fitted using the Rietveld refinement for phase quantification and determined their most critical microstructural parameters. It was determined that by using nitrogen as a sintering atmosphere, Ti_4AlN_3 MAX phase and TiN were increased at the expense of the Ti_2AlN. In the samples prepared from the activated powders, secondary phases like Ti_5Si_3 and Al_2O_3 were formed. However, the higher densification level presented in the sample produced by using both nitrogen atmosphere and MAP powder mixture is remarkable. Moreover, the high-purity Ti_2AlN zone of the MAX-1200 presented a hardness of 4.3 GPa, and the rest of the samples exhibited slightly smaller hardness values (4.1, 4.0, and 4.2 GPa, respectively) which are matched with the higher porosity observed on the SEM images.

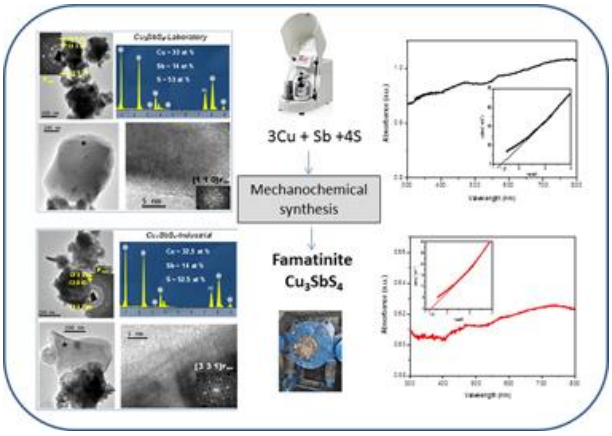
Effects of an Illite Clay Substitution on Geopolymer Synthesis as an Alternative to Metakaolin

Eliche-Quesada, D; Bonet-Martínez, E; Pérez-Villarejo, L; Castro, E; Sánchez-Soto, PJ
Journal of Materials in Civil Engineering, **33** (2021) 04021072
Mayo 2021 | **DOI:** 10.1061/(ASCE)MT.1943-5533.0003690

In this study, a calcined illite clay from Bailén, Jaén, Spain, was valorized as a substitute of metakaolin in the synthesis of new geopolymeric materials. The raw materials, raw clay and commercial kaolin, were pretreated at 750 °C (4 h). Several samples (0%-100% by weight of clay) were activated by mixing NaOH solution and sodium silicate solution. The specimens were cured (60 °C and 99% relative humidity) for 24 h, then demolded and kept at ambient conditions for 7, 28, and 90 days. The prepared geopolymers were characterized by X-ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy. Physical, mechanical, and thermal properties were determined. The results indicated that the specimens based on the illite raw clay and metakaolin present an amorphous consolidated appearance, characteristic of the polycondensation reactions. The incorporation of up to 50% by weight of raw clay provided geopolymers with higher mechanical strength (39.6 MPa) and bulk density (1,455 kg/m^3), lower apparent porosity (19.6%), and similar although slightly higher thermal conductivity (0.25 W/mK) than control geopolymers containing only metakaolin as a precursor after 28 days of curing. Control geopolymers presented compressive strength, bulk density, apparent porosity, and thermal conductivity of 23 MPa, 1,251 kg/m^3, 41.03% and 0.224 W/mk, respectively, at the same age of cured geopolymers. The mechanical properties increased with curing time due to a greater advance of the geopolymerization reaction. Therefore, this illite clay can be thermally activated together with metakaolin to obtain geopolymers with suitable technological properties. The results demonstrate that the finished materials can be used for construction applications.

Mechanochemically synthesized ternary chalcogenide Cu_3SbS_4 powders in a laboratory and an industrial mill

Dutkova, E; Sayagués, MJ; Fabián, M; Balaz, M; Achimovicova, M
Materials Letters, **291** (2021) 129566
Mayo 2021 | **DOI:** 10.1016/j.matlet.2021.129566



In this work, we demonstrate the use of elemental precursors (Cu, Sb, S) to synthesize famatinite Cu_3SbS_4 using a laboratory planetary ball milling and an industrial eccentric vibratory milling. Cu_3SbS_4 was prepared for 120 min and 180 min in laboratory and industrial mill, respectively, with the utilization of protective atmosphere. The Cu_3SbS_4 prepared in the laboratory and industrial mill with crystallite size 14 nm and 10 nm, respectively, was confirmed by both LeBail refinement of the X-ray powder diffraction data and transmission electron microscopy. The determined band gap energy 1.31 eV and 1.24 eV is blue-shifted relative to the bulk Cu_3SbS_4. The synthesis of Cu_3SbS_4 by a scalable milling process represents a prospective route for mass production of material with potential photovoltaic properties.

A first insight into the microstructure and crack propagation in novel boron nitride nanosheet/3YTZP composites

Muñoz-Ferreiro, C; Morales-Rodríguez, A; Gallardo-López, A; Poyato, R
Boletín de la Sociedad Española de Cerámica y Vidrio, **60** (2021) 128-136
Mayo 2021 | **DOI:** 10.1016/j.bsecv.2020.02.003

In this work, novel 3 mol% yttria tetragonal zirconia polycristalline (3YTZP) ceramic composites with boron nitride nanosheets (BNNS) are investigated for the first time. Highly densified composites with 1 and 4 vol% BNNS were obtained by spark plasma sintering (SPS) after BNNS synthesis using a solution exfoliation method and BNNS dispersion into the ceramic powder by ultrasonication. The BNNS presented homogeneous distribution throughout the ceramic matrix and preferential alignment in the plane perpendicular to the pressing axis during SPS. The BNNS incorporation had practically no effect on the Vickers hardness of the material nor on the Young's modulus. Anisotropy in crack development was found in the composite with 4% vol BNNS, together with a mechanism of extensive microcracking. Several energy-absorbing mechanisms during crack propagation, such as crack deflection, crack bridging, crack branching, BNNS pull-out and BNNS debonding, were identified in the composites by a close observation of the indentation-induced fracture paths.

Relevance of Particle Size Distribution to Kinetic Analysis: The Case of Thermal Dehydroxylation of Kaolinite

Arcenegui-Troya, J; Sánchez-Jiménez, PE; Perejón, A; Pérez-Maqueda, LA
Processes, **9** (2021) 1852
Octubre 2021 | **DOI:** 10.3390/pr9101852

Kinetic models used for the kinetic analysis of solid-state reactions assume ideal conditions that are very rarely fulfilled by real processes. One of the assumptions of these ideal models is that all sample particles have an identical size, while most real samples have an inherent particle size distribution (PSD). In this study, the influence of particle size distribution, including bimodal PSD, in kinetic analysis is investigated. Thus, it is observed that PSD can mislead the identification of the kinetic model followed by the reaction and even induce complex thermoanalytical curves that could be misinterpreted in terms of complex kinetics or intermediate species. For instance, in the case of a bimodal PSD, kinetics is affected up to the point that the process resembles a reaction driven by a multi-step mechanism. A procedure for considering the PSD in the kinetic analysis is presented and evaluated experimentally by studying the thermal dehydroxylation of kaolinite. This process, which does not fit any of the common ideal kinetic models proposed in the literature, was analyzed considering PSD influence. However, when PSD is taken into account, the process can be successfully described by a 3-D diffusion model (Jander's equation). Therefore, it is concluded that the deviations from ideal models for this dehydroxylation process could be explained in terms of PSD.

Mining Wastes of an Albite Deposit as Raw Materials for Vitrified Mullite Ceramics

Sánchez-Soto, PJ; Garzón, E; Pérez-Villarejo, L; Angelopoulos, GN; Eliche-Quesada, D
Minerals, **11** (2021) 232
Marzo 2021 | **DOI:** 10.3390/min11030232

In this work, an examination of mining wastes of an albite deposit in south Spain was carried out using X-ray Fluorescence (XRF), X-ray diffraction (XRD), particle size analysis, thermodilatometry and Differential Thermal Analysis (DTA) and Thermogravimetric (TG) analysis, followed by the determination of the main ceramic properties. The albite content in two selected samples was high (65-40 wt. %), accompanied by quartz (25-40 wt. %) and other minor minerals identified by XRD, mainly kaolinite, in agreement with the high content of silica and alumina determined by XRF. The content of Na_2O was in the range 5.44-3.09 wt. %, being associated with albite. The iron content was very low (<0.75 wt. %). The kaolinite content in the waste was estimated from ~ 8 to 32 wt. %. The particle size analysis indicated values of 11-31 wt. % of particles <63 μm. The ceramic properties of fired samples (1000-1350 °C) showed progressive shrinkage by the thermal effect, with water absorption and open porosity almost at zero at 1200-1250 °C. At 1200 °C, the bulk density reached a maximum value of 2.38 g/cm^3. An abrupt change in the phase evolution by XRD was found from 1150 to 1200 °C, with the disappearance of albite by melting in accordance with the predictions of the phase diagram SiO_2-Al_2O_3-Na_2O and the system albite-quartz. These fired materials contained as main crystalline phases quartz and mullite. Quartz was present in the raw samples and mullite was formed by decomposition of kaolinite. The observation of mullite forming needle-shape crystals was revealed by Scanning Electron Microscopy (SEM). The formation of fully densified and vitrified mullite materials by firing treatments was demonstrated.

Mechanochemical synthesis of ternary chalcogenide chalcostibite $CuSbS_2$ and its characterization

Dutkova, E; Sayagués, MJ; Fabián, M; Kovac, J; Kovat, J; Balaz, M; Stahorsky, M
Journal of Materials Science-Materials in Electronics, **32** (2021) 22898-22909
Agosto 2021 | **DOI:** 10.1007/s10854-021-06767-9

In this work, the very rapid one-step mechanochemical synthesis of nanocrystalline ternary chalcogenide chalcostibite $CuSbS_2$ prepared from copper, antimony, and sulfur precursors by high-energy milling for only 30 min in a planetary mill is reported. XRD confirmed the orthorhombic crystal structure of $CuSbS_2$. The crystallite size of CuSbS2 calculated by LeBail refinement of the X-ray powder diffraction data was 25 nm. The nanocrystalline chalcostibite $CuSbS_2$ was also confirmed by transmission electron microscopy. The purity of $CuSbS_2$ was verified by Raman spectroscopy. The synthesized chalcostibite exhibits the specific surface area value of 2.4 m^2g^{-1}. UV-Vis spectroscopy showed the optical bandgap of $CuSbS_2$ as 1.54 eV with wide range of absorption in visible region. Photoresponse of $CuSbS_2$ was confirmed by I-V measurements under dark and light illumination. The proposed mechanochemical synthesis provides an alternative approach to prepare also other ternary semiconductor nanomaterials. $CuSbS_2$ semiconductor nanocrystals have the potential to be used as light absorbers in photovoltaics.

Influence of Successive Chemical and Thermochemical Treatments on Surface Features of Ti6Al4V Samples Manufactured by SLM

González, JE; de Armas, G; Negrin, J; Beltran, AM; Trueba, P; Gotor, FJ; Peon, E; Torres, Y
Metals, **11** (2021) 313
Febrero 2021 | **DOI:** 10.3390/met11020313

Ti6Al4V samples, obtained by selective laser melting (SLM), were subjected to successive treatments: acid etching, chemical oxidation in hydrogen peroxide solution and thermochemical processing. The effect of temperature and time of acid etching on the surface roughness, morphology, topography and chemical and phase composition after the thermochemical treatment was studied. The surfaces were characterized by scanning electron microscopy, energy dispersive X-ray spectroscopy, X-ray diffraction and contact profilometry. The temperature used in the acid etching had a greater influence on the surface features of the samples than the time. Acid etching provided the original SLM surface with a new topography prior to oxidation and thermochemical treatments. A nanostructure was observed on the surfaces after the full process, both on their protrusions and pores previously formed during the acid etching. After the thermochemical treatment, the samples etched at 40 °C showed macrostructures with additional submicro and nanoscale topographies. When a temperature of 80 °C was used, the presence of micropores and a thicker anatase layer, detectable by X-ray diffraction, were also observed. These surfaces are expected to generate greater levels of bioactivity and high biomechanics fixation of implants as well as better resistance to fatigue.

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Revista QUÍMICOS DEL SUR, 113 (2021) 60-64

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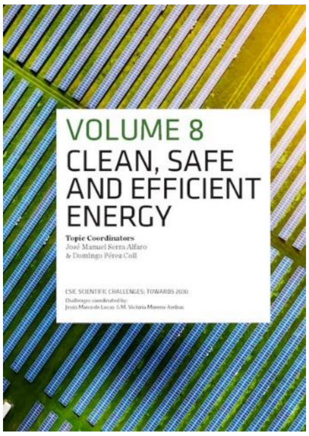

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páginas 45-70

Book of Abstracts II International Congress on Vitrification, Geopolymerization, Wastes Management, Green Cements and Circular Economy (II Vitrogeowastes)
L. Pérez-Villarejo, S. Martínez-Martínez, P.J. Sánchez-Soto (Editors)
Editado por UNIA y Universidad de Jaén, 210 páginas
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Characterization, thermal and ceramic properties of clays from Alhabia (Almería-Spain)
E. Rat; E. Garzón; J.A. Sánchez-Garrido; L. Pérez-Villarejo; P.J. Sánchez-Soto
6th Central and Eastern European Conference on Thermal Analysis and Calorimetry (CEEC-TAC6) and 15th Mediterranean Conference on Calorimetry and Thermal Analysis (Medicta2021), pág. 60
Book of Abstracts (Editors A. Rotaru, M. Erceg), SITECH, Rumanía
ISBN: 978-606-11-7861-2

Thermal behaviour of raw clay materials containing muscovite (illite/sericite), kaolinite and pyrophyllite: analysis of vitrification rate and estimation of optimum firing conditions
E. Rat, E. Garzón, L. Pérez-Villarejo, D. Eliche-Quesada, S. Martínez-Martínez, P.J. Sánchez-Soto
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CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

PARTICIPACIÓN EN LA ORGANIZACIÓN DE CONGRESOS Y REUNIONES / PARTICIPATION IN ORGANISING CONGRESSES AND MEETINGS

II International Congress on Vitrification, Geopolymerization, Waste Management, Green Cements and Circular Economy

24 – 26 mayo [Baeza, España]

Pedro José Sánchez Soto (Member of the Scientific and Technical Organizing Committee)

6th Central and Eastern European Conference on Thermal Analysis and Calorimetry (CEEC-TAC6) and & 15 th Mediterranean Conference on Calorimetry and Thermal Analysis (Medicta2021)

20-24 Julio [Split-Croatia]

Luis A. Pérez-Maqueda (Member of the International Organizing Committee, Chairman of Scientific Session Thursday 22nd July)

COMUNICACIONES / COMMUNICATIONS

II International Congress on Vitrification, Geopolymerization, Wastes Management, Green Cements and Circular Economy VITROGEOWASTES 2021

23-26 mayo [Baeza, Jaén]

Morphological and microstructural study of a low energy cement clinker with high dicalcium silicate content made from industrial wastes at different clinkering temperatures
S. Martinez-Martínez, D. Eliche-Quesada, P.J. Sánchez-.Soto, L. Pérez-Villarejo, A. Christogerou, G.N. Angelopoulos
Comunicación oral

Study of a waste kaolin deposit as raw material for mullite ceramics
P.J. Sánchez-Soto, D. Eliche-Quesada, S. Martínez-Martínez, L. Pérez-Villarejo, E. Garzón, J.M. Rincón
Comunicación oral

Study of the evolution with hydration of low-energy cement with high dicalcium silicate content made from industrial wastes
S. Martínez-Martínez, D. Eliche-Quesada, P.J. Sánchez-Soto, L. Pérez-Villarejo, A. Christogerou, G.N. Angelopoulos
Comunicación oral

Use of construction waste generated in the city of Almería in the manufacture of bases and sub-bases of rural roads and concrete of low strength
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Comunicación oral

6th Central and Eastern European Conference on Thermal Analysis and Calorimetry (CEEC-TAC6) and 15th Mediterranean Conference on Calorimetry and Thermal Analysis (Medicta2021)

20-24 julio [Split- Croacia]

Characterization, thermal and ceramic properties of clays from Alhabia (Almería-Spain)
E. Rat, E. Garzón, J.A. Sánchez-Garrido, L. Pérez-Villarejo, P.J. Sánchez-Soto
Comunicación oral

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L.A. Pérez-Maqueda, P.E. Sánchez-Jiménez, A. Perejón, N. Amghar, V. Moreno, J. Arcenegui-Troya, J.M. Valverde, C. Ortiz, R. Charcartegui
Comunicación oral

Thermal behaviour of raw clay materials containing muscovite (illite/sericite), kaolinite and pyrophyllite: analysis of vitrification rate and estimation of optimum firing conditions
E. Rat, E. Garzón, L. Pérez-Villarejo, D. Eliche-Quesada, S. Martínez-Martínez, P.J. Sánchez-Soto
Póster

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A. Taibi, E. Gil-González, S. Chaguetmi, P.E. Sánchez-Jiménez, A. Perejón, J.E. García, H. Satha, L.A. Pérez-Maqueda
Póster

9th International Conference on Fracture, Fatigue and Wear FFW 2021

2-3 agosto [Online]

Influence of graphene-based nanostructures morphology on the tribology properties in zirconia composites
C.Muñoz-Ferreiro, R. Moriche, A. Gallardo-López, R. Poyato, F. Gutiérrez
Comunicación oral

European Congress and Exhibition on Advanced Materials Advanced Materials and Processes EUROMAT 2021

13-17 septiembre [Online]

Electrical-discharge machinable zirconia composites with graphene nanostructures: Influence of nanostructure type and processing technique
A. Gallardo-López, R. Moriche, A. Gommeringer, F. Kern, C. López-Pernía, C. Muñoz-Ferreiro, R. Poyato
Comunicación oral

Mechanical properties and fracture mechanisms of graphene-based nanostructures/yttria-stabilized tetragonal zirconia (3YTZP) and their dependence on nanostructure aspect ratio and processing conditions
R. Moriche, C. Muñoz-Ferreiro, R. Poyato A. Gallardo-López
Comunicación oral

Processing and mechanical characterization of few layered graphene / zirconia composites
C. Muñoz-Ferreiro, H. Reveron, S. Cottrino, J. Chevlier, A. Morales-Rodríguez, R. Poyato, A. Gallardo-López
Comunicación oral

R-Curve behavior of GBN/3YTZP composites
C. López-Pernía, A. Morales-Rodríguez, C. Muñoz-Ferreiro, H. Reveron, J. Chevalier, R. Poyato, A. Gallardo-López
Comunicación oral

Studying the slow crack growth in a few layered graphene / zirconia composite
C. Muñoz-Ferreiro, A. Gallardo-López, R. Poyato, A. Morales-Rodríguez, H. Reveron, J. Chevalier
Comunicación oral

17th European Inter-Regional Conference on Ceramics CIEC17

23 Noviembre [Online]

Crack growth resistant graphene nanosheets / 3Y-TZP composite
C.Muñoz-Ferreiro, A. Gallardo-López, R. Poyato, A. Morales-Rodríguez, H. Reveron, J. Chevalier
Póster

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título: **Circularidad en la producción de cemento. Valorizando subproductos industriales en cementos innovadores de baja energía**
Autor: Sergio Martínez Martínez
Directores: Dolores Eliche Quesada (Universidad de Jaén) y Pedro José Sánchez Soto
Centro: Universidad de Jaén
Fecha Defensa: 22 de abril de 2021

Título: **Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno**
Autor: Cristina López Pernía
Directores: Rosalía Poyato Galán y Ángela Gallardo López
Centro: Universidad de Sevilla
Fecha Defensa: 1 de octubre de 2021

Formación de Graduados / Master Degree Thesis

Título: **Procesado y sinterización de matrices de circona cúbica con nanoestructuras de grafeno/ Processing and sintering of cubic circona matrices with Graphene nanostructures**
Autor: Tomás Espuny Plaza
Tutores: Rocío Moriche y Rosalía Poyato
Grado: Trabajo Fin de Grado del Grado en Ingeniería de Materiales
Centro: Facultad de Química, Universidad de Sevilla

Título: **Influencia de las condiciones de pirólisis de la biomasa procedente de la poda de naranjos en la naturaleza de los residuos obtenidos**
Autor: Patricia Rocío Espinar Gutiérrez
Tutores: María Dolores Alcalá González, Concepción Real Pérez
Grado: Trabajo Fin de Grado
Fecha Defensa: 11 enero 2021

Título: **Caracterización y fabricación mediante técnicas pulvimetalúrgicas de matrices de titania reforzadas para aplicaciones industriales**
Autor: Araceli Ruano Castaño
Tutores: Rosalía Poyato Galán y Juan Lozano Suárez (Escuela de Ingenieros - Universidad de Sevilla)
Grado: Trabajo Fin de Grado
Fecha Defensa: 22 julio 2021

Título: **Diseño de electrolitos para pilas de combustible de óxido sólido obtenidos mediante prensado uniaxial**
Autor: Mario Meléndez Mellado
Tutores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado en Ingeniería Mecánica
Fecha Defensa: 23 julio 2021

Título: **Procesado y caracterización microestructural de composites cerámicos con nanoláminas de nitruro de boro**
Autor: Ana Castro Chincho
Tutores: Rosalía Poyato Galán y Ana Morales Rodríguez
Grado: Trabajo Fin de Grado
Fecha Defensa: 17 septiembre 2021

Título: **Tenacidad y resistencia a flexión de compuestos cerámicos de circona con grafeno**
Autor: Ivonne Torres Delgado
Tutores: Rosalía Poyato Galán y Ángela Gallardo López
Grado: Trabajo Fin de Grado
Fecha Defensa: 25 noviembre 2021

Título: **Caracterización de superficies electromecanizadas en compuestos cerámicos con grafeno**
Autor: Pablo Montero Oliva
Tutores: Rosalía Poyato Galán y Ángela Gallardo López
Grado: Trabajo Fin de Máster
Fecha Defensa: 10 diciembre 2021

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales y en titulaciones de Grado y doble Grado de la Universidad de Sevilla (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Microdurómetro FM700 Future-Tech
- Máquina de ensayo universal AGS-J, Shimadzu
- 3 Termobalanzas CI Electronics
- 2 molinos planetarios PULVERISETTE 7 (Fritsch)
- 1 molino planetario PULVERISETTE 7 Premium Line (Fritsch)
- 1 molino planetario PULVERISETTE 4 (Fritsch)
- 1 molino planetario PM100 (Retsch)
- 1 molino vibratorio MM301 (Retsch)
- 1 molino vibratorio SPEX 8000
- 1 molino centrífugo PULVERISETTE (Fritsch)
- 1 molino de atrición O1HD (Union Process)
- Horno horizontal alta temperatura 1800 °C AGNI
- Horno horizontal alta temperatura 1600 °C Carbolite
- Horno horizontal 1200 °C Carbolite
- Microcortadora metalográfica manual EVOLUTION (REMET)
- Microcortadora metalográfica MICROMET (REMET)
- Prensa automática metalográfica IPA30 (REMET)
- Pulidora automática LS2 (REMET)
- Analizador Termomecánico TMA 1000 (Linseis)
- Calorímetro diferencial de barrido DSC (TA instruments Q200)
- Horno horizontal 1150 °C Hobersal
- Impedancímetro Agilent 4294ª
- Multímetro Keysight B2901A
- Equipo de Hot-Press TERMOLAB

MATERIALES FUNCIONALES NANOESTRUCTURADOS
NANOSTRUCTURED FUNCTIONAL MATERIALS

GRUPOS DE INVESTIGACIÓN

Materiales Nanoestructurados y Microestructura
Nanostructured Materials and Microstructure [642015]
http://nanomatmicro.icmse.csic.

Materiales para Bioingeniería y Regeneración Tisular
Materials for Bioengineering and Tissue Regeneration [642014]

Nanotecnología en Superficies y Plasma
Nanotechnologies on Surfaces and Plasma [642012]
http://sincaf.icms.us-csic.es/

Tribología y Protección de Superficies
Tribology and Protection of Surfaces [861494]

PERSONAL / PERSONNEL

Profesores de Investigación	Dr. Juan Pedro Espinós Manzorro
	Dra. Asunción Fernández Camacho
	Dr. Agustín Rodríguez González-Elipe
Catedráticos	Dr. José Cotrino Bautista
Investigadores Científicos	Dr. Ángel Barranco Quero
	Dr. Juan Carlos Sánchez López
	Dr. Francisco Yubero Valencia
Científicos Titulares	Dra. Ana Isabel Borrás Martos
	Dra. María Aránzazu Díaz Cuenca
	Dr. Alberto Palmero Acebedo
	Dra. T. Cristina Rojas Ruiz
	Dr. Juan Ramón Sánchez Valencia
Profesores Titulares	Dr. Rafael Álvarez Molina
Profesores Contratados Doctores	Dra. Ana María Gómez Ramírez
Contratados Plan Propio US	Dra. Carmen López Santos
Doctores Contratados	Dr. Francisco J. Aparicio Rebollo
	Dr. Jorge A. Budagosky Marcilla
	Dra. Lidia Contreras Bernal
	Dr. Ali Ghaffarinejad
	Dr. Jorge Gil Rostra
	Dra. Vanda Fortio Godinho
	Dr. Víctor López Flores
	Dr. Manuel Oliva Ramírez
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Personal Investigador en Formación	Ldo. Javier Castillo Seoane
	Gdo. Jaime Del Moral Jalón
	Ldo. Xabier García Casas
	Gdo. Darío Jumilla Núñez
	Gdo. Antonio José Márquez Alcaide
	Gda. Laura Montes Montañez
	Gda. Gloria Patricia Moreno Martínez
	Gdo. Fernando Núñez Gálvez
	Ldo. José Manuel Obrero Pérez
	Ldo. Noel Orozco Corrales
	Ldo. Álvaro Perea Brenes
	Lda. Xiaozhe Song
JAE Intro	D. Antonio Del Valle Hernández
	D. Servando Marín Meana
Personal Técnico Contratado	Ldo. Dirk Hufschmidt
	Lda. Miriam Sánchez Pérez

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Descongelación inteligente y sostenible mediante ingeniería de ondas acústicas aplicadas a superficies | SOUNDOFICE	**Sustainable Smart De-Icing by Surface Engineering of Acoustic Waves | SOUNDOFICE**
Código/Code:	H2020-FET-OPEN/0717
Periodo/Period:	01-11-2020 / 31-10-2024
Organismo Financiador/Financial source:	Comisión Europea
Importe total/Total amount:	690.602 €
Investigador responsable/Research head:	Ana Isabel Borrás Martos
Componentes/Research group:	Agustín R. González-Elipe, Juan Pedro Espinós, Francisco Yubero, Ángel Barranco, Víctor Rico, María del Carmen López Santos

ABSTRACT

Icing on surfaces is commonplace in nature and industry and too often causes catastrophic events. SOUNDofICE ultimate goal is to overcome costly and environmentally harmful de-icing methods with a pioneering strategy based on the surface engineering of MHz Acoustic Waves for a smart and sustainable removal of ice. This technology encompasses the autonomous detection and low-energy-consuming removal of accreted ice on any material and geometry. For the first time, both detection and de-icing will share the same operating principle. The visionary research program covers the modeling of surface wave atom excitation of ice aggregates, integration of acoustic transducers on large areas, and the development of surface engineering solutions to stack micron-size interdigitated electrodes together with different layers providing efficient wave propagation, anti-icing capacity, and aging resistance. We will demonstrate that this de-icing strategy surpasses existing methods in performance, multifunctionality, and capacity of integration on industrially relevant substrates as validated with proof of concept devices suited for the aeronautic and wind power industries. SOUNDofICE high-risks will be confronted by a strongly interdisciplinary team from five academic centers covering both the fundamental and applied aspects. Two SMEs with first-hand experience in icing will be in charge of testing this technology and its future transfer to key EU players in aeronautics, renewable energy, and household appliances. An Advisory Board incorporating relevant companies will contribute to effective dissemination and benchmarking. The flexibility of the R&D plan, multidisciplinarity, and assistance of the AdB guarantee the success of this proposal, bringing up a unique opportunity for young academia leaders and SMEs from five different countries to strengthen the EU position on a high fundamental and technological impact field, just on the moment when the climate issues are of maxima importance.

Diseño de nanomateriales tridimensionales para la solución todo en uno a la recolección de energía ambiental de fuentes múltiples | 3DSCAVENGERS	**Three-dimensional nanoscale design for the all-in-one solution to environmental multisource energy scavenging | 3DSCAVENGERS**
Código/Code:	H2020-ERC-STG/0655
Periodo/Period:	01-03-2020 / 28-02-2025
Organismo Financiador/Financial source:	Comisión Europea
Importe total/Total amount:	1.498.414 €
Investigador responsable/Research head:	Ana Isabel Borrás Martos

ABSTRACT

Thermal and solar energy as well as body movement are all sources of energy. They can be exploited by advanced technology, obviating the need for battery recharging. These local ambient sources of energy can be captured and stored. However, their low intensity and intermittent nature reduces the recovery of energy by microscale instruments, highlighting the need for an integrated multisource energy harvester. Existing methods combine different single source scavengers in one instrument or use multifunctional materials to concurrently convert various energy sources into electricity.

The EU-funded 3DScavengers project proposes a compact solution based on the nanoscale architecture of multifunctional three-dimensional materials to fill the gap between the two existing methods. These nanoarchitectures will be able to simultaneous and individual harvesting from light, movement and temperature fluctuations. 3DScavengers ultimate goal is to apply a scalable and environmental friendly one-reactor plasma and vacuum approach for the synthesis of this advanced generation of nanomaterials.

Plasmas atmosféricos de arco deslizante para procesos sostenibles	**Atmospheric Pressure Gliding-Arc Plasmas for Sustainable Applications | FIREBOW**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2020-114270RA-I00
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	108.900 €
Investigador responsable/Research head:	Ana María Gómez Ramírez
Componentes/Research group:	José Javier Brey Sánchez (US), José Cotrino Bautista, Paula de Navascués Garvín, Manuel Oliva Ramírez, Antonio Rodero Serrano (US)

RESUMEN / ABSTRACT

La necesidad de propiciar una transición efectiva desde una economía basada en el uso intensivo de combustibles fósiles a otra donde los criterios de desarrollo se basen en procesos sostenibles que no impliquen la generación de CO_2 hace necesaria la puesta a punto de nuevos procesos, donde la fuente de energía primaria sea la electricidad generada a partir de fuentes renovables. El proyecto "Plasmas Atmosféricos de Arco Deslizante para Procesos Sostenibles", FIREBOW en adelante, pretende el desarrollo de tecnologías de plasma atmosférico que usan la electricidad como vector energético directo para llevar a cabo procesos químicos convencionalmente abordados mediante técnicas catalíticas (a altas presiones y temperaturas, con bajos rendimientos y obtención de subproductos no deseados). En concreto se persigue la puesta a punto de un reactor de Plasma Atmosférico de Arco Deslizante (PAAD) para inducir tres procesos de gran impacto industrial y medioambiental, como son la síntesis de amoniaco (NH_3), la producción de hidrógeno (H_2) y la descontaminación de agua. El amoniaco es la sustancia base de los fertilizantes usados en agricultura, y su demanda aumenta conforme las necesidades de alimentación mundiales. En cuanto al hidrógeno, es conocido que el camino hacia una economía basada en dicho combustible es uno de los retos del siglo XXI. Por otro lado, el desarrollo de técnicas novedosas para la depuración de aguas es cada vez más necesaria, debido al aumento de contaminante emergentes, sustancias tales como pesticidas, compuestos derivados de la industria farmacéutica y química, microorganismos e incluso productos de higiene personal que los métodos convencionales no son capaces de eliminar en su totalidad. FIREBOW propone, en una primera etapa, desarrollar la tecnología PAAD mediante el diseño, construcción, modelización y puesta a punto de un reactor de arco deslizante. Se explorarán posibles modificaciones sobre los modelos de reactores PAAD actuales, contemplándose el efecto de la incorporación de materiales piezoeléctricos para inducir fenómenos de emisión secundaria de electrones, la modificación de las características superficiales de los electrodos o la geometría del sistema a fin de propiciar en el futuro una mejora en el rendimiento de los procesos estudiados. La complejidad de los procesos básicos involucrados en este tipo de reactores implicará un estudio fundamental de su respuesta eléctrica y de los fenómenos de transporte de masa y carga, así como una caracterización exhaustiva y diagnosis del plasma en función de parámetros como flujo de gases, interacción entre especies excitadas, tiempo de residencia y otros parámetros básicos de operación. Tanto la caracterización experimental como la simulación teórica del reactor, esta última llevada a cabo mediante métodos computacionales, serán fundamentales para su correcto funcionamiento y optimización de los procesos propuestos. En una segunda etapa se abordará el estudio de las reacciones de obtención de H_2 y NH_3, con el objetivo de maximizar el rendimiento energético de dichos procesos, así como de la purificación de agua. El desarrollo científico-tecnológico propuesto en FIREBOW es de gran interés para diferentes actores socio-económicos, planteándose actividades de transferencia a las empresas y entidades que ya han mostrado su interés en el desarrollo del mismo.

The need to promote an effective transition from an economy based on the intensive use of fossil fuels to another where the development criteria are based on sustainable processes that do not involve the generation of CO_2 makes it necessary to develop new processes using the electricity generated from renewable sources as primary source of energy. The project "Atmospheric Pressure Gliding-Arc Plasmas for Sustainable Applications", FIREBOW hereinafter, aims at developing atmospheric plasma technologies that use electricity as a direct energy vector to induce chemical processes that are currently carried out through catalytic techniques (i.e., at high pressures and temperatures, with low yields and harmful by-products). Specifically, FIREBOW pursues the development of a Gliding Arc Atmospheric Plasma reactor (GA) to induce three processes of great industrial and environmental impact, such as the synthesis of ammonia (NH_3), the production of hydrogen (H_2) and the decontamination of water. Ammonia is the main source to produce fertilizers, which are used in agriculture with an increasing demand according to the increasingly higher needs of foods at global scale. In the case of hydrogen, it is well-known that the path to an economy based on this fuel is one of the challenges of the 21st century. Research in novel techniques for water purification is also increasingly necessary, due to its scarcity and the increase in emergent contaminants, polluting substances such as pesticides, compounds derived from the pharmaceutical and chemical industry, microorganisms and even personal hygiene products that conventional methods are unable to remove completely. FIREBOW proposes, in a first stage, to develop the GA technology through the design, construction, modelling and commissioning of a GA reactor. Possible modifications on the current GA reactors will be explored, considering the effect of the incorporation of piezoelectric materials to induce phenomena of secondary emission of electrons, the modification of the electrode surface materials or the geometry of the system in order to improve the performance of the analysed processes with respect to the current state of the art. The complexity of the basic mechanisms involved in this type of reactors will require a fundamental study of

their electrical response and the phenomena of mass and charge transport, as well as an exhaustive characterization and diagnosis of the plasma as a function of operating parameters such as gas flow, interaction between excited species, residence time and other basic operating conditions. Both the experimental and theoretical characterization of the reactor, the latter carried out using computational methods, will be crucial for its correct operation and for the optimization of the proposed processes. In a second stage, the study of the reactions to obtain H_2 and NH_3 will be approached, with the aim of maximizing the energy efficiency, as well as that for the case of the purification of water. The scientific-technological developments proposed in FIREBOW are of the outmost interest to different socio-economic sectors and in the project they are considered knowledge-transfer actions to companies and entities that have already shown their interest in the proposal.

Procesos de Nucleación y Crecimiento en Superficies Piezoeléctricas Excitadas Acústicamente en Atmósferas de Plasma/Vacío	**Nucleation and growth mechanisms on piezoelectric surfaces under acoustic excitation in plasma/vacuum environments**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2020-112620GB
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	106.480 €
Investigador responsable/Research head:	Alberto Palmero Acebedo
Componentes/Research group:	Rafael Álvarez Molina, Víctor J. Rico Gavira, Agustín R. González-Elipe

RESUMEN / ABSTRACT

Este proyecto aborda el estudio de los fenómenos de nucleación atómica y crecimiento de películas delgadas sobre materiales piezoeléctricos sometidos a excitación acústica en atmósferas de plasma y vacío. Los materiales piezoeléctricos se caracterizan por la aparición de una polarización eléctrica no nula al someterlos a una deformación mecánica, y por el efecto contrario, la aparición de una deformación mecánica al someterlos a una excitación eléctrica y, en la actualidad, se emplean en multitud de aplicaciones y dispositivos, tales como sensores de lluvia, pantallas táctiles o manipulación de líquidos en la microescala, entre otros. En un trabajo seminal publicado por el grupo investigador se demostró que, al crecer una película delgada mediante

técnicas de plasma sobre este tipo de superficies excitadas, ésta se estructuraba de acuerdo al patrón definido por las ondas. Este resultado inicial demostraba que la interacción entre la onda/película delgada en crecimiento podría utilizarse como nueva metodología de nanoestructuración de superficies. En este proyecto se aborda esta problemática de tipo fundamental, estudiando específicamente dos mecanismos principales de interacción: i) la transferencia directa de energía/momento de la onda acústica a las especies depositadas, y ii) la interacción entre la onda de polarización eléctrica que se propaga por el material piezoeléctrico y las líneas de campo eléctrico en el plasma, con incidencia directa en el transporte de especies cargadas y al bombardeo superficial selectivo del piezoeléctrico durante el crecimiento de la película. De esta manera, este proyecto se centra en la descripción, desarrollo y comprensión de una nueva fenomenología, y en el desarrollo de todo el marco teórico y conceptual que permita entender dicha interacción. Se espera que la activación acústica de piezoeléctricos y su efecto en atmósferas de plasma se convierta en un nuevo procedimiento para inducir la formación de centros de nucleación para la micro- y nano-estructuración de películas delgadas, permitiendo nuevos desarrollos en el campo de la física de superficies. Asimismo, en el campo de la física del plasma, la posibilidad de modular la interacción entre el plasma y una superficie de acuerdo a un patrón definido por ondas electro-acústicas podría abrir procedimientos alternativos para operar dispositivos de microplasmas o pantallas de plasma.

This project aims at studying atomic nucleation and thin film growth phenomena on piezoelectric surfaces under acoustic excitation in vacuum/plasma environments. Piezoelectric materials are characterized by a non-zero polarization vector when subjected to mechanical deformation and the reverse, a mechanical deformation when subjected to an electrical excitation. While piezoelectric surfaces under acoustic excitation are being used for numerous applications, e.g. raindrop sensors, touch-sensitive screens, or handling of liquids at the microscale, among others, a systematic survey of the literature reveals that only a seminal work published by the research team addresses the effect of acoustic waves in nucleation and growth processes in a plasma environment. There, we demonstrated a strong correlation between the features of the acoustic wave, the associated polarization pattern on the piezoelectric material and the structural features of a surface grown in the presence of a plasma, suggesting that this interaction can be employed as a new methodology to tailor the film nanostructure. Two main sources of interaction are analyzed in this project: i) the mechanical influence of the propagating acoustic wave on the surface-induced mobility processes of ad-atoms, ii) the interaction between the polarization wave on the piezoelectric and the plasma electric field lines, that may affect the transport of charged species and their impingement on the piezoelectric material during growth. In this way, this project focusses on the description, development and understanding of a new phenomenology, and on the provision of the fundamental and theoretical framework to describe this interaction. It is expected that acoustic waves activation and its effect on surrounding plasmas represents a radically new procedure to activate thin film growth and nuclei formation and that the proposed methodology goes beyond any present paradigm in the field of surface physics, envisaging new routes of nanostructuration. Similarly, in the field of plasma dynamics, the possibility of modulating the plasma/surface interaction by acoustic waves is an option that may open alternative procedures for the operation of advanced microplasmas devices or flat plasma displays.

Tecnología de plasma para la fabricación de celdas solares de perovskita eficientes y duraderas a prueba de agua	**Plasma technology for efficient and DURAble waterproof perovskite SOLar cells**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2019-109603RA-I00
Periodo/Period:	01-06-2020 / 31-05-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	96.800 €
Investigador responsable/Research head:	Juan Ramón Sánchez Valencia y María del Carmen López Santos
Componentes/Research group:	Juan Pedro Espinós Manzorro, Javier Castillo Seoane, Xabier García Casas, Víctor López Flores

RESUMEN / ABSTRACT

Las celdas solares –dispositivos que transforman directamente la luz solar en electricidad- son de vital interés para el futuro sostenible del planeta. Durante los últimos años y conscientes de este hecho, la comunidad científica ha realizado un gran esfuerzo por mejorar la eficiencia de estos dispositivos. Un ejemplo particular de celda solar que contiene una perovskita de haluro organometálico como absorbedor de luz han centrado la atención de la comunidad científica durante la última década debido, sobre todo, a su alta eficiencia y bajo coste. Esta tecnología de celda solar supone una alternativa prometedora a las celdas actuales (basadas en Si y en calcogenuros), aunque se enfrentan a un reto científico y tecnológico que no ha sido resuelto en 10 años desde su descubrimiento: para que la realización final y comercial de las celdas de perovskita sea posible, necesitan alcanzar una mayor estabilidad, durabilidad y reproducibilidad. El principal problema radica en la alta sensibilidad que presentan estas perovskitas al oxígeno y humedad ambiental, que producen una rápida degradación del comportamiento de la celda en un tiempo extremadamente corto, haciendo inviable su comercialización.

DuraSol persigue abordar este gran reto científico y tecnológico mediante la fabricación de componentes de la celda mediante tecnología de vacío y plasma. Estas metodologías son escalables industrialmente y presentan grandes ventajas con respecto a las metodologías en disolución (las más usadas), entre las que destacan: su alta versatilidad, control de composición y microestructura, bajo coste, que son respetuosas con el medio ambiente ya que no precisan disolventes, no producen emisiones contaminantes y son compatibles con la tecnología actual de semiconductores.

El objetivo principal de DuraSol es la fabricación de celdas solares de perovskita "a prueba de agua" mediante integración de componentes fabricados por metodologías de vacío y plasma en forma de películas delgadas y nanoestructuras, que actúan como sellantes hidrofóbicos. La viabilidad de DuraSol se basa en resultados recientes que demuestran que la fabricación asistida por plasma de distintos componentes de la celda solar puede ser una de las vías más prometedoras para aumentar su estabilidad y durabilidad, que es hoy en día el cuello de botella que impide su comercialización. Cabe señalar que no hay ningún ejemplo en la literatura de este enfoque sintético, y se espera que esta oportunidad demuestre las ventajas y la versatilidad de esta metodología innovadora en un campo de muy alto impacto. La investigación propuesta en DuraSol se enmarca dentro de las áreas prioritarias del programa Horizon 2021-2027 de la Unión Europea y responden a varios de los retos propuestos en la presente convocatoria de "Energía segura, eficiente y limpia" (Reto 3) y de "Cambio climático y utilización de recursos y materias primas" (Reto 5).

Solar cells – devices that transform sunlight into electricity – are of vital interest for the sustainable future of the planet. During the last years and aware of this fact, the scientific community has made a great effort to improve the efficiency of these devices. A particular example of a solar cell that contains an organometallic halide perovskite as light absorber has focused the attention of the scientific community during the last decade due, above all, to its high efficiency and low cost. This solar cell technology is a promising alternative to currently existing ones (based on Si and chalcogenides), although they face a scientific and technological challenge that has not been solved in 10 years since its discovery: for the commercial realization of the perovskite cells possible, they need to achieve higher stability, durability and reproducibility. The main problem lies in the high sensitivity of these perovskites to oxygen and environmental humidity, which produce a rapid degradation of the cell's behaviour in an extremely short time, making commercialization unfeasible.

DuraSol seeks to address this great scientific and technological challenge by manufacturing cell components using vacuum and plasma technology. These methodologies are industrially scalable and present great advantages over solution methods (the most used), among which are: their high versatility, control of composition and microstructure, low cost, environmentally friendly since they do not require solvents, do not produce pollutant emissions and are compatible with current semiconductor technology.

The main objective of DuraSol is the fabrication of waterproof perovskite solar cells by integrating components manufactured by vacuum and plasma methodologies in the form of thin films and nanostructures, which act as hydrophobic sealants. The viability of DuraSol is based on recent results that demonstrate that plasma-assisted synthesis of different components of the solar cell can be one of the most promising ways to increase its stability and durability, which is today the bottleneck that prevents their commercialization. It is worth to highlight that there is no example in the literature about this synthetic approach, and this opportunity is expected to demonstrate the advantages and versatility of this innovative methodology in a field of very high impact. The research proposed in DuraSol falls within the priority areas of the European Union Horizon 2021-2027 program and responds to several of the challenges proposed in this call for "Energía segura, eficiente y limpia" (Challenge 3) and "Cambio climático y utilización de recursos y materias primas" (Challenge 5).

Estructuras adaptativas multiresponsivas para fotónica integrada, piezo/tribotrónica y monitorización optofluídica|AdFunc	**Adaptive multiresponsive nanostructures for integrated photonics, piezo/tribotronics and optofluidic monitoring| AdFunc**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2019-110430GB-C21
Periodo/Period:	01-06-2020 / 31-05-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	211.750 €
Investigador responsable/Research head:	Ángel Barranco Quero y Ana Isabel Borrás Martos
Componentes/Research group:	José Cotrino Bautista, Victor J. Rico Gavira, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Agustín R. González-Elipe

RESUMEN / ABSTRACT

AdFunc es un proyecto muy interdisciplinar que tiene como principal objetivo conseguir un progreso significativo en dos temáticas en la frontera de la Ciencia de Materiales: el desarrollo de sensores con capacidad multirespuesta y de sistemas de energía activados por luz. Los denominadores comunes de AdFunc son el diseño inteligente de arquitecturas complejas en la nanoescala y el desarrollo de demostradores a escala de laboratorio.

Estamos convencidos de que el proyecto nos abre una ventana de oportunidad para realizar investigaciones que podemos clasificar en cuatro áreas: i) Aplicaciones y dispositivos: Desarrollaremos los recientemente descubiertos efectos tribotrónicos y piezotrónicos para fabricar dispositivos sensores autoalimentados. Con estos materiales, en combinación con varias tecnologías avanzadas de sensado fotónico y espectro-electroquímico, expandiremos la eficiencia, multiactuación y multirespuesta de sistemas adaptativos optofluídicos. Estos sistemas, manteniendo una arquitectura común, presentarán una respuesta diferenciada frente a escenarios reales diversos y complejos, que se simularán en el proyecto (alteraciones medioambientales como vertidos, accidentes, amenazas químicas o de explosivos). También se plantean dispositivos captadores de energía solar en condiciones de baja iluminación, captadores de energía mecánica y dispositivos que sean capaces de acoplar luz y movimiento a la activación de procesos de descomposición electroquímica del agua. ii) Nanomateriales: Adfunc es un proyecto donde concurren un equipo de especialistas en el desarrollo de nanoestructuras soportadas por distintas tecnologías. Esto nos permitirá, por primera vez, implementar un conjunto de nanoarquitecturas 3D (nanohilos, nanotubos, core@shell) y el diseño de materiales con estructuras nanoporosas controladas (capas esculturales, nanocanales, porosidad asociada en varias escalas, multicapas ópticas porosas, desarrollos pioneros de redes metaloorgánicas (MOFs) en estructuras fotónicas porosas) directamente a la mejora de los componentes activos de los dispositivos del proyecto. iii) Estrategia. El proyecto nos da la oportunidad de trabajar simultáneamente en rutas sintéticas nuevas, caracterización avanzada de materiales y propiedades, integración de materiales en dispositivos, y esto a la vez que se tiene información de modelado y simulación. iv) Perspectiva de escalabilidad: En todos los casos se utilizarán métodos y técnicas compatibles con procesos industriales establecidos, como el plasma y el vacío típicos de la industria optoelectrónica y microelectrónica, y procesos de síntesis en disolución. Otro aspecto interesante, es la posibilidad de introducir plásticos y polímeros para fabricar dispositivos, lo que puede permitir revalorizar residuos de la industria del plástico, en un esfuerzo de economía circular en el que investigadores del proyecto están comprometidos.

AdFunc sólo es posible gracias al esfuerzo conjunto de un gran número de investigadores, en su mayoría del ICMS-CSIC y la Universidad Pablo de Olavide, que se completa con un grupo de investigadores de otros centros y colaboradores internacionales con experiencia e interés complementarios. Es precisamente la coordinación de un número tan elevado de especialistas (25 doctores en los dos subproyectos) lo que nos permite plantear el desarrollo de un conjunto de actividades tan completo y ambicioso.

AdFunc is a highly interdisciplinary project whose main objective is to achieve significant progress in two areas at the frontier of Materials Science: the development of multi-response sensors and light-activated energy systems. The common denominators of AdFunc are the intelligent design of complex architectures at the nanoscale and the development of laboratory scale demonstrators.

We are convinced that the project opens a window of opportunity for us to carry out research that can be classified into four areas: i) Applications and devices: We will develop the recently discovered tribotronic and piezotronic effects to manufacture self-powered sensor devices. With these materials, in combination with several advanced photonic sensing and spectro-electrochemical technologies, we will expand the efficiency, multiactuation and multiresponse of optofluidic adaptive systems. These systems, maintaining a common architecture, will present a differentiated response to diverse and complex real scenarios, which will be simulated in the project (environmental alterations such as spills, accidents, chemical or explosive threats).

Another fundamental aspect of the project are the photovoltaic devices, which will be optimized to be able to work in low light conditions, and mechanical energy collectors and devices that are capable of coupling light and movement to the activation of the water electrochemical decomposition. ii) Nanomaterials: AdFunc is a project where a team of specialists in the development of supported nanostructures by different technologies come together. This will allow us, for the first time, to implement a set of 3D nanoarchitectures (nanowires, nanotubes, core@shell) and the design of materials with controlled nanoporous structures (sculptural layers, nanochannels, porosity associated in several scales, porous optical multilayers, pioneering developments of metalloorganic networks (MOFs) in porous photonic structures) directly to the improvement of the active components of the project devices. Iii) Strategy: The project gives us the opportunity to work simultaneously on new synthetic routes, advanced characterization of materials and properties, integration of materials into devices, and this while simultaneously obtaining modeling and simulation information. iv) Perspective of scalability: In all cases, methods and techniques compatible with established industrial processes will be used, such as plasma and vacuum, typical of the optoelectronic and microelectronic industry, and synthesis processes in solution. Another interesting aspect is the possibility of introducing plastics and polymers to manufacture devices, which may allow the valorization of waste from the plastic industry, in an effort of circular economy in which researchers of the project are committed.

AdFunc is only possible thanks to the joint effort of a large number of researchers, mostly from ICMS-CSIC and the Pablo de Olavide University, which is completed by a group of researchers from other national and international institutions with complementary experience and interest. It is precisely the coordination of such a large number of specialists (25 doctors in the two subprojects) that allows us to propose the development of such a complete and ambitious set of activities.

Recubrimientos innovadores preparados por magnetron sputtering para absorción solar selectiva	**MAGnetron sputtered Innovative COatings for Solar Selective absorption MAGICOS2**
MINISTERIO DE CIENCIA E INNOVACIÓN	AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	PID2019-104256RB-I00
Periodo/Period:	01-06-2020 / 31-05-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Juan Carlos Sánchez López y Ramón Escobar Galindo (US)
Componentes/Research group:	Cristina Rojas Ruiz, Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

El cambio climático ocasionado por las emisiones de gases con efecto invernadero y el agotamiento de los combustibles fósiles a corto-medio plazo hacen necesaria la búsqueda de nuevas fuentes de energía alternativas, limpias y renovables. De entre ellas, la energía solar es una de las mejores opciones por su gran disponibilidad para la generación de calor y electricidad.

El objetivo de este proyecto va encaminado al desarrollo de nuevos recubrimientos absorbedores solares selectivos crecidos en forma de multicapas basados en nitruros metálicos de cromo y aluminio (CrAlN). Las propiedades de resistencia a la oxidación y estabilidad térmica del CrAlN unidas a un diseño nanoestructurado adecuado permitirán mantener unas buenas prestaciones ópticas (alta absorbancia y baja emitancia) y mejorar su durabilidad a alta temperatura. El incremento de la temperatura de trabajo ($T > 550$ °C) conllevará una mejora de la eficiencia y una reducción de costes de las plantas de concentración de solar térmica, haciéndolas más competitivas. Para su preparación se utilizará la técnica de pulverización catódica mediante impulsos de alta intensidad (HiPIMS), una variante reciente de la pulverización catódica convencional que permite mejorar la densidad y compacidad de las capas gracias a un mayor grado de ionización del plasma. Estas propiedades son de interés para mejorar la adhesión al sustrato y ralentizar los procesos de degradación térmica. Además de los nitruros se ensayarían otras configuraciones cambiando el tipo de material absorbedor (oxinitruros y nanocomposites de carburos metálicos).

El proyecto comprenderá todas las etapas, desde la síntesis de los materiales componentes de las estructuras solares selectivas, diseño y simulación de su comportamiento óptico, a su validación en condiciones similares a la aplicación final (a nivel de laboratorio y ensayos de campo). La caracterización estructural, química y de

estabilidad térmica y resistencia a la oxidación discurrirá en paralelo con el fin de optimizar los recubrimientos solares selectivos con mejores prestaciones y durabilidad.

The climatic change produced by the gas pollutants emissions and the greenhouse effect along to the short mid-term depletion of the energy fossil fuels make necessary the search of alternative energy sources, clean and renewable. Among them, the solar energy is one the best options due to the major availability to generate heat and electricity.

The goal of the present project is the development of new solar multilayered absorber coatings based on chromium and aluminum nitride (CrAlN). The good oxidation resistance and thermal stability of CrAlN, together with a nanostructured design will ensure a good optical performance (high absorbance and low emissivity) and increase their durability at high temperature. The increment of the working temperature ($T>550$ °C) will improve the efficiency and reduce the costs of the solar thermal power plants, make them more competitive. The High Power Impulse Magnetron Sputtering technique (HiPIMS) will be used for the preparation of the coatings. This recent innovation of the conventional magnetron sputtering technology allows increasing the film density and compactness thanks to an increased ionization of the plasma. These properties are interesting for the improvement of the adhesion to the substrate and decrease the thermal degradation. In addition to abovementioned strategy, other alternative configurations changing the nature of the material absorber (metal oxynitrides and carbides nanocomposites) would be tried.

The project will comprise all the stages, from the synthesis of the material components of the solar selective structures, design and simulation of the optical behaviour, to the validation in conditions similar to the final application (both in lab and field tests). The structural and chemical characterization, the evaluation of the thermal stability and oxidation resistance will run simultaneously with the aim of optimizing the solar absorber selective coatings with the best performance and durability.

Desarrollo de catalizadores y soportes para procesos de almacenamiento químico de energía neutros en CO_2 basados en líquidos orgánicos portadores de hidrógeno	**Development of catalysts and supports for CO_2 neutral chemical energy storage processes based on liquid organic hydrogen carriers**
MINISTERIO DE CIENCIA E INNOVACIÓN	UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN
Código/Code:	RTI2018-093871-B-I00
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	176.176 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	María del Carmen Jiménez de Haro

RESUMEN / ABSTRACT

El agotamiento de los combustibles fósiles (a corto y largo plazo) y el calentamiento global derivado del efecto invernadero son consecuencias del uso extensivo de estos combustibles. Por lo tanto, es muy conveniente utilizar y desarrollar energías renovables y así eliminar nuestra dependencia de los combustibles fósiles. Esto hace que el almacenamiento de energía producida por fuentes renovables (que son intermitentes) sea un objetivo importante de investigación. En proyectos anteriores, hemos trabajado en el estudio de nanomateriales y catalizadores para el almacenamiento de hidrógeno como vector de transporte y almacenamiento de energía (ciclo del H_2). En este nuevo proyecto, el grupo de investigación propone avanzar en la Implementación de líquidos orgánicos como portadores de hidrógeno (LOHC) como una forma prometedora de combinar los ciclos del CO_2 y del H_2 que conduzca a un almacenamiento de energía sostenible en un ciclo neutro en carbono. Pequeñas moléculas orgánicas, como el ácido fórmico o el metanol, se pueden usar para almacenar el H_2 (y la energía) proveniente de fuentes renovables. Estos combustibles alternativos se pueden quemar o usarse para generar H_2 que alimente directamente a una pila de combustible.

En este proyecto se llevarán a cabo investigaciones para la implementación de dos procesos relacionados con las tecnologías LOHC:

i) La descomposición térmica selectiva del ácido fórmico por catálisis heterogénea para la producción bajo demanda de hidrógeno exento de monóxido de carbono.

ii) La producción de hidrógeno por reformado de alcoholes (biometanol) en procesos fotocatalíticos heterogéneos. La catálisis desempeña un papel clave en la implementación de estos dos procesos. Por lo tanto, los principales objetivos y actividades del proyecto son el diseño racional y la preparación de catalizadores y

soportes para estudiar las relaciones composición-estructura desempeño en los dos procesos mencionados anteriormente. El enfoque innovador es la aplicación de técnicas asistidas por plasma, como la pulverización catódica para el crecimiento de películas delgadas, y los tratamientos con plasmas de oxidación, reducción y grabado, para el desarrollo de recubrimientos catalíticos nanoestructurados y nanopartículas soportadas. Se desarrollarán espumas de carbono poroso y catalizadores basados en Pd que incluyen Pd, Pd-C, Pd-B o Pd-Cu para el estudio de la reacción de descomposición de ácido fórmico. Se investigarán películas fotocatalíticas de TiO_2-TiO_x con Pt (y/o Au) como co-catalizadores para el foto-reformado de metanol.

The depletion of fossil fuels (in short and long term) and the global warming derived from greenhouse effect are consequences of the extensive use of these fuels. It is therefore highly desirable to use and develop renewable energies and so eliminate our dependence on fossil fuels. This makes the storage of energy produced by renewable sources (which are intermittent) an important target. In previous projects we have been working in the study of nanomaterials and catalysts for the storage of hydrogen as a vector of energy transport and storage (H_2 cycle). In this new project the research group propose to move into the implementation of the liquid organic hydrogen carriers (LOHC) as a promising way of combining the CO_2 and de H_2 cycles leading to a sustainable energy storage in a carbon neutral cycle.

Small organic molecules, like formic acid or methanol, can be used to store the H_2 (and energy) coming from renewable sources. These alternative fuels can be combusted themselves or be used to generate H_2 directly feeding a fuel cell.

Research will be conducted in this project to the implementation of two processes related to the LOHC technologies:

i) The selective low temperature decomposition of formic acid by heterogeneous catalysis to the on-demand production of carbon monoxide free hydrogen.

ii) The hydrogen production by reforming of alcohols (i.e. biomethanol) in heterogeneous photocatalytic processes.

Catalysis is playing the key role in the implementation of these lwo processes. Therefore the main objectives and activities in the project are the rational design and preparation of catalysts and supports to study composition-structure-performance relationships for the two above mentioned processes. The innovative approach is the application of plasma assisted techniques, like the magnetron sputtering for

thin film growth, as well as plasma treatments of oxidation, reduction and etching for the development of nanostructured catalytic coatings and supported nanoparticles. Porous carbon foam supports and Pd based catalysts including Pd, Pd-C, Pd-B or Pd-Cu will be developed for the study of the formic acid decomposition reaction. TiO_2-TiO_x photocatalytic films with Pt (and/or gold) as co-catalysts will be investigated for the photo-reforming of methanol.

Dispositivo optofluídico NIR para análisis de líquidos	**NIR Optofluidic device for liquid analysis**
MINISTERIO DE CIENCIA E INNOVACIÓN Financiado por la Unión Europea NextGenerationEU Plan de Recuperación, Transformación y Resiliencia AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	PDC2021-121379-I00
Periodo/Period:	01-12-2021 / 30-11-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	144.900 €
Investigador responsable/Research head:	Francisco Yubero Valencia
Componentes/Research group:	Juan Pedro Espinós Manzorro, Ramón González García, Victor J. Rico Gavira, Agustín R. González-Elipe

RESUMEN / ABSTRACT

NIRFLOW es un proyecto I+D+i para la realización de una Prueba de Concepto en el que se plantea desarrollar un prototipo precomercial para análisis óptico en el infrarrojo cercano de fluidos en condiciones de flujo en entornos industriales relevantes. El proyecto se basa en varias innovaciones no contempladas en equipos comerciales basados en análisis NIR hoy en el mercado. De un lado, sustituir la óptica de análisis NIR convencional operada por espectrómetros NIR basados en redes de difracción o óptica de Fourier por una selección de la longitud de onda de análisis basada en combinaciones de filtros ópticos de paso alto y paso bajo variables de forma continua con respuesta sintonizada (centro y anchura de banda) a voluntad en el NIR. De otro lado, la celda optofluídica a desarrollar, operada en modo transflectancia, se caracteriza por tener camino óptico de análisis variable y sintonizable a los sobretonos de las absorciones características de las moléculas presentes en el fluido problema. De esta manera, los análisis estadísticos característicos de la espectroscopía NIR se van a ver apoyados por variables independiente (medidas correspondientes no sólo a variaciones de longitud de onda, sino también a distintos caminos ópticos de análisis), lo cual va a propiciar análisis estadísticos más robustos que los convencionales. Finalmente, el equipo se va a desarrollar con una concepción microfluídica de análisis automática, para su operación en remoto mediante tecnología wireless. Estas tres innovaciones hacen de NIRFLOW un proyecto I+D+i en el que parte de los conocimientos y uno de los desarrollos realizado en un proyecto de investigación previo del Plan Estatal (MAT2016-79866-R), parcialmente protegido con una patente, se pretende transferir a la sociedad a través del desarrollo de un equipo precomercial que demuestre sus capacidades de análisis en entornos operacionales significativos, en particular para el seguimiento de procesos de fermentación ligados a la producción de vinos.

NIRFLOW is a R+D+i Project for the realization of a Proof of Concept in which it is aimed to develop a pre-commercial prototype for the optical analysis in the near infrared of fluids in flow conditions in relevant industrial environments. The project is based on several innovations that are not implemented in conventional NIR apparatus in the market so far. First, to substitute the conventional NIR optics mainly operated by spectrometers based on diffraction gratings or Fourier optics by a selection of the wavelength of analysis based on combinations of continuously variable short and long pass filters designed to tune a NIR passband (regarding center and width). Second, to develop an optofluidic cell, operated in transflectance mode, characterized by a tunable optical pathlength to optimize the info obtained by the different overtones of the characteristic molecules present in the fluid under analysis. This innovation will offer the possibility of more robust statistical analysis than conventional NIR spectroscopy operated with single optical pathlength. Finally, the prototype will be developed within a microfluidic approach with automate analysis concept, for its operation within a wireless remote technology. These three innovations make NIRFLOW a R&D+i project in which part of the knowledge and one of the developments done in previous research project from the Spanish Plan Estatal (MAT2016-79866-R), partially protected by a patent claim, is aimed to be transferred to the society through the development of a precommercial prototype that showed ability of analysis in industrial operational environments, in particular to follow the evolution of fermentation processes linked to wine production.

Modelado e implementación de la técnica Freeze-Casting: gradientes de porosidad con un equilibrio tribo-mecánico y comportamiento celular electro-estimulado	**Modeling and implementation of the freeze casting technique: gradients of porosity with a tribo-mechanical equilibrium and electro-stimulated cellular behavior**
Junta de Andalucía	Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa
Código/Code:	US-1259771
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	90.000 €
Investigador responsable/Research head:	Yadir Torres Hernández (US) y Juan Carlos Sánchez López
Componentes/Research group:	Ana María Beltrán Custodio, Alberto Olmo Fernández, Paloma Trueba Muñoz, María de los Ángeles Vázquez Gámez

RESUMEN / ABSTRACT

El titanio comercialmente puro (Ti c.p.) y la aleación Ti6Al4V, son los biomateriales metálicos con el mejor pronóstico para la reparación clínica del tejido óseo. Sin embargo, a pesar de sus ventajas, 5-10% de los implantes fallan durante los cinco años post-implantación. Éstos se asocian fundamentalmente al apantallamiento de tensiones (diferencias de rigidez entre el implante-hueso), el empleo de criterios de diseño (fractura y fatiga) no adecuados para biomateriales, a los fenómenos de tribo-corrosión en condiciones de servicio y a los problemas que ocurren en la intercara (micromovimientos y/o presencia de bacterias) que limitan la capacidad de oseointegración. En este proyecto se propone fabricar e implementar un dispositivo sencillo y económico para obtener cilindros con porosidad controlada (gradiente) y alargada mediante la técnica de congelación dirigida. Se desarrollarán modelos de elementos finitos para estimar el crecimiento geométrico de las dendritas de hielo y el comportamiento mecánico de los cilindros porosos (distribución de esfuerzos y deformaciones), usando radiografías en tiempo real del proceso de congelación dirigida, así como los parámetros que caracterizan la microestructura (proporción, tamaño, morfología de la porosidad) y el comportamiento a compresión (rigidez y límite de fluencia). Además, se plantea la generación de patrones de rugosidad superficial mediante el bombardeo de iones, encaminados a mejorar la unión intima entre el implante y el tejido óseo. Por otra parte, se plantean protocolos in-vitro adecuados para evaluar la citotoxicidad, la adhesión, diferenciación y proliferación celular. Finalmente, se desarrollará un sistema de medida de bio-impedancia que permita racionalizar la influencia de la porosidad, el acabado superficial y los estímulos eléctricos en el comportamiento in-situ de osteoblastos. En este contexto, el objetivo principal es fabricar cilindros con una porosidad controlada y su superficie modificada, que permita garantizar un mejor equilibrio biomecánico, tribo-corrosivo y biofuncional (in-growth y oseointegración del tejido óseo y el implante).

Commercial pure Titanium (c.p. Ti) and Ti6Al4V alloy are metal biomaterials with the best properties for clinical repair bone tissue. However, despite their advantages, 5-10 % of implants fail during the five years post-implantation. They are mainly associated with stress shielding (difference stiffness between bone and implant), the use of design criteria (fracture and fatigue) not suitable for biomaterials, the tribo-corrosion phenomena in service conditions and the interface problems (micro-movements and / or the presence of bacteria) that limit the capacity of osseointegration. This project proposes the manufacture and implementation of a simple and economical device to obtain cylinders with controlled (gradient) and elongated porosity by the freeze casting technique. Finite element models will be developed to estimate the geometric growth of the ice dendrites and the mechanical behaviour of the porous cylinders (distribution of stresses and deformations), using real-time radiographs of the directed freezing process, as well as the parameters that characterize the microstructure (amount, size and morphology of porosity) and compression behaviour (stiffness and yield strength). In addition, the generation of surface roughness patterns by ion sputtering is proposed, with the aim to improve the close bond between the implant and the bone tissue.

Furthermore, suitable in-vitro protocols are proposed to evaluate cytotoxicity, adhesion, differentiation and proliferation cell. Finally, a bio-impedance measuring system will be developed in order to rationalize the influence of porosity, finished surface and electrical stimulus on the in-situ behaviour of osteoblasts. In this context, the main objective is to manufacture cylinders with a controlled porosity and modified surface, with enhanced biomechanical, tribo-corrosive and biofunctional balance (in-growth and osseointegration of the bone tissue and the implant).

Plasmas atmosféricos de arco deslizante para la producción sostenible de amoniaco e hidrógeno (ARCPLAS)	**Atmospheric Pressure Gliding-arc Plasmas for the Sustainable Production of Ammonia and Hydrogen (ARCPLAS)**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	US-1380977
Periodo/Period:	01-02-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	77.987 €
Investigador responsable/Research head:	José Cotrino Bautista y Ana María Gómez Ramírez
Componentes/Research group:	Rafael Álvarez Molina, José Javier Brey Sánchez (US), Jesús Cuevas Maraver (US), Alberto Palmero Acebedo, Juan F. Rodríguez Archilla (US)

RESUMEN / ABSTRACT

El proyecto "*Plasmas atmosféricos de arco deslizante para la producción sostenible de amoniaco e hidrógeno*", ARCPLAS en adelante, pretende el desarrollo de procesos de transformación química de gases mediante tecnologías de plasma atmosférico que usan la electricidad como vector energético directo. En concreto se persigue la puesta a **punto de un reactor de Plasmas Atmosféricos de Arco Deslizante (PAAD) para inducir dos procesos de gran impacto industrial y medioambiental, como son la síntesis de amoniaco (NH_3) y la producción de hidrógeno (H_2).** El amoniaco es la sustancia base de los fertilizantes usados en agricultura, y su demanda aumenta conforme las necesidades de alimentación mundiales. En cuanto al hidrógeno, de sobra es conocido que el camino hacia una economía basada en dicho combustible es uno de los retos del siglo XXI. ARCPLAS propone, en una primera etapa, desarrollar la tecnología PAAD mediante el diseño, construcción, modelización y puesta a punto de un reactor de arco

deslizante. La complejidad de los procesos básicos implicados en este tipo de reactores implicará un estudio fundamental de su respuesta eléctrica y de los fenómenos de transporte de masa y carga, así como una caracterización exhaustiva y diagnosis del plasma en función de parámetros como flujo de gases, interacción entre especies excitadas, tiempo de residencia, características químicas de los gases implicados y otros parámetros básicos de operación. Tanto la caracterización experimental como teórica del reactor, esta última llevada a cabo mediante métodos computacionales, serán fundamentales para su correcto funcionamiento y optimización de los procesos propuestos. En una segunda etapa se abordará el estudio de las reacciones de obtención de H_2 y NH_3, con el objetivo de maximizar su rendimiento químico, así como el rendimiento energético del reactor. Finalmente, en una última etapa del proyecto, se explorarán posibles modificaciones sobre el modelo de reactor PAAD desarrollado, contemplándose el efecto de la incorporación de materiales piezoeléctricos para inducir fenómenos de emisión secundaria de electrones, la modificación de las características superficiales de los electrodos o la geometría del sistema a fin de propiciar en el futuro una mejora en el rendimiento de los procesos estudiados.

The project "*Atmospheric Gliding-arc Plasmas for the Sustainable Production of Ammonia and Hydrogen*", hereinafter ARCPLAS, **aims to develop gas chemical transformation processes through atmospheric pressure plasma technologies that use electricity as a direct energy vector. Specifically, the objective is to fine-tune a Plasma Atmospheric Gliding Arc Reactor (PAAD) to induce two processes of great industrial and environmental impact, such as the synthesis of ammonia (NH_3) and the production of hydrogen (H_2).** Ammonia is the base substance of fertilizers used in agriculture, and its demand is increasing in line with world food needs. Regarding hydrogen, it is well known that the path towards an economy based on it is one of the challenges of the 21st century. ARCPLAS proposes, in a first stage, to develop PAAD technology through the design, construction, modelling and commissioning of a gliding arc reactor. The complexity of the basic processes involved in this type of reactors will require a fundamental study of their electrical response and mass and charge transport phenomena, as well as an exhaustive characterization and diagnosis of the plasma based on parameters such as gas flow, interaction between excited species, residence time, chemical characteristics of the gases involved and other basic operating parameters. Both the experimental and theoretical characterization of the reactor, the latter carried out using computational methods, will be essential for its correct operation and optimization of the processes. In a second stage, the study of the reactions to obtain H2 and NH3 will be addressed, with the aim of maximizing their chemical yield, as well as the energy yield of the reactor. Finally, possible modifications of the PAAD reactor will be explored, contemplating the effect of the incorporation of piezoelectric materials to induce secondary electron emission phenomena, the modification of the surface of the electrodes or the geometry of the system in order to promote an improvement in the performance of the processes studied.

Tecnología de plasma para el desarrollo de una nueva generación de conductores de huecos en celdas solares de perovskita. PlasmaCells	**Plasma technology for the development of a new generation of hole transport layers in perovskite solar cells**
Junta de Andalucía	Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa
Código/Code:	US-1263142
Periodo/Period:	01-02-2020 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	30.000 €
Investigador responsable/Research head:	Juan Ramón Sánchez Valencia
Componentes/Research group:	Ángel Barranco Quero, Juan Pedro Espinós Manzorro, Cristina Rojas Ruiz, José Cotrino Bautista

RESUMEN / ABSTRACT

Las celdas solares (CSs) de tercera generación son dispositivos nanotecnológicos que convierten directamente la luz solar en electricidad y suponen el paradigma de la investigación en energías renovables de cuyo aprovechamiento dependerá el futuro energético del planeta. Recientemente, un ejemplo particular de CSs que contienen una perovskita de haluro organometálico como absorbedor de luz han centrado la atención de la comunidad científica debido, ante todo, a su alta eficiencia y bajo coste. Estas características las convierten en una alternativa prometedora a las celdas actuales (de Si y calcogenuros). Sin embargo, para que la realización final y comercial de las celdas de perovskita sea posible es necesario que alcancen una mayor estabilidad, durabilidad y reproducibilidad. Los avances más importantes alcanzados se han debido a la intensa investigación sobre los elementos que integran esta CS: conductor de electrones, perovskita y conductor de huecos. En concreto, este último elemento ha tenido una importancia crucial en su evolución tras la implementación de los conductores de huecos en estado sólido.

PlasmaCells persigue abordar por primera vez la síntesis de una nueva familia de conductores de huecos por técnicas de vacío y plasma. Estas metodologías son escalables industrialmente y presentan grandes ventajas con respecto a las metodologías en disolución (las más usadas), entre las que destacan: su alta versatilidad, control de composición y microestructura, bajo coste, que son respetuosas con el medio ambiente ya que no precisan disolventes, no producen emisiones contaminantes y son compatibles con la tecnología actual de semiconductores.

El objetivo principal de PlasmaCells es la integración de estos nuevos conductores de huecos procesados por plasma en CSs de perovskita. La importancia del proyecto se

basa en resultados recientes obtenidos por el Investigador Principal (IP) que demuestran que la aproximación propuesta puede ser una de las vías más prometedoras para el aumento de la estabilidad, durabilidad y reproducibilidad de estas CSs, que actualmente suponen el cuello de botella que impide su industrialización. Cabe destacar que no existe en la bibliografía ningún ejemplo sobre esta aproximación sintética para el desarrollo de conductores de huecos. Se espera que esta oportunidad permita demostrar las ventajas y versatilidad de esta metodología innovadora en un campo de alto impacto, que se enmarca dentro de las áreas prioritarias RIS3 Andalucía y en el PAIDI 2020 de crecimiento sostenible, eficiencia energética y energías renovables.

Third generation solar cells (SCs) are nanotechnological devices that directly convert sunlight into electricity and represent the paradigm of research in renewable energies, the use of which will depend on the energy future of the planet. Recently, a particular example of SCs containing an organometallic halide perovskite as a light absorber have attracted the attention of the scientific community due, above all, to their high efficiency and low cost. These characteristics make them a promising alternative to current cells (Si and chalcogenides). However, for the commercial realization of perovskite cells, it is necessary to achieve greater stability, durability and reproducibility. The most important advances have been achieved due to the intense research on the elements that integrate a SC: electron transport layer, perovskite and hole transport layer. Specifically, this latter element has been crucial for its evolution after the implementation of solid state hole conductors.

PlasmaCells pursuits to address for the first time the synthesis of a new family of hole transporters by vacuum and plasma techniques. These methodologies are industrially scalable and have great advantages over solution methodologies (the most used), among which stand out: their high versatility, composition and microstructural control, low cost, are environmental friendly since they do not require solvents, do not produce polluting emissions and are compatible with current semiconductor technology.

The main objective of PlasmaCells is the integration of these new plasma-processed hole transport layers into perovskite SCs. The importance of the project is based on recent results obtained by the Principal Investigator (PI) that demonstrate that the proposed approach may be one of the most promising ways to increase the stability, durability and reproducibility of these SCs, which currently represent the bottleneck that prevents their industrialization. It should be noted that there is no example in the literature of this synthetic approach for the development of hole transporters. It is expected that this opportunity will allow to demonstrate the advantages and versatility of this innovative methodology in a high-impact field, which is framed within the priority areas RIS3 Andalucía and in the PAIDI 2020 of sustainable growth, energy efficiency and renewable energies.

Nuevos recubrimientos nanoestructurados para absorción eficiente de la radiación solar en dispositivos de concentración	**New nanostructured coatings for efficient absorption of solar radiation in concentrated devices**
Código/Code:	P18-RT-2641
Periodo/Period:	01-02-2020 / 31-03-2023
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	102.268 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Cristina Rojas Ruiz, Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

La mejora de los materiales empleados en los dispositivos usados en el campo de las energías renovables permitirá incrementar la eficiencia de los mismos haciéndolos más competitivos y rentables. El presente proyecto pretende desarrollar nuevos recubrimientos absorbedores selectivos de la energía solar aptos para trabajar a temperaturas superiores a las posibles con los materiales actualmente en uso en dispositivos de concentración solar térmica (500 °C en vacío – media concentración; 800 °C al aire – alta concentración). Los sistemas serán preparados en forma de multicapas por la novedosa tecnología de pulverización catódica donde los materiales son evaporados mediante impulsos de alta energía (HiPIMS - High Power Impulse Magnetron Sputtering). Los materiales preparados deberán cumplir los requisitos ópticos y de estabilidad química para soportar las condiciones de alta irradiación solar y temperaturas de trabajo. Este ambicioso proyecto se llevará a cabo mediante la colaboración de dos grupos de investigación pertenecientes al Instituto de Ciencia de Materiales de Sevilla CSIC-ICMS (grupo TEP958) y a la plataforma solar de Almería CIEMAT-PSA (Grupo TEP247). El grupo CSIC-ICMS se encargará del diseño, preparación y caracterización de los recubrimientos. Por su parte CIEMAT-PSA, diseñará y desarrollará los ensayos de campo, validando los recubrimientos en condiciones de trabajo similares a las de la aplicación final en términos de flujo solar concentrado incidente y temperaturas de operación. Dichos ensayos incluirán tanto determinación de parámetros térmicos y ópticos en condiciones nominales de operación, así como ciclado térmico de alta frecuencia (tratamiento térmico y envejecimiento).

The improvement of the materials employed in the devices used in the renewable energy sector will enable to increase the efficiency of these systems to become more competitive and profitable. The current project aims to develop new solar selective

coatings able to operate at temperatures beyond the working temperature limits of the materials currently being used in concentrated solar systems (500 °C in vacuum- mid concentration; 800 °C in air –high concentration). The systems will be prepared in the form of multilayers using the novel technology of magnetron sputtering where the materials are evaporated by means of high energy pulses (HiPIMS - High Power Impulse Magnetron Sputtering). The developed materials should fulfill the optical requirements and thermal stability to withstand the high solar irradiance flux and working temperatures. This project will be carried out through the collaboration of two research groups belonging to the "Instituto de Ciencia de Materiales de Sevilla", CSIC-ICMS (TEP958 group) and the "Plataforma Solar de Almería", CIEMAT-PSA (TEP247 group). The ICMS-CSIC group will perform the design, preparation and characterization of the coatings. Meanwhile, the CIEMAT-PSA group will be in charge of designing the bench tests, validating the coatings in working conditions similar to the final application in terms of high incident solar flux and operation temperatures. Such tests will include both the determination of thermal and optical parameters in nominal operating conditions, as well as the thermal cycling at high frequency (thermal treatment and aging).

Nanoscopías y Espectroscopías integradas para el análisis en la nano-escala de nuevos materiales funcionales	**Integrated nanoscopies and spectroscopies for the analysis of novel functional materials at the nano-scale**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional	Andalucía se mueve con Europa
Código/Code:	P20_00239
Periodo/Period:	05-10-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	46.695 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	María del Carmen Jiménez de Haro

RESUMEN / ABSTRACT

El desarrollo de los nanomateriales y materiales funcionales, así como sus aplicaciones nanotecnológicas, vienen determinados por las capacidades actuales para la caracterización de la microestructura, la composición y las propiedades de los materiales en la nano-escala. El proyecto propone potenciar una investigación de frontera en la caracterización microestructural de materiales. Se integrarán las técnicas nanoscópicas y espectroscópicas, ligadas a la microscopía electrónica (sonda de electrones), con las técnicas asociadas a las sondas de fotones (rayos-X) y de haces de iones (técnicas IBA en

general). La caracterización se asociará a materiales funcionales seleccionados de alto interés actual en la temática de recubrimientos y láminas delgadas en las que el equipo de trabajo es experto.

Será objetivo central el desarrollo y aplicación de manera integrada de las técnicas disponibles con múltiples sondas, tanto en el ICMS, como en otros centros de las Universidades de Sevilla (CITIUS, CNA) y Cádiz (servicios centrales). Igualmente, a través de colaboraciones y solicitudes de medidas se tendrá acceso a otras instalaciones internacionales.

En el proyecto se dispondrá de materiales seleccionados en dos tecnologías emergentes: i) Láminas delgadas y recubrimientos nanoporosos que estabilizan gases a ultra-alta densidad y presión. ii) Catalizadores para los procesos de almacenamiento y generación de hidrógeno en líquidos orgánicos portadores de hidrógeno (LOHCs). La caracterización avanzada que se propone contribuirá a la comprensión fundamental de las relaciones síntesis-microestructura-propiedades con el objetivo de alcanzar un diseño racional de nuevos materiales funcionales en las líneas seleccionadas. El proyecto incide directamente en las tecnologías facilitadoras o emergentes como son “la nanotecnología” y “los materiales avanzados”. Incide también en los retos sociales y objetivos RIS3 de Andalucía en relación al almacenamiento de energías renovables.

The current development of nanomaterials and functional materials in general, as well as their nanotechnological applications, are determined to a large extent by the current capacities on the characterization of microstructure, composition and even properties of the materials at the nano-scale. The project is proposed to promote an innovative research in the microstructural characterization of materials. The nanoscopic and spectroscopic techniques linked to the electron microscopes (electron beam probe), will be integrated together with techniques associated with photon beam (X-rays) and ion beam (IBA techniques) probes. This characterization will be associated with selected functional materials, also within advanced research lines of high current interest, in the topic of coatings and thin films in which the work team has strong experience.

The development and application of the available techniques with multiple probes will be a first central objective, both in the ICMS and in other centers of the Universities of Seville (CITIUS, CNA) and Cádiz (TEM central services). Likewise, through collaborations and measurement time applications, access to other international facilities will be achieved. In the project, selected materials will be available in two emerging technologies: i) Nanoporous thin films and coatings that stabilize gases at ultra-high density and pressure. ii) Catalysts for hydrogen storage and on demand hydrogen generation through the use of liquid organic hydrogen carriers (LOHCs). The advanced characterization proposed in the nano-scale will contribute to the fundamental understanding of the synthesis-microstructure-properties relationships with the final objective of achieving a rational design of new functional materials in the selected priority lines. The project has a direct impact on enabling or emerging technologies such as "nanotechnology" and "advanced materials", as well as on the Andalusian societal challenges and RIS3 objectives in relation to the storage of renewable energies "Topic: Hydrogen and fuel cells".

Nanorecubrimientos dieléctricos para dispositivos electrónicos Flexibles por tecnología de plasma (FLEXDIELEC)	**Dielectric Nanocoatings for Flexible Electronic Devices by Plasma Technology (FLEXDIELEC)**
Junta de Andalucía	Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa
Código/Code:	EMERGIA20_00346
Periodo/Period:	01-09-2021 / 30-08-2025
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	256.000 €
Investigador responsable/Research head:	Francisco Javier Aparicio Rebollo

RESUMEN / ABSTRACT

Dadas sus características físicas y mecánicas de la tecnología de dispositivos electrónicos flexibles emergente combina estructuras multicapas de láminas delgadas flexibles, nanomateriales 2D, o nanoconductores 1D, como son los nanotubos de carbono y los nanohilos. Sin embargo, estos presentan diferentes limitaciones relacionadas con su degradación frente a agentes ambientales e incompatibilidad con las técnicas de fabricación convencionales más presentes a nivel industrial. El proyecto FlexDielec persigue el desarrollo de una nueva generación de materiales dieléctricos para el desarrollo de dispositivos electrónicos flexibles avanzados, superando estas limitaciones. Con este fin, se empleará una técnica pionera de plasma remotos, desarrollada por el IP, que regula en un amplio rango la composición y propiedades de nanocomposites orgánicos funcionales. Esta es una metodología de vía seca (ausencia de disolventes) y a temperatura ambiente, lo que asegura su completa compatibilidad con el uso de sustratos sensibles, como muchos de los que tienen mayores perspectivas de implementación en campo de la electrónica flexible (materiales poliméricos, tejidos, papel, nanomateriales 2D, nanofibras orgánicas…).

Due to its physical and mechanical characteristics, the emerging technology of flexible electronic devices combines multilayer structures of flexible thin films, 2D nanomaterials, or 1D nanoconductors, such as carbon nanotubes and nanowires. However, these present different limitations related to their degradation against environmental agents and incompatibility with the conventional manufacturing techniques. FLEXDIELEC pursues the development of a new generation of dielectric materials for the development of advanced flexible electronic devices, overcoming these limitations. To this end, a pioneering remote plasma technique will be used, developed by the IP, which regulates the composition and properties of functional organic nanocomposites over a wide range, will be used. This is a dry and room temperature

method that ensures complete compatibility with sensitive substrates, such as those with high prospects for implementation in the field of flexible electronics (polymeric materials, fabrics , paper, 2D nanomaterials, organic nanofibers…).

Funcionalización superficial y modelos de difusión de factores de germinación en semillas tratadas con plasmas PLASMASEED	**Surface functionalization and diffusion models of germination factors in plasma-treated seeds**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	US-1381045
Periodo/Period:	01-01-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	79.866 €
Investigador responsable/Research head:	María del Carmen López Santos y Antonio Prados Montaño (US)
Componentes/Research group:	Agustín Rodríguez González-Elipe, Francisco Yubero Valencia

RESUMEN / ABSTRACT

PLASMASEED aborda la inclusión de la tecnología de vacío y plasma para la funcionalización superficial de semillas como una estrategia eficaz y limpia para que los cultivos sean menos dependientes de los cambios del entorno. Se pretenden analizar los factores y mecanismos básicos que inciden en la mejora de la germinación tratando las semillas desde una aproximación multidisciplinar que combina conceptos básicos de biofísica, caracterización avanzada y procesado por vacío y plasma. El efecto de campos eléctricos asociados a los plasmas y las características físico-químicas de estos, la influencia de la difusión de otros factores de germinación además del agua (oxígeno, luz, etc.), la difusión de nutrientes como especies nitratos u otras de interés para la germinación, etc., son factores experimentales que se modelizan usando procedimientos de Monte Carlo y mecánica estadística para proponer modelos holísticos de difusión de factores de germinación a través de las membranas de semillas y de la influencia de los tratamientos superficiales con técnicas de plasma para modificar y / o controlar tales procesos.

PLASMASEED addresses the inclusion of vacuum and plasma technology for the surface functionalization of seeds as an effective and clean strategy to make crops less dependent on environmental changes. The aim is to analyze the basic factors and

mechanisms that affect the improvement of germination by treating the seeds from a multidisciplinary approach that combines basic concepts of biophysics, advanced characterization and vacuum and plasma processing. The effect of electric fields associated with plasmas and their physical-chemical features, the influence of the diffusion of other germination factors besides water (oxygen, light, etc.), the diffusion of nutrients such as nitrates or other species of interest for germination, etc., are experimental factors that are simulated using Monte Carlo procedures and statistical mechanics to propose holistic models of diffusion of germination factors through seed membranes and the influence of surface treatments by plasma techniques to modify and/or control such processes.

Recubrimientos termocrómicos inteligentes para la climatización eficiente y el control ambiental (TOLERANCE)	**Smart thermochromic coatings for smart windows and environmental control (TOLERANCE)**
Código/Code:	P18-RT-2641
Periodo/Period:	01-01-2020 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	119.800 €
Investigador responsable/Research head:	Ángel Barranco Quero y Alberto Palmero Acebedo
Componentes/Research group:	Ana María Gómez Ramírez, Juan Ramón Sánchez Valencia, Víctor J. Rico Gavira, Rafael Álvarez Molina, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Ana Isabel Borrás Martos, Agustín R. González-Elipe

RESUMEN / ABSTRACT

La Agencia Internacional de la Energía considera que el uso sistemático de procedimientos autónomos de control ambiental representa una de las mejores apuestas tecnológicas para reducir el consumo energético asociado a la climatización de edificios (más del 40% del consumo global en países desarrollados, muy superior al porcentaje debido al transporte), reduciendo el impacto ambiental y mejorando además el confort habitacional. TOLERANCE persigue introducir y desarrollar en Andalucía la tecnología de los recubrimientos termocrómicos como elemento inteligente y autónomo de control

de la irradiación solar en edificios. El interés de la propuesta se centra en nichos de aplicación como el cerramiento de edificios, el mobiliario urbano, la mejora de sistemas de producción de agua caliente sanitaria o la mejora de invernaderos. Un recubrimiento termocrómico se caracteriza por transmitir todo el espectro solar a bajas temperaturas y reflejar selectivamente parte de éste (el infrarrojo) a altas temperaturas. En esta línea, el proyecto propone diversas acciones de I+D para el desarrollo de capas delgadas con composición VO2, óxido termocrómico caracterizado con una temperatura de transición cercana a la temperatura ambiente, sobre vidrio y plásticos mediante técnicas escalables industrialmente, así como su nanoestructuración, dopado e integración en sistemas multicapas a fin de mejorar sus características y prestaciones multifuncionales.

The International Energy Agency considers that the systematic use of autonomous procedures for environmental control is one of the best technological approaches to minimize the energy employed to cool down buildings and other urban structures (it represents more than 40% of the global energy use in developed countries, much above the use in transportation, for instance), thus reducing the environmental impact and improving human comfort. TOLERANCE aims at introducing and developing a technology based on thermochromic materials in Andalusia as a smart and autonomous element to control the penetration of solar radiation in buildings. This project focusses on various applications such as smart windows in buildings and urban furniture, improvement of sanitary water systems or environmental control in greenhouses. While at low temperatures, a thermochromic coating transmits most solar spectrum, it selectively filters out the infrared region of this spectrum at high temperatures. In this research, TOLERANCE proposes several R+D actions to grow thin films with composition VO2, a thermochromic oxide with transition temperature near room temperature, on glass and plastic by means of industrial scalable techniques, as well as its nanostructuration, doping and integration in multilayer systems to improve its features and multifunctional properties.

Prototipo para la detección por imagen de contaminantes emergentes en aguas residuales mediante nanotecnología y deep learning (nanovision)

Junta de Andalucía

Unión Europea
Fondo Europeo de Desarrollo Regional

Andalucía se mueve con Europa

Código/Code:	6079
Periodo/Period:	31-10-2019 / 31-07-2021
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	70.117 €
Investigador responsable/Research head:	Ángel Barranco Quero

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de electrolizadores más eficientes y competitivos basados en electrodos preparados mediante pulverización catódica

Periodo/Period:	2020 / 2021
Entidad Financiadora:	Ayuda a la Investigación "Fundación Domingo Martínez"
Importe total/Total amount:	50.000 €
Investigador responsable/Research head:	Antonio de Lucas Consuegra (Universidad Castilla La Mancha)
Componentes/Research group:	Agustín R. González

Estudios preliminares para la aplicación de nanocomposites sólido-gas como blancos sólidos en estudios de reacciones nucleares y alta densidad de energía (PRE-TARGETS)

Periodo/Period:	1-07-2021 / 31-12-2022
Entidad Financiadora:	CSIC (Intramural)
Importe total/Total amount:	14.000 €
Investigador responsable/Research head:	Asunción Fernández Camacho

Ondas acústicas superficiales y su transferencia para aplicaciones de relevancia industrial

Periodo/Period:	1-02-2021 / 31-01-2024
Entidad Financiadora:	CSIC (Intramural)
Importe total/Total amount:	47.640 €
Investigador responsable/Research head:	Agustín R. González-Elipe, Francisco Yubero Valencia

Nanostructures with atomic precision for next generation energy harvesting devices

Código/Code:	LINKA20346
Periodo/Period:	01-01-2021 / 31-12-2022
Organismo Financiador/Financial source:	CSIC
Importe total/Total amount:	16.093 €
Participantes del ICMS:	Ángel Barranco Quero, Ana Isabel Borrás Martos, Xabier García Casas, Víctor J. Rico Gavira, Agustín R. González-Elipe

Sensores optofluídicos y electroquímicos 1D preparados mediante técnicas de PVD a ángulo rasante

Código/Code:	201860E050
Periodo/Period:	01-06-2018 / 31-05-2021
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	223.574 €
Investigador responsable/Research head:	Agustín R. González-Elipe

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Goniómetro para medidas de ángulo de contacto de superficies en ambiente controlado (IE_19_142 USE)

Financia: Junta de Andalucía
Importe Concedido: 82.000,00 €
Periodo: 29-12-2020 / 28-12-2022

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Nueva matriz multisensora óptica reconfigurable para detección de contaminantes en el agua

Periodo/Period:	02-12-2019 / 02-12-2021
Organismo Financiador/Financial source:	EXPACE ON BOARD SYSTEMS, S.L.
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Ángel Barranco Quero

Desarrollo de nuevos plasmas fríos para aceleración de germinación en condiciones de sequía

Periodo/Period:	02-12-2019 / 01-12-2021
Organismo Financiador/Financial source:	ARQUIMEA INGENIERIA, S.L.
Importe total/Total amount:	227.480 €
Investigador responsable/Research head:	Agustin R. González-Elipe

Nuevo sistema de propulsión espacial basado en el principio de Mach

Periodo/Period:	02-12-2019 / 30-12-2022
Organismo Financiador/Financial source:	ARQUIMEA INGENIERIA, S.L.
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	Agustín R. González-Elipe

Sistema de generación de hidrógeno a partir de gasoil
Periodo/Period: 09-04-2020 / 1-11-2022
Entidad Financiadora: INYECCIONES PLÁSTICAS MECACONTROL, S.L.
Importe total/Total amount: 45.000 €
Investigador responsable/Research head: Ana María Gómez Ramírez
Componentes/Research group: Agustín R. González-Elipe, José Cotrino Bautista, Manuel Oliva Ramírez

Caracterización superficial de membranas de ósmosis inversa
Periodo/Period: 01-05-2020 / 06-10-2022
Organismo Financiador/Financial source: ACCIONA AGUA, S.A.U.
Importe total/Total amount: 18.198,40 €
Investigador responsable/Research head: Juan Pedro Espinós Manzorro

Optimización del proceso de oxidación de anillos de Kovar para la fabricación de uniones vidrio-metal
Periodo/Period: 16-06-2019 / 15-01-2021
Organismo Financiador/Financial source: CLARTON HORN
Importe total/Total amount: 7.260 €
Investigador responsable/Research head: Asunción Fernández Camacho

Caracterización microestructural y química de materiales para avisadores sonoros
Periodo/Period: 19-01-2010 / 31-12-2021
Organismo Financiador/Financial source: CLARTON HORN
Importe total/Total amount: 45.950,11 €
Investigador responsable/Research head: Asunción Fernández Camacho

Proyecto de consultoría "PULSAR"
Periodo/Period: 19-10-2021 / 18-04-2022
Organismo Financiador/Financial source: ARQUIMEA CENTRO DE INVESTIGACIONES AVANZADAS, S.L.U.
Importe total/Total amount: 27.000 €
Investigador responsable/Research head: Agustín R. González-Elipe

Fabricación, caracterización y análisis de eficiencia de electrocatalizadores para electrolizadores de intercambio aniónico

Periodo/Period:	13-12-2021 / 31-12-2024
Organismo Financiador/Financial source:	H2B2 ELECTROLYSIS TECHNOLOGIES, S.L.
Importe total/Total amount:	178.620,20 €
Investigador responsable/Research head:	Francisco Yubero Valencia

PATENTES / PATENTS

Procedimiento de obtención de un material sólido con agregados gaseosos mediante pulverización catódica por magnetrón en condiciones estáticas o cuasiestáticas para reducir el consumo de gas
Inventores: Asunción Fernández Camacho, María del Carmen Jiménez de Haro, Dirk Hufschmidt, Vanda Cristina Fortio Godinho
Tipo de Patente: Nacional
Número de Solicitud: 19884840
Fecha Solicitud: 13 de mayo de 2021
Entidad Titular: Consejo Superior de Investigaciones Científicas

Dispositivo de eliminación y prevención de formación de hielo en superficies
Inventores: Ana Isabel Borrás Martos, Agustín R. González-Elipe, Víctor J. Rico Gavira, Aurelio García Valenzuela. Laura Montes Montañez
Tipo de Patente: Nacional
Número de Solicitud: 202130570
Fecha Solicitud: 18 de junio de 2021
Entidades Titulares: Consejo Superior de Investigaciones Científicas, Universidad de Sevilla

Energy harvesting device and self-powered sensor using the same
Inventores: Xabier García Casas, Ángel Barranco Quero, Ali Ghaffarinejad, Juan Ramón Sánchez Valencia, Ana Isabel Borrás Martos
Tipo de Patente: Nacional. Licenciada
Número de Solicitud: 21382786
Fecha Solicitud: 27 de agosto de 2021
Entidades Titulares: Consejo Superior de Investigaciones Científicas, Universidad de Sevilla

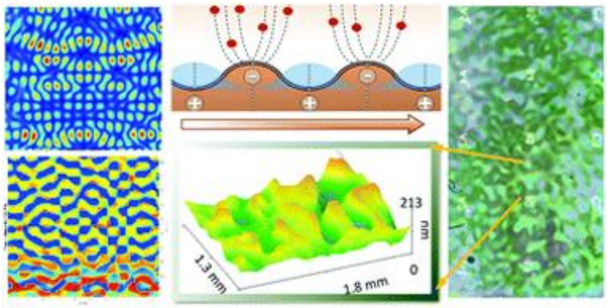
Porous Ionomer free layered metal alloy electrocatalyst electrode

Inventores: Jorge Gil Rostra, Agustín R. González-Elipe, Francisco Yubero Valencia, Ester López Fernández, Juan Pedro Espinós Manzorro
Tipo de Patente: Nacional
Número de Solicitud: 21383006
Fecha Solicitud: 5 de noviembre de 2021
Entidades Titulares: Consejo Superior de Investigaciones Científicas, Universidad de Castilla-La Mancha

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Patterning and control of the nanostructure in plasma thin films with acoustic waves: mechanical vs. electrical polarization effects

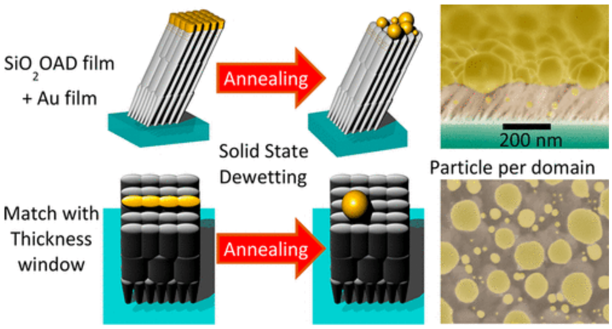
García-Valenzuela, A; Fakhouri, A; Oliva-Ramírez, M; Rico-Gavira, V; Rojas, TC; Álvarez, R; Menzel, SB; Palmero, A; Winkler, A; González-Elipe, AR
Materials Horizons, **8** (2021) 515-524
Febrero 2021 | **DOI:** 10.1039/D0MH01540G



Nanostructuration and 2D patterning of thin films are common strategies to fabricate biomimetic surfaces and components for microfluidic, microelectronic or photonic applications. This work presents the fundamentals of a surface nanotechnology procedure for laterally tailoring the nanostructure and crystalline structure of thin films that are plasma deposited onto acoustically excited piezoelectric substrates. Using magnetron sputtering as plasma technique and TiO_2 as case example, it is demonstrated that the deposited films depict a sub-millimetre 2D pattern that, characterized by large lateral differences in nanostructure, density (up to 50%), thickness, and physical properties between porous and dense zones, reproduces the wave features distribution of the generated acoustic waves (AW). Simulation modelling of the AW propagation and deposition experiments carried out without plasma and under alternative experimental conditions reveal that patterning is not driven by the collision of ad-species with mechanically excited lattice atoms of the substrate, but emerges from their interaction with plasma sheath ions locally accelerated by the AW-induced electrical polarization field developed at the substrate surface and growing film. The possibilities of the AW activation as a general approach for the tailored control of nanostructure, pattern size, and properties of thin films are demonstrated through the systematic variation of deposition conditions and the adjustment of AW operating parameters.

Solid-State Dewetting of Gold on Stochastically Periodic SiO_2 Nanocolumns Prepared by Oblique Angle Deposition

Oliva-Ramírez, M; Wang, D; Flock, D; Rico, V; González-Elipe, AR; Schaaf, P
ACS Applied Materials & Interfaces, **13** (2021) 11385-11395
Marzo 2021 | **DOI:** 10.1021/acsami.0c19327

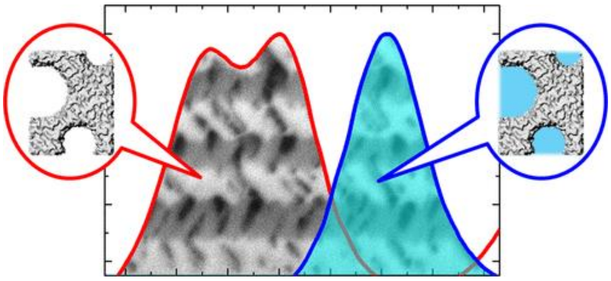


Solid-state dewetting (SSD) on patterned substrates is a straightforward method for fabricating ordered arrays of metallic nanoparticles on surfaces. However, a drawback of this procedure is that the patterning of substrates usually requires time-consuming and expensive two-dimensional (2D) fabrication methods. Nanostructured thin films deposited by oblique angle deposition (OAD) present at the surface a form of stochastically arranged periodic bundles of nanocolumns that might act as a patterned template for fabricating arrays of nanoparticles by SSD. In this work, we explore this concept and investigate the effect of three different types of OAD SiO_2 thin films on the SSD of Au deposited on their surface. We demonstrate that the size and spatial distribution of the particles can be tailored through the surface morphology of these OAD film substrates. It has been found that the SSD of the evaporated Au layer gives rise to a bimodal size distribution of particles. A majority of them appeared as mesoparticles with sizes.100 nm and the rest as nanoparticles with similar to 10 nm, respectively, located either on top of the nanocolumns following their lateral distribution (i.e., resulting from a patterning effect) or incorporated inside the open mesopores existing among them. Moreover, on the SiO_2-OAD thin films where interconnected nanocolumnar bundles arrange in the form of discrete motifs, the patterning effect gave rise to the formation of approximately one Au mesoparticle per motif, which is one of the assets of patterned SSD. The morphological, optical (i.e., plasmon resonance), and crystalline structural characteristics of Au mesoparticles suggest that the interplay between a discontinuous nanocolumnar surface acting as a template and the poor adhesion of Au onto SiO_2 are key factors for the observed template effect controlling the SSD on the surface of OAD thin films.

Form Birefringence in Resonant Transducers for the Selective Monitoring of VOCs under Ambient Conditions

Oliva-Ramírez, M; López-Santos, C; Berthon, H; Goven, M; Portoles, J; Gil-Rostra, J; González-Elipe, AR; Yubero, F
ACS Applied Materials & Interfaces, **13** (2021) 19148-19158
Abril 2021 | **DOI:** 10.1021/acsami.1c02499

In this work, we have developed a new kind of nanocolumnar birefringent Bragg microcavity (BBM) that, tailored by oblique angle deposition, behaves as a selective transducer of volatile



organic compounds (VOCs). Unlike the atomic lattice origin of birefringence in anisotropic single crystals, in the BBM, it stems from an anisotropic self-organization at the nanoscale of the voids and structural elements of the layers. The optical adsorption isotherms recorded upon exposure of these nanostructured systems to water vapor and VOCs have revealed a rich yet unexplored phenomenology linked to their optical activity that provides both capacity for vapor identification and partial pressure determination. This photonic response has been reproduced with a theoretical model accounting for the evolution of the form birefringence of the individual layers upon vapor condensation in nanopores and internanocolumnar voids. BBMs that repel water vapor but are accessible to VOCs have been also developed through grafting of their internal surfaces with perfluorooctyltriethoxysilane molecules. These nanostructured photonic systems are proposed for the development of transducers that, operating under environmental conditions, may respond specifically to VOCs without any influence by the degree of humidity of the medium.

Anisotropic Resistivity Surfaces Produced in ITO Films by Laser-Induced Nanoscale Self-organization

López-Santos, C; Puerto, D; Siegel, J; Macías-Montero, M; Florián, C; Gil-Rostra, J; López-Flores, V; Borrás, A; González-Elipe, AR; Solis, J

Advanced Optical Materials, **9** (2021) 2001086

Enero 2021 | **DOI:** 10.1002/adom.202001086

Highly anisotropic resistivity surfaces are produced in indium tin oxide (ITO) films by nanoscale self-organization upon irradiation with a fs-laser beam operating at 1030 nm. Anisotropy is caused by the formation of laser-induced periodic surface structures (LIPSS) extended over cm-sized regions. Two types of optimized structures are observed. At high fluence, nearly complete ablation at the valleys of the LIPSS and strong ablation at their ridges lead to an insulating structure in the direction transverse to the LIPSS and conductive in the longitudinal one. A strong diminution of In content in the remaining material is then observed, leading to a longitudinal resistivity $\rho L \approx 1.0\ \Omega{\cdot}cm$. At a lower fluence, the material at the LIPSS ridges remains essentially unmodified while partial ablation is observed at the valleys. The structures show a longitudinal conductivity two times higher than the transverse one, and a resistivity similar to that of the pristine ITO film ($\rho \approx 5 \times 10^{-4}\ \Omega{\cdot}cm$). A thorough characterization of these transparent structures is presented and discussed. The compositional changes induced as laser pulses accumulate, condition the LIPSS evolution and thus the result of the structuring process. Strategies to further improve the achieved anisotropic resistivity results are also provided.

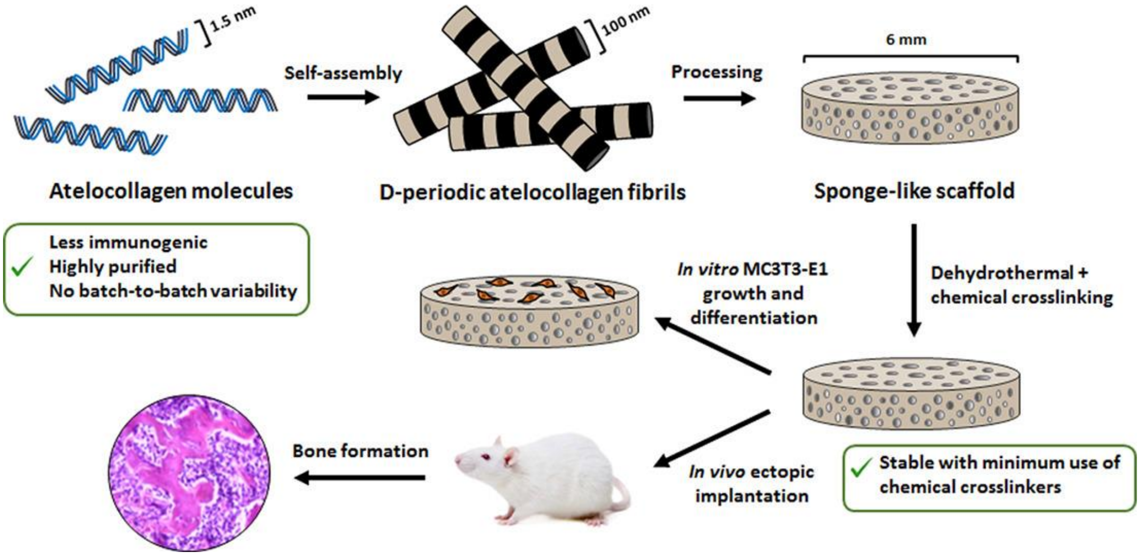
Photonic sensor systems for the identification of hydrocarbons and crude oils in static and flow conditions

Gil-Rostra, J; Quintero-Moreno, S; Rico, VJ; Yubero, F; Sanza, FJ; Casquel, R; Gallo-Valverde, E; Jara-Galán, ME; Sanz-Sanz, P; Holgado, M; González-Elipe, AR
Sensors and Actuators B-Chemical, **344** (2021) 130265
Octubre 2021 | **DOI:** 10.1016/j.snb.2021.130265

Identification of hydrocarbons and crude oils is typically carried out with samples that, taken from natural sources or refineries, must be brought to the laboratory for their analysis with rather sophisticated instruments. Alternatively, "in situ" procedures have been also developed for this purpose. In this work, we propose the use of a series of several sensor systems based on photonic transducers in the form of chips for the identification and classification of crude oils and hydrocarbons through the determination of their refractive index in the visible and absorption in the near infrared regions of the electromagnetic spectrum. Two of the photonic transducers rely on modifications of a Bragg microcavity and they monitor the changes in visible light interference phenomena that occur in response to the variation of the refractive index of oils. The third one, in the form of a dielectric mirror, monitors the near infrared absorption of crude oils and hydrocarbons through the recording of a transflectance spectrum. The capacity of these transducers for crude oil identification is proved by the analysis of a series of oils and distilled fractions that have been properly identified and classified as a function of their density and partition of long hydrocarbon chains. The three photonic transducers are operated with optical fibers and can be used in static and dynamic modes, this latter under conditions that are especially well-suited for "insitu" analysis of oil streams in real facilities. The proved resistance of the chips to high pressure and temperature conditions supports their suitability to withstand harsh working environments as those existing in extraction wells.

Sponge-like processed D-periodic self-assembled atelocollagen supports bone formation in vivo

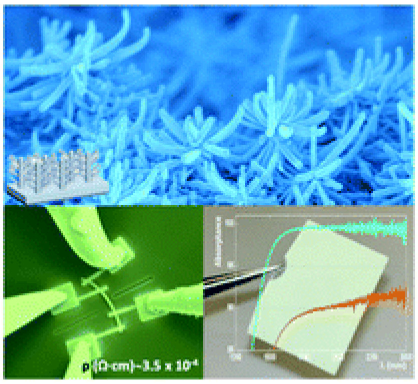
Borrego-González, S; Rico-Llanos, G; Becerra, J; Díaz-Cuenca, A; Visser, R
Materials Science & Engineering C-Materials for Biological Applications, **120** (2021) 111679
Enero 2021 | **DOI:** 10.1016/j.msec.2020.111679



Fibrous biopolymeric collagen extracted from animal tissues has been widely used for fabricating matrices for bone tissue engineering (BTE). However, animal extracted collagens can trigger immune reactions when implanted in vivo and the presence of native crosslinks leads to batch-to-batch variability. Atelocollagen, a monomeric form of collagen, is free of telopeptides, which are mainly responsible for the immunogenicity of collagen, and can self-assemble in vitro to obtain fibrils with the characteristic D-periodic staining pattern of native collagen. However, atelocollagen-based biomaterials have not extensively been studied and, hence, their suitability for BTE remains relatively unexplored. Besides, to stabilize collagen biomaterials, chemical and physical crosslinking are used, although chemical agents are cytotoxic while the physical methods yield a less effective crosslinking. A combination of physical and chemical crosslinking is a suitable alternative that has rarely been tested in BTE programs. In this work, a sponge-like biomaterial (DCol-S) was processed from D-periodic self-assembled atelocollagen and its stabilization was studied using the combination of a dehydrothermal treatment (DHT) and minimal glutaraldehyde (GTA) exposition crosslinking, to increase the resistance to degradation of the scaffold without a major effect on the biomaterial structure. The microstructural features of the final sponges were characterised and compared to a commercial biomaterial processed from native bovine collagen (Helistat (R), Integra Lifesciences, NJ, USA), demonstrating that a D-periodic nanostructure was obtained and maintained after processing of the sponges. MC3T3-E1 preosteoblast adhesion, proliferation and differentiation assays in vitro showed that DCol-S is biocompatible. Furthermore, intramuscular implantation of the biomaterials loaded with rhBMP-2 revealed that the double-crosslinked sponges were able to support ectopic bone formation, while sponges stabilised only with the DHT treatment were not. Altogether, these findings show that atelocollagen-based sponges stabilised with a DHT treatment followed by a mild GTA crosslinking are a suitable alternative to polymeric extracted collagen for BTE applications.

One-reactor vacuum and plasma synthesis of transparent conducting oxide nanotubes and nanotrees: from single wire conductivity to ultra-broadband perfect absorbers in the NIR

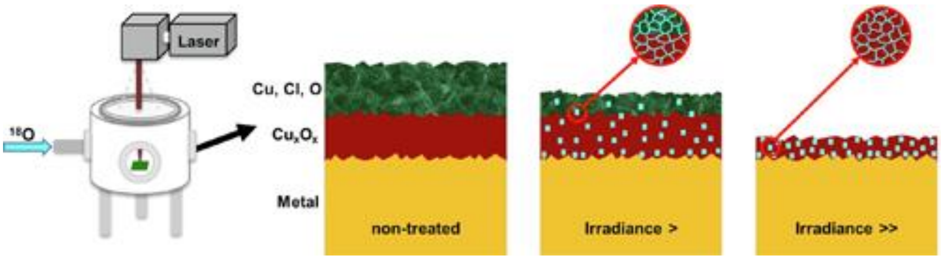
Castillo-Seoane, J; Gil-Rostra, J; López-Flores, V; Lozano, G; Ferrer, FJ; Espinós, JP; Ostrikov, K; Yubero, F; González-Elipe, AR; Barranco, A; Sánchez-Valencia, JR; Borrás, A
Nanoscale, **13** (2021) 13882-13895
Agosto 2021 | **DOI:** 10.1039/d1nr01937f



The eventual exploitation of one-dimensional nanomaterials needs the development of scalable, high yield, homogeneous and environmentally friendly methods capable of meeting the requirements for fabrication of functional nanomaterials with properties on demand. In this article, we demonstrate a vacuum and plasma one-reactor approach for the synthesis of fundamental common elements in solar energy and optoelectronics, i.e. the transparent conducting electrode but in the form of nanotube and nanotree architectures. Although the process is generic and can be used for a variety of TCOs and wide-bandgap semiconductors, we focus herein on indium doped tin oxide (ITO) as the most previously researched in previous applications. This protocol combines widely applied deposition techniques such as thermal evaporation for the formation of organic nanowires serving as 1D and 3D soft templates, deposition of polycrystalline layers by magnetron sputtering, and removal of the templates by simply annealing under mild vacuum conditions. The process variables are tuned to control the stoichiometry, morphology, and alignment of the ITO nanotubes and nanotrees. Four-probe characterization reveals the improved lateral connectivity of the ITO nanotrees and applied on individual nanotubes shows resistivities as low as $3.5 \pm 0.9 \times 10^{-4}\ \Omega$ cm, a value comparable to that of single-crystalline counterparts. The assessment of diffuse reflectance and transmittance in the UV-Vis range confirms the viability of the supported ITO nanotubes as random optical media working as strong scattering layers. Their further ability to form ITO nanotrees opens a path for practical applications as ultra-broadband absorbers in the NIR. The demonstrated low resistivity and optical properties of these ITO nanostructures open a way for their use in LEDs, IR shields, energy harvesting, nanosensors, and photoelectrochemical applications.

Novel procedure for studying laser-surface material interactions during scanning laser ablation cleaning processes on Cu-based alloys

Di Francia, E; Lahoz, R; Neff, D; Rico, V; Nuns, N; Angelini, E; Grassini, S
Applied Surface Science, **544** (2021) 178820
Abril 2021 | **DOI:** 10.1016/j.apsusc.2020.148820



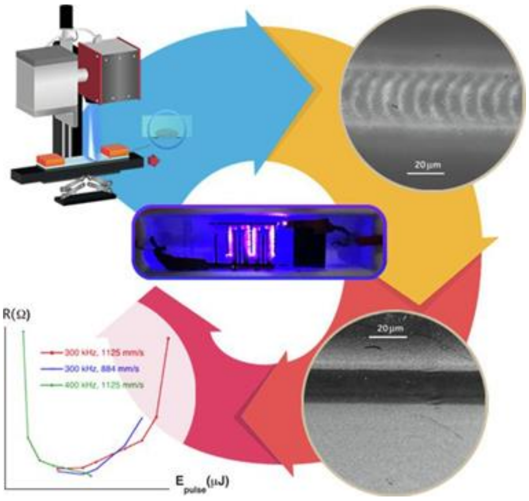
Laser ablation is an effective method to clean Cu-based alloys. A novel procedure of characterisation was developed involving ^{18}O isotopes evaluated by ToF-SIMS spectroscopy to assess the driving mechanisms of laser-surface interactions. The presence of re-oxidised compounds was detected, discerning between the oxygen from the corrosion layer and the one introduced by the interaction with the laser (that was generated in a controlled atmosphere of ^{18}O diluted in N_2). A set of samples treated with different laser conditions were characterised by FESEM and µRaman. The results have shown that re-oxidation phenomenon can occur and its selectivity depends on the laser conditions.

Laser-induced scanning transfer deposition of silver electrodes on glass surfaces: A green and scalable technology

Molina, R; Ertugrul, M; Larrea, A; Navarro, R; Rico, V; Yubero, F; González-Elipe, AR: De la Fuente, GF; Angurel, LA

Applied Surface Science, **556** (2021) 149673

Agosto 2021 | **DOI:** 10.1016/j.apsusc.2021.149673



A pulsed laser ablation backwriting technique with high repetitive rates is implemented for the fabrication of silver coatings on glass surfaces. This method enables geometrical constraint-free deposition of metallic coatings. These exhibit sufficiently low electrical resistance to be used as electrodes in dielectric barrier discharge (DBD) plasma elements. Ambient air deposition of metallic silver electrodes on standard glass slides is explored using a sub-ns UV laser source, combined with hybrid beam scanning methods. The green nature of the overall deposition process includes a preliminary irradiation scan to homogenise the target surface before the subsequent backwriting step. Metal transfer is achieved by combining two phenomena within a simple beam scanning process: LIRT (laserinduced reverse transfer) of silver from the target to the glass, with a partial and secondary LIFT (Laser-Induced Forward Transfer) of silver from the glass to the target. Appropriate selection of pulse energy and pulse repetition rates, beam scanning velocities and target motion enable the growth of sufficiently thick Ag deposits on glass with the required low electrical resistivity and nearly 2D constraint-free geometry. This method avoids the use of vacuum and liquids, resulting in a cheap, facile and green methodology for the deposition of silver electrodes onto transparent substrate surfaces.

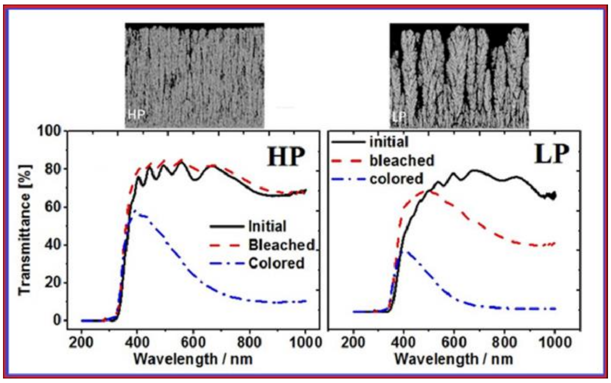
Electrochromic response and porous structure of WO_3 cathode layers

Louloudakis, D; Mouratis, K; Gil-Rostra, J; Koudoumas, E; Álvarez, R; Palmero, A; González-Elipe, AR

Electrochimica Acta, **376** (2021) 138049

Abril 2021 | **DOI:** 10.1016/j.electacta.2021.138049A

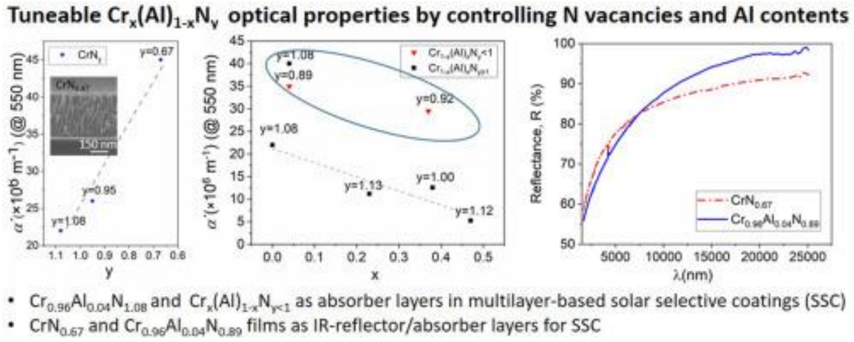
Maximizing the electrochromic response of tungsten oxide-based systems demands highly porous electrode layers that facilitate the incorporation of electrolyte cations during the reduction



process. In this work, amorphous and porous WO_3 thin films were grown on indium tin dioxide glass substrates by magnetron sputtering at oblique angles at two different plasma gas pressures. Remarkably, the film that showed higher porosity presented a worse electrochromic response in terms of durability, time response and charge density capacity. This result is analyzed and explained on the basis of the features of the porous structure of the films: While the typical nanostructure developed at low pressures possesses large and connected pore voids with few ramifications, the nanostructure generated at a higher pressure presents a rather sponge-like porous structure with numerous and small well-connected voids. A general discussion on the role of the porous structure and, particularly, on the accessible pore volume and area is carried out. It is concluded that not only the accessible pore volume, defining the volume of electrolyte that stays inside the layer, but also the accessible pore area, which defines the efficiency of the incorporation/release of Li^+ cations within the electrode material, determine the efficiency and reversibility of the electrochromic response.

High-temperature solar-selective coatings based on Cr(Al)N. Part 1: Microstructure and optical properties of CrN_y and $Cr_{1-x}Al_xN_y$ films prepared by DC/HiPIMS

Rojas, TC; Caro, A; Lozano, G.; Sánchez-López, JC
Solar Energy Materials and Solar Cells, **223** (2021) 110951
Mayo 2021 | **DOI:** 10.1016/j.solmat.2020.110951


Tuneable $Cr_x(Al)_{1-x}N_y$ optical properties by controlling N vacancies and Al contents

- $Cr_{0.96}Al_{0.04}N_{1.08}$ and $Cr_x(Al)_{1-x}N_{y<1}$ as absorber layers in multilayer-based solar selective coatings (SSC)
- $CrN_{0.67}$ and $Cr_{0.96}Al_{0.04}N_{0.89}$ films as IR-reflector/absorber layers for SSC

In order to explore the potentialities of $Cr_{1-x}(Al)_xN_y$ materials in multilayer-based solar selective coatings (SSC) for high temperature applications (T > 500 °C), the optical behavior of $Cr_{1-x}(Al)_xN_y$ films is studied in this work. Two sets of layers (CrN_y and $Cr_{1-x}Al_xN_y$) were prepared by direct current (DC) and high-power impulse magnetron sputtering (HiPIMS) technology. The deposition parameters: N_2 flux, HiPIMS frequency and aluminum sputtering power, were modified to get a wide variety of stoichiometries. The composition, morphology, phases and electronic structure of the films were characterized in depth. The optical behavior was determined by UV–Vis–NIR and FTIR spectroscopies, and the optical constants were obtained from the measured transmittance and reflectance spectra based on appropriate dielectric function models. Our results indicate that small changes in the layer composition influence the optical constants. In particular, a metallic-like behavior was obtained for CrN_y layers with N vacancies ($CrN_{0.95}$ and $CrN_{0.67}$ films) while a semiconductor-like behavior was observed for $CrN_{1.08}$. Thus, the CrN_y films can be used as effective absorber layer in multilayer-based SSC, and namely, the $CrN_{0.67}$ film as an IR reflector/absorber layer too. Likewise, the optical properties of $Cr_{1-x}Al_xN_y$ layers can also be tuned from metallic to semiconductor-like behavior depending on the chemical composition. Indeed, the absorption coefficients of $Cr_{1-x}Al_xN_y$ films with optimized Al content and N-vacancies are comparable to those reported for state-of-the-art materials such as TiAlN or TiAlCrN. In addition, a $Cr_{0.96}Al_{0.04}N_{0.89}$ film was found to be a suitable IR reflector/absorber layer.

Plasma-Assisted Deposition of TiO_2 3D Nanomembranes: Selective Wetting, Superomniphobicity, and Self-Cleaning

Montes, L; Román, JM; García-Casas, X; Castillo-Seoane, J; Sánchez-Valencia, JR; Barranco, A; López-Santos, C; Borrás, A
Advanced Materials Interfaces, **8** (2021) 2100767
Octubre 2021 | **DOI:** 10.1002/admi.202100767

Fabrication of tunable wetting surfaces is sought for the last years given its importance on energy, biomaterials and antimicrobials, water purification, microfluidics, and smart surfaces. Liquid management on surfaces mainly depends on the control at the micro- and nanoscale of both roughness and chemical composition. Herein, the combination of a soft-template method and plasma-enhanced chemical vapor deposition is presented for the synthesis of TiO_2 nanofibers on porous substrates such as cellulose and stainless-steel membranes. The protocol, carried out under mild conditions, produces 3D nanomembranes with superhydrophobicity and oleophilicity that are tested as microliter water/oil filters. Photoactivation of TiO_2 by UV illumination provides a straightforward approach for wetting tunability that converts the surface into amphiphilic. A final chemical modification of the TiO_2 nanofibers by embedding them in an elastomeric polymeric shell and by fluorine-based grafting opens the path toward the formation of superomniphobic and self-cleaning surfaces with long-lasting lifetimes. Thus, a reliable procedure is demonstrated for the fabrication of TiO_2 nanofibers, which allows the modification of porous supports and provides an innovative route for the development of 3D nanomembranes with under design wetting. This protocol is extendable to alternative metal oxides, metals, and core@shell nanoarchitectures with potential multifunctionalities.

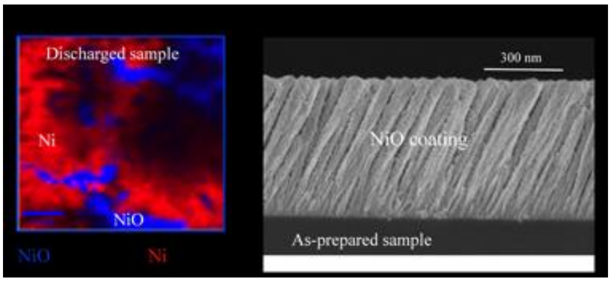
Pd-C Catalytic Thin Films Prepared by Magnetron Sputtering for the Decomposition of Formic Acid

Arzac, GM; Fernández, A; Godinho, V; Hufschmidt, D; de Haro, MCJ; Medrán, B; Montes, O
Nanomaterials, **11** (2021) 2326
Septiembre 2021 | **DOI:** 10.3390/nano11092326

Formic acid is an advantageous liquid organic hydrogen carrier. It is relatively nontoxic and can be synthesized by the reaction of CO_2 with sustainable hydrogen or by biomass decomposition. As an alternative to more widely studied powdery catalysts, supported Pd-C catalytic thin films with controlled nanostructure and compositions were newly prepared in this work by magnetron sputtering on structured supports and tested for the formic acid decomposition reaction. A two-magnetron configuration (carbon and tailored Pd-C targets) was used to achieve a reduction in Pd consumption and high catalyst surface roughness and dispersion by increasing the carbon content. Activity and durability tests were carried out for the gas phase formic acid decomposition reaction on SiC foam monoliths coated with the Pd-C films and the effects of column width, surface roughness and thermal pre-reduction time were investigated. Activity of 5.04 $mol_{H2}\cdot g_{Pd}^{-1}\cdot h^{-1}$ and 92% selectivity to the dehydrogenation reaction were achieved at 300 degrees C for the catalyst with a lower column width and higher carbon content and surface roughness. It was also found that deactivation occurs when Pd is sintered due to the elimination of carbon and/or the segregation and agglomeration of Pd upon cycling. Magnetron sputtering deposition appears as a promising and scalable route for the one-step preparation of Pd-C catalytic films by overcoming the different deposition characteristics of Pd and C with an appropriate experimental design.

New Insights on the Conversion Reaction Mechanism in Metal Oxide Electrodes for Sodium-Ion Batteries

Mosa, J; García-García, FJ; González-Elipe, AR; Aparicio, M
Nanomaterials, **11** (2021) 966
Abril 2021 | **DOI:** 10.3390/nano11040966



Due to the abundance and low cost of exchanged metal, sodium-ion batteries have attracted increasing research attention for the massive energy storage associated with renewable energy sources. Nickel oxide (NiO) thin films have been prepared by magnetron sputtering (MS) deposition under an oblique angle configuration (OAD) and used as electrodes for Na-ion batteries. A systematic chemical, structural and electrochemical analysis of this electrode has been carried out. The electrochemical characterization by galvanostatic charge-discharge cycling and cyclic voltammetry has revealed a certain loss of performance after the initial cycling of the battery. The conversion reaction of NiO with sodium ions during the discharge process to generate sodium oxide and Ni metal has been confirmed by X-ray photoelectron spectra (XPS) and micro-Raman analysis. Likewise, it has been determined that the charging process is not totally reversible, causing a reduction in battery capacity.

Mechanically Switchable Wetting Petal Effect in Self-Patterned Nanocolumnar Films on Poly(dimethylsiloxane)

Parra-Barranco, J; López-Santos, C; Sánchez-Valencia, JR; Borrás, A; González-Elipe, AR; Barranco, A
Nanomaterials, **11** (2021) 2566
Octubre 2021 | **DOI:** 10.3390/nano11102566

Switchable mechanically induced changes in the wetting behavior of surfaces are of paramount importance for advanced microfluidic, self-cleaning and biomedical applications. In this work we show that the well-known polydimethylsiloxane (PDMS) elastomer develops self-patterning when it is coated with nanostructured TiO_2 films prepared by physical vapor deposition at glancing angles and subsequently subjected to a mechanical deformation. Thus, unlike the disordered wrinkled surfaces typically created by deformation of the bare elastomer, well-ordered and aligned micro-scaled grooves form on TiO_2/PDMS after the first post-deposition bending or stretching event. These regularly patterned surfaces can be reversibly modified by mechanical deformation, thereby inducing a switchable and reversible wetting petal effect and the sliding of liquid droplets. When performed in a dynamic way, this mechanical actuation produces a unique capacity of liquid droplets (water and diiodomethane) transport and tweezing, this latter through their selective capture and release depending on their volume and chemical characteristics. Scanning electron and atomic force microscopy studies of the strained samples showed that a dual-scale roughness, a parallel alignment of patterned grooves and their reversible widening upon deformation, are critical factors controlling this singular sliding behavior and the possibility to tailor their response by the appropriate manufacturing of surface structures.

Insights into the role of the layer architecture of Cr-Ti-N based coatings in long-term high temperature oxidation experiments in steam atmosphere

Mato, S; Sánchez-López, JC; Barriga, J; Pérez, FJ; Alcalá, G
Ceramics International, **47** (2021) 4257-4266
Febrero 2021 | **DOI:** 10.1016/j.ceramint.2020.10.003

Knowledge on hard coatings has been applied in the energy field extending their use as protecting coatings of steam power generation plants components. The role of the layer architecture of Cr-Ti-N based coatings deposited by reactive cathodic arc evaporation on P92 steel substrates was studied with the focus on their oxidation resistance at 650 °C in 100% steam atmosphere up to 2000 h. Characterization of the coatings was performed by gravimetry, scanning electron microscopy, electron probe microanalysis, glow discharge optical emission spectroscopy, X-ray diffraction, thermodynamic simulations using the CALPHAD method, Rockwell C indentation and nanoindentation. The layered arrangement improves the oxidation resistance of TiN under the working conditions of steam power plants, as well as the mechanical properties of CrN. The produced architectures performance under the described working conditions boosts the understanding of the processes taking place at high temperature, making possible the design of optimal coatings combining the best behavior of both nitrides for each specific application, reaching a corrosion protection at high temperature in water vapor comparable to that of CrN and a hardness and Young's modulus as high as those of TiN.

Recent Advances in Alkaline Exchange Membrane Water Electrolysis and Electrode Manufacturing

López-Fernández, E; Sacedón, CG; Gil-Rostra, J; Yubero, F; González-Elipe, AR; De Lucas-Consuegra, A

Molecules, **26** (2021) 6326

Noviembre 2021 | **DOI:** 10.3390/molecules26216326

Water electrolysis to obtain hydrogen in combination with intermittent renewable energy resources is an emerging sustainable alternative to fossil fuels. Among the available electrolyzer technologies, anion exchange membrane water electrolysis (AEMWE) has been paid much attention because of its advantageous behavior compared to other more traditional approaches such as solid oxide electrolyzer cells, and alkaline or proton exchange membrane water electrolyzers. Recently, very promising results have been obtained in the AEMWE technology. This review paper is focused on recent advances in membrane electrode assembly components, paying particular attention to the preparation methods for catalyst coated on gas diffusion layers, which has not been previously reported in the literature for this type of electrolyzers. The most successful methodologies utilized for the preparation of catalysts, including co-precipitation, electrodeposition, sol-gel, hydrothermal, chemical vapor deposition, atomic layer deposition, ion beam sputtering, and magnetron sputtering deposition techniques, have been detailed. Besides a description of these procedures, in this review, we also present a critical appraisal of the efficiency of the water electrolysis carried out with cells fitted with electrodes prepared with these procedures. Based on this analysis, a critical comparison of cell performance is carried out, and future prospects and expected developments of the AEMWE are discussed.

Effect of Al content on the hardness and thermal stability study of AlTiN and AlTiBN coatings deposited by HiPIMS

Méndez, A; Monclus, MA; Santiago, JA; Fernández-Martínez, I; Rojas, TC; García-Molleja, J; Avella, M; Dams, N; Panizo-Laiz, M; Molina-Aldareguia, JM

Surface & Coatings Technology, **422** (2021) 127513

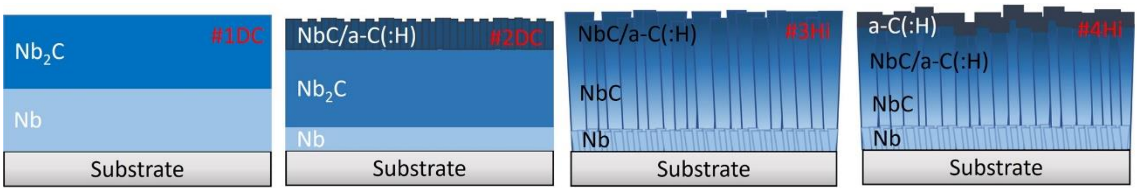
Septiembre 2021 | **DOI:** 10.1016/j.surfcoat.2021.127513

The microstructure, mechanical properties and thermal stability of $AT_xTi_{1-x}N$ and $Al_1Ti_{1-x}BN$ coatings grown by reactive high-power impulse magnetron sputtering (HiPIMS) have been analyzed as a function of *Al*/(*Al* + *Ti*) ratio (x) between 0.5 and 0.8. The coatings were predominantly formed by a face-centered cubic Ti(Al)N crystalline phase, both with and without B, even for x ratios as high as 0.6, which is higher than the ratio typically encountered for $Al_xTi_{1-x}N$ coatings deposited by reactive magnetron sputtering. B doping, in combination with the highly energetic deposition conditions offered by HiPIMS, results in the suppression of the columnar grain morphology typically encountered in $Al_xTi_{1-x}N$ coatings. On the contrary, the $Al_xTi_{1-x}N$ coatings grown by HiPIMS present a dense nanocomposite type microstructure, formed by nanocrystalline Ti(Al) N domains and amorphous regions composed of Ti(Al)B 2 and BN. As a result, high-Al content (x approximate to 0.6) $Al_xTi_{1-x}N$ coatings grown by HiPIMS offer higher hardness, elasticity and fracture toughness than $Al_xTi_{1-x}N$ coatings. Moreover, the thermal stability and the hot hardness are substantially enhanced, delaying the onset of formation of the detrimental hexagonal AlN phase from 850 °C in the case of $Al_{0.6}Ti_{0.4}N$, to 1000 °C in the case of $Al_{0.6}Ti_{0.4}N$.

Nb-C thin films prepared by DC-MS and HiPIMS: Synthesis, structure, and tribomechanical properties

Sala, N; Abad, MD; Sánchez-López, JC; Caro, J; Colominas, C

Surface & Coatings Technology, **422** (2021) 127569
Septiembre 2021 | **DOI:** 10.1016/j.surfcoat.2021.127569



Nanostructured Nb-C thin films were prepared by direct current magnetron sputtering (DC-MS) and high-power impulse magnetron sputtering (HiPIMS). The films were characterized in depth by X-ray diffraction (XRD), grazing incidence X-ray diffraction, scanning electron microscopy, atomic force microscopy, electron probe microanalysis, and Raman spectroscopy. The mechanical properties were measured by nanoindentation, and the tribological properties were measured by pin-on-disk tests in ambient air. The wear tracks and ball scars were analyzed by Raman spectroscopy to elucidate the tribochemical reactions that occurred at the contact area and to determine the wear mechanism for each specimen type. The thermal stability of the coatings was studied up to 1000 °C using Raman spectroscopy and XRD. The samples prepared by DC-MS were very dense, and the phase composition changed from purely nanocrystalline (Nb_2C and NbC) to a mixture of NbC crystals embedded in an amorphous carbon-based matrix (NbC/a-C(:H)). However, the samples prepared by HiPIMS developed a marked columnar morphology with a NbC/a-C(:H) nanocomposite structure. The hardness values ranged from 11 to 20 GPa depending on the deposition technique and the amount of the soft a-C(:H) phase present in the sample. The tribological properties of all the coatings were remarkably good when the carbon content was approximately 50 at.%. The formation of a lubricating sp^2-rich C tribofilm between the ball and coating during the pin-on-disk tests was observed by Raman spectroscopy. The tribofilm formed preferentially on the samples prepared by HiPIMS, which had higher C contents. At 750 °C, the degradation of the NbC phases resulted in the formation of an additional a-C phase and niobium oxides.

Influence of helium incorporation on growth process and properties of aluminum thin films deposited by DC magnetron sputtering

Ibrahim, S; Lahboub, FZ; Brault, P; Petit, A; Caillard, A; Millon, E; Sauvage, T; Fernández, A; Thomann, AI

Surface & Coatings Technology, **426** (2021) 127808
Noviembre 2021 | **DOI:** 10.1016/j.surfcoat.2021.127808

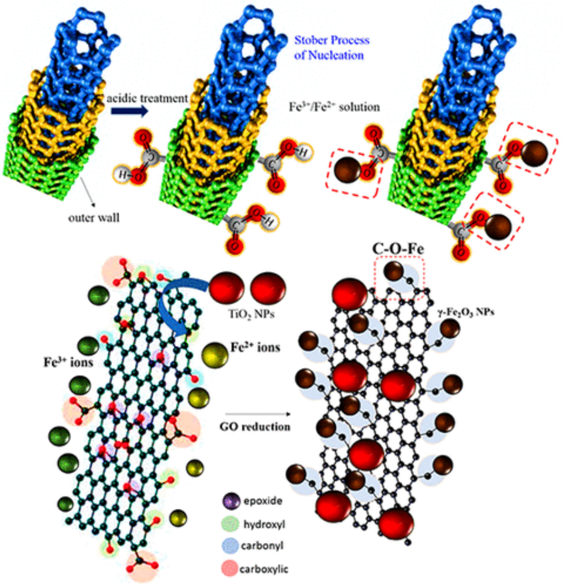
The effect of helium content on the morphology, crystallinity, and composition of aluminum films was investigated by depositing He-loaded Al films onto Si substrates via direct current (DC) magnetron sputtering in different Ar/He plasma mixtures. Three different plasma regimes were identified depending on the percentage of He in the gas phase. For a low He to total gas ratio (ΓHe ≤ 70%), the plasma is dominated by argon, where Ar+ ions contribute to sputter out the target atoms. The films deposited in this regime exhibited the classical dense columnar structure and contain very low amount of He (below 2%). Then, as ΓHe increases, helium ions begin to be formed and more fast He neutrals reach the substrate, affecting the film growth. As He amount increased in the gas phase up to 95%, the proportion of He inserted in the films rised up to ~15 at. %. Moreover, bubbles/porosity were formed inside the films; those obtained in pure He plasma presented a highly porous fiberform nanostructure. All results confirmed that the modification of the film characteristics was related to the change of the deposition conditions when Ar was replaced by He and to the insertion/release mechanisms of He during the growth.

Extraction of microstructural parameters from sculptured thin films nanoindentation
Gaillard, Y; Jiménez-Piqué, E; Oliva-Ramírez, M; Rico, VJ; González-Elipe, AR
Surface & Coatings Technology, **425** (2021) 127696
Noviembre 2021 | **DOI:** 10.1016/j.surfcoat.2021.127696

This work deals with the indentation analysis of nanocolumnar thin films and the difficulties encountered to deduce relevant mechanical parameters by this methodology. SiO_2 thin films prepared by physical vapour oblique angle deposition with different nanocolumnar microstructures have been subjected to indentation analysis. Despite the fact that the films had been made of the same material, deposited on the same substrate and had similar thickness, their indentation responses were different and depended on their particular microstructure. It has been also realised that the measured hardness and elastic modulus variation with the indentation depth were length scale dependent and that there is not a unique analytical thin-film nanoindentation model to extract the mechanical properties from the experimental nanoindentation curves. To overcome these limitations a numerical finite element model (FEM) of the nanocolumnar coatings has been built to figure out the contributions of the different physical phenomena intervening in the indentation process. This FEM simulation relies on a description of the elasto-plastic microstructural units of the coatings and the contact friction interactions between them. Based on this simulation a parametrical representation, incorporating two length scales and the contributions of densification and/or the buckling of nanocolumnar units, has been developed to account for the evolution of the apparent elastic modulus deduced from numerical indentation tests. A Hall-Petch modification of this description considering two length scales instead of the common approximation considering a single length scale has rendered the best agreement with the elastic values determined experimentally. Although, at the present stage, the particular microstructure of the films can not be deduced from the evolution of their elastic moduli with the indentation depth, the obtained results and their interpretation constitute a first though essential step for the elaboration of an inverse analysis methodology capable of correlating microstructure and elastic response of nanocolumnar coatings.

Rietveld Refinement, μ-Raman, X-ray Photoelectron, and Mössbauer Studies of Metal Oxide-Nanoparticles Growth on Multiwall Carbon Nanotubes and Graphene Oxide
Ramos-Guivar, JA; González-González, JC; Litterst, FJ; Passamani, EC
Crystal Growth & Design, **21** (2021) 2128-2141
Abril 2021 | **DOI:** 10.1021/acs.cgd.0c01551

Applying a modified coprecipitation method, maghemite and anatase nanoparticles embedded in graphene oxide and multiwall carbon nanotube frameworks were prepared, and a detailed structural characterization is presented. Transmission electron images have revealed that the



multiwall carbon nanotubes and graphene oxide act as substrates to reduce the nanoparticle agglomeration with narrow sizes of ca. 9-20 nm, in agreement with the results of the Rietveld refinement, which have also indicated their crystallite apparent size and shapes using the spherical harmonics approach. In structural studies of maghemite nanoparticles by Raman spectroscopy, it was found that the effect of optical density and laser power intensity plays a significant role. When no optical filter was located between the powder sample and the laser source, a transformation from the γ-Fe_2O_3 to the α-Fe_2O_3 phase was observed, as demonstrated by the disappearance of the characteristic broad Raman peak (A_{1g}) of the γ-Fe_2O_3 phase when increasing the laser power. X-ray photoelectron spectroscopy has also brought insights into the functionalization mechanism, suggesting that the one-pot reduction of the graphene oxide is favored by the alkaline γ-Fe_2O_3 nanoparticle growth. The temperature dependence of the ^{57}Fe Mössbauer spectra has indicated that the effective anisotropy constant of Fe oxide-based nanoparticles is similar to that of bulk maghemite, and magnetic relaxation of Fe^{3+} spins depends on particle sizes.

Physicochemical surface analysis and germination at different irrigation conditions of DBD plasma-treated wheat seeds

Molina, R; Lalueza, A; López-Santos, C; Ghobeira, R; Cools, P; Morent, R; de Geyter, N; González-Elipe, AR

Plasma Processes and Polymers, **18** (2021) 2000086

Enero 2021 | **DOI:** 10.1002/ppap.202000086

Plasma treatment is increasingly being explored as an effective presowing treatment improving seed germination. This study examines the synergetic effect of the irrigation condition and the physicochemical surface properties of wheat seeds subjected to atmospheric dielectric barrier discharge plasma activation on their water uptake and germination. Extensive surface analysis revealed a remarkably enhanced wettability of plasma-treated seeds due to the insertion of oxygen-containing functionalities on their surface. However, long plasma exposures damaged the outermost layers of the pericarp due to a pronounced oxidative etching effect. Although the seed germination capacity was not affected by the plasma treatments, short plasma exposures were shown to enhance water uptake and accelerate seed germination, especially under water-scarcity conditions.

Electrical and reaction performances of packed-bed plasma reactors moderated with ferroelectric or dielectric materials

Gómez-Ramírez, A; Álvarez, R; Navascúes, P; García-García, FJ; Palmero, A; Cotrino, J; González-Elipe, AR
Plasma Processes and Polymers, **18** (2021) 2000193
Enero 2021 | **DOI:** 10.1002/ppap.202000193

The operational behavior of packed-bed plasma reactors depends on the dimension, shape, and chemical properties of the pellets used as moderators, but little information exists about the influence of their specific dielectric properties. Herein, we comparatively study the electrical behavior of a packed-bed reactor filled with pellets of either dielectric (Al_2O_3 and glass) or ferroelectric ($BaTiO_3$ and lead zirconate titanate) materials. We found that plasma current was higher for ferroelectrics and presented a nonlineal dependence on voltage. Moreover, for $BaTiO_3$, we found a drastic decrease at around its relatively low Curie temperature. Differences in electrical behavior have a direct effect on the reactor performance, as illustrated for the ammonia synthesis, demonstrating the importance of moderator material dielectric properties and their dependence on temperature.

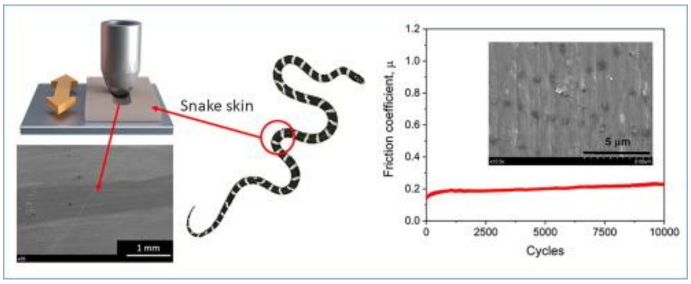
Nanofibrous Gelatin-Based Biomaterial with Improved Biomimicry Using D-Periodic Self-Assembled Atelocollagen

Borrego-González, S; Dalby, MJ; Díaz-Cuenca, A
Biomimetics, **6** (2021) 20
Marzo 2021 | **DOI:** 10.3390/biomimetics6010020

Design of bioinspired materials that mimic the extracellular matrix (ECM) at the nanoscale is a challenge in tissue engineering. While nanofibrillar gelatin materials mimic chemical composition and nano-architecture of natural ECM collagen components, it lacks the characteristic D-staggered array (D-periodicity) of 67 nm, which is an important cue in terms of cell recognition and adhesion properties. In this study, a nanofibrous gelatin matrix with improved biomimicry is achieved using a formulation including a minimal content of D-periodic self-assembled atelocollagen. We suggest a processing route approach consisting of the thermally induced phase separation of the gelatin based biopolymeric mixture precursor followed by chemical-free material cross-linking. The matrix nanostructure is characterized using field emission gun scanning electron microscopy (FEG-SEM), transmission electron microscopy (TEM), wide angle X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FT-IR). The cell culture assays indicate that incorporation of 2.6 wt.% content of D-periodic atelocollagen to the gelatin material, produces a significant increase of MC3T3-E1 mouse preosteoblast cells attachment and human mesenchymal stem cells (hMSCs) proliferation, in comparison with related bare gelatin matrices. The presented results demonstrate the achievement of an efficient route to produce a cost-effective, compositionally defined and low immunogenic “collagen-like” instructive biomaterial, based on gelatin.

Long-term low friction maintenance and wear reduction on the ventral scales in snakes

Sánchez-López, JC; Schaber, CF; Gorb, SN
Materials Letters, **285** (2021) 129011
Febrero 2021 | **DOI:** 10.1016/j.matlet.2020.129011



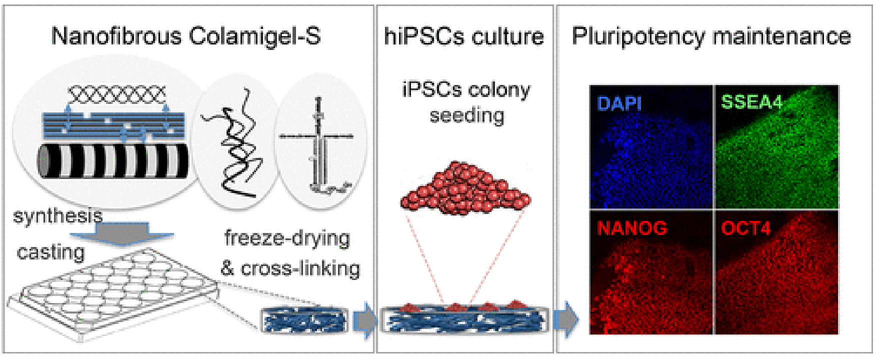
Snake skins evolved to withstand permanent friction and wear during sliding. Here, the microstructure of ventral scales of the snake *Lampropeltis getula californiae* was analyzed using scanning electron microscopy, and the long-term dynamic friction behavior was investigated by reciprocating sliding friction tests. A smooth epoxy resin with similar elasticity modulus and hardness was used for comparison purposes. Strong differences in frictional and wear mechanisms between the two materials were revealed in spite of similar mechanical properties. Snake skin showed a considerably lower frictional coefficient that kept stable over several thousands of sliding cycles. A reduction of the stick-slip behavior was also denoted by analyzing the variation of the friction coefficient in the forward and reverse motion influencing the wear mechanism. This frictional behavior can be explained by three different but complementary mechanisms: fibrous layered composite material of the skin with a gradient of material properties, surface microstructure, and the presence of ordered layers of lipid molecules at the skin surface.

Characterizing the physicochemical and mechanical properties of ZrN thin films deposited on Zr substrates by pulsed laser technique

Ghemras, I; Abdelli-Messaci, S; Alili, B; González-Elipe, AR; Rico, VJ; Izerrouken, M;
Khereddine, AY; Hadj-Larbi, F
European Physical Journal-Applied Physics, **95** (2021) 10301
Julio 2021 | **DOI:** 10.1051/epjap/2021210064

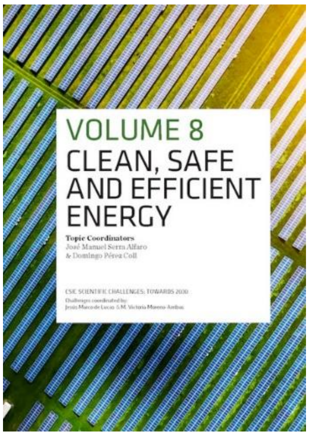
Due to their outstanding physical and mechanical features, ZrN thin films are increasingly used as coatings to protect materials intended for nuclear applications such as Zirconium. To our knowledge, there is no report of pulsed laser deposition (PLD) of ZrN thin films on a Zr substrate. In this work, we have successfully prepared ZrN thin films on Zr substrates using the PLD technique with a KrF excimer laser, in a N_2 environment at 2 Pa pressure and a fixed substrate temperature of 500 °C. The deposited 200 nm ZrN thin films exhibited a homogeneous surface and showed a face-centered cubic polycrystalline structure. The surface roughness was 3.69 nm. X-ray diffraction, Raman and X-ray photoelectron spectroscopy measurements confirmed the presence of ZrN. The coated sample's mean value of hardness (11.6 GP) doubled that of the uncoated sample.

Nanofibrous Matrix of Defined Composition Sustains Human Induced Pluripotent Stem Cell Culture
Borrego-González, S; de la Cerda, B; Díaz-Corrales, FJ; Díaz-Cuenca, A
ACS Applied Bio Materials, **4** (2021) 3035-3040
Abril 2021 | **DOI:** 10.1021/acsabm.0c00425



Human induced pluripotent stem cells (hiPSCs) represent the most promising biological material for regenerative medicine applications. In this work, a 3D solid nanofibrous matrix of defined composition (Colamigel-S) consisting of 97 wt % gelatin, 2.6 wt % atelocollagen, and 0.4 wt % laminin has been reproducibly processed and characterized and exhibits a homogeneous nanofibrillar network of high surface area, interconnected microcavities, and typical D-periodic collagen fibril nanostructural features. The purpose of the study was to test the performance of Colamigel-S as substrate for in vitro hiPSCs culture, finding that these cells efficiently attach and grow keeping their characteristic stem morphology and undifferentiated state.

Libros y Otras Publicaciones / Books and Other Publications



Clean, safe and efficient energy Volumen 8
CSIC Scientific Challenges : Towards 2030
Consejo *Superior de Investigaciones Científicas* 2021
ISBN Vol. 8: 978-84-00-10752-9

Challenge 1: Renewable Energy Production
Hernán Míguez, Mariano Campoy Quiles, Luis A. Pérez-Maqueda, Agustín R. González-Elipe, Xavier Granados, Víctor Vilarrasa, Xavier Odradors, Teresa Puig, José María Ripalda, Mariona Coll, Josep Fontcuberta, Alejandro Goñi, Mónica Lira Cantú, María Bernechea, Pedro Atiénzar, Mauricio Calvo
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Challengue 3: Energy Efficiency and Harvesting
Marisol Martín González, José Francisco Fernández, José Antonio Tenorio, Agustín R. González-Elipe, Gabriel Lozano, Ana I. Borrás, Olga Caballero, Luis Fonseca, Mariano Campoy, Neus Sabaté
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Challengue 7: Catalysis for Industrial Production and of Energy Resources
José Carlos Conesa, Manuel López, Vicente Cortés, Francisco Plou, Miguel Antonio Peña, Juan Manuel Coronado, Ana Iglesias, Pedro Ávila, Miguel Ángel Bañares, Ana Serrano, Agustín Martínez, Mercedes Boronat, Miguel Ángel Centeno, Agustín R. González-Elipe, Jesús Pérez Torrente
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Challengue 8: Hydrogen Technologies
Antonio Chica, Félix Barreras, Javier Dufour, Asunción Fernández, Miguel A. Laguna-Becerro, María Jesús Lázaro, Glenn C. Mather, Fernando Palacín, Miguel Antonio Peña, Luis Valiño
páginas 207-235

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

2021 Virtual MRS Spring Meeting & Exhibit
17 – 23 abril [Online, Estados Unidos]

Design, spectral properties and thermal stability of high-temperature solar-selective coatings based on Cr(Al)N multilayer stacks
R. Escobar-Galindo, T.C. Rojas, A. Caro, J.C. Sánchez-López
Comunicación oral

Plasma Nanoengineering for the Development of Hybrid Piezo and Tribonanogenerators
Xabier García-Casas, Nicolas Filippín, Javier Castillo-Seoane, Francisco J. Aparicio, Ali Ghafarinejad, Jorge Budagoski, Carmen López-Santos, Ángel Barranco, Juan R. Sánchez-Valencia, Ana Borrás
Comunicación oral

Design of high-temperature solar selective coatings based on Cr(Al)N: Microstructure and optical properties of CrN_y and $Cr_{1-x}Al_xN_y$ films
T.C. Rojas, A. Caro, G. Lozano, J.C. Sánchez-López
Póster

Opportunities of HiPIMS technology for designing complex multilayered Cr(Al)N coatings for solar selective applications
J.C. Sánchez-López, A. Caro, J. Martínez-Romero, T.C. Rojas
Póster

Remote Plasma Assisted Vacuum Deposition of Functional Organic and Polymeric Layers for the Development of thin Film Piezoelectric Nanogenerators and UV Piezotronic Detectors
Xabier García-Casas, Francisco J. Aparicio, José M. Obrero, Jorge Budagoski, Ángel Barranco, Juan R. Sánchez-Valencia, Ana Borrás
Póster

Organic Materials in Perovskite-based Optoelectronic Devices (OrgMatPerPV)
29 – 30 abril [Online]

Enhancing Moisture and Water Resistance in Perovskite Solar Cells by Encapsulation with Ultrathin Plasma Polymers
Jesus Idígoras, Francisco J. Aparicio, Lidia Contreras-Bernal, Susana Ramos-Terrón, María Alcaire, Juan Ramón Sánchez-Valencia, Ana Borrás, Ángel Barranco, Juan A. Anta
Comunicación oral

13th International Conference on Hybrid and Organic Photovoltaics HOPV21
24 – 28 mayo [Online]

Vacuum sublimation of Dopant-Free Crystalline Spiro-OmMeTAD films to enhance the stability of perovskite Solar Cells
Juan Ramón Sánchez-Valencia, Jose Obrero-Pérez, Lidia Contreras Bernal, Darío Jumilla, Javier Castillo-Seoane, Francisco J. Aparicio, M. Carmen López-Santos, Ana Borrás, Juan A. Anta, Angel Barranco
Comunicación oral

Encapsulation of Perovskite Solar Cells: From Buit-in Potential and Interfacial Passivation Perspectives
Jesús Idígoras, Francisco J. Aparicio, Lidia Contreras-Bernal, Susana Ramos-Terrón, María Alcaire, Darío Jumilla, Juan Ramón Sánchez-Valencia, Ana Borrás, Angel Barranco, Juan A. Anta
Comunicación oral

2021 Conference on Lasers and Electro-Optics/Europe – European Quantum Electronics Virtual Conferences (CLEO®/Europe-EQEC 2021)

21 – 25 junio [Online, Alemania]

Anisotropic Resistivity ITO Surfaces produced by Laser-induced Self-organization at the Nanoscale
M. Macías-Montero, C. López-Santos, D. Puerto, J. Siegel, C. Florián, J. Gil-Rostra, V. López-Flores, A. Borrás, A.R. González-Elipe, J. Solís
Comunicación oral

3rd Workhop on Size-Dependent Effect in Materials for Environmental Protection and Energy Application

12 – 15 septiembre [Pomorie, Bulgaria]

Mesoporous ceramics: applications in biomedical research
Aránzazu Díaz-Cuenca
Conferencia Plenaria

Porous electrodes prepared by magnetron sputtering: from electrochemical sensing to energy applications
Agustín R. González-Elipe
Conferencia Plenaria

Plasma Thin film International Union Meeting PLATHINIUM 2021

13 – 17 septiembre [Virtual Meeting]

Helium-charged aluminum and silicon films deposited by Direct Current Magnetron Sputtering
S. Ibrahim, P. Brault, A. Caillard, T. Sauvage, P. Desgardin, M.F. Barthe, D. Hufschmidt, A. Fernández, A.L. Thomann
Comunicación oral

European Congress and Exhibition on advanced Materials and Processes EUROMAT 2021

13 – 17 septiembre [Virtual Conference]

Surface engineering solutions for additive manufactured biomedical materials
R. Escobar-Galindo, J. Hernández-Saz, E. Acena, P. Andrés-Cano, B. Palacios, T.C. Rojas, J.C. Sánchez-López, S.I. Molina
Comunicación oral

8th Plasma Science & Entrepreneurship Workshop
8 – 9 noviembre [Online, República Checa]

Ultraporous conformal nanostructures developed by plasma deposition and processing of metal phthalocyanines and porphyrins
J.M. Obrero, A.N. Filippín, J.R. Sánchez-Valencia, F.J. Aparicio, Z. Shagui, T.C. Rojas, Z. Saghi, A. Borrás, A. Barranco
Conferencia Invitada

ImagineNano2021/NanoSpain Conference 2021
23 – 25 noviembre [Bilbao, España]

Next-generation of tribological lubricants modified with magnetic nano-microwires
Mª Fe Menéndez Suarez, Ana L. Martínez Díez, Pascal Sánchez, Juan Carlos Sánchez López
Póster

European Fuel Cells and Hydrogen Piero Lunghi Conference 2021 EFC21
15 – 17 diciembre [Virtual Meeting]

A hydrogen generator coupled to a hydrogen heater for small scale portable applications
Dirk Hufschmidt, Gisela M. Arzac, María del Carmen Jiménez de Haro, Asunción Fernández
Póster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

VIII Jornada Investigación, Desarrollo e Innovación
27 – 28 octubre [Sevilla, España]

Surface functionalization of additive manufactured biomedical materials
R. Escobar Galindo, J. Hernández Saz, E. Acena, E. de Armiñan, C. Casal, P. Andrés Cano, B. Palacios I, T.C. Rojas, J.C. Sánchez López, S. I. Molina
Comunicación oral

41 Congreso AEDE 2021
29 – 31 octubre [Online, España]

Características físico-químicas y proceso de hidratación de nuevas formulaciones de cemento endodóncico bioactivo compuestas de Agregado Trióxido Mineral (MTA) y vidrio bioactivo mesoporoso funcionalizado con Zinc (MBG-Zn): MTA-MBG-Zn
M.C. Jiménez-Sánchez, J.J. Segura Egea, A. Díaz-Cuenca
Comunicación oral

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Simulación de campos electromagnéticos en intercaras: plasmones, ondas de Bloch y radiación Cherenkov sintonizada**
Autor: Antonio Gandorfina Andrades
Tutores: Francisco Yubero Valencia
Grado: Trabajo Fin de Máster
Fecha Defensa: 13 enero 2021

Título: **Novel Perfluorinated Polymeric Thin Films Deposited by Remote Plasma Assisted Vacuum Deposition (RPAVD) for the Development of Controlled Wetting Surfaces**
Autor: Triana Czermak Álvarez
Tutores: Francisco Javier Aparicio Rebollo, Ángel Barranco Quero, Rosa Pereñíguez Rodríguez
Grado: Trabajo Fin de Máster
Fecha Defensa: 17 septiembre 2021

Título: **Characterization set-up and conditioning circuits for nanogenerators based on kinetic energy harvesters**
Autor: Jaime del Moral Jalón
Tutores: Francisco Javier Aparicio Rebollo, Ali Ghaffarinejad, Antonio José Gines Arteaga
Grado: Trabajo Fin de Máster
Fecha Defensa: 21 septiembre 2021

DOCENCIA / TEACHING

Plasma y vacío

1er Curso de Vacío Otoño 2021
María del Carmen López Santos
Lugar: Asociación Española del Vacío y sus Aplicaciones ASEVA

Investigadores de esta unidad participan en el Máster Interuniversitario "Láser, Plasma y Tecnología de Superficies" y en titulaciones de Grado y doble Grado de la Universidad de Sevilla (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Best Poster Winners-2021 Virtual MRS Spring Meeting
Dra. Teresa Cristina Rojas

FEMS Master Thesis Award Winner 2021
Xabier García Casas from SOCIEMAT was awarded first place for his presentation on '1D and 3D nanomaterial fabrication and surface modification for developing piezoelectric and triboelectric nanogenerators.'

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de magnetron putttering para la preparación de láminas delgadas
- Potenciostato y sistema electroquímico
- Sistema de medida de ángulos de contacto (advancing and receding)
- Equipo de crecimiento en vacío de nanofibras y otros materiales nanoestructurados
- Microscopio de efecto túnel (STM)
- Sistema de desorción térmica programada
- Espectrómetro de Fotoemisión (XPS)
- Colorímetro Dr. Lange: para la medida de parámetros de color (x, y, Y/L*a*b*, etc.) de superficies y polvos
- Ellipsómetro espectroscópico Woolan VB-400 con rango de frecuencias entre 300 y 1700 nm. Medida de índices de refracción y coeficiente de extinción de capas delgadas y superficies.
- Espectrómetro visible-UV CARY-100. Medidas de coeficiente de absorción con luz normal y polarizada.

- Fluorímetro espectroscópico (HORYBA Jobin Yvon Fluorolog) con accesorio para la determinación de tiempos de vida. Microscopio de fluorescencia (HORYBA Jobin Yvon sigle photon controller: FluoroHub).
- Medidor de ángulos de contacto líquidos. Medidas ángulos de avance y retroceso, así como de energías de adhesión de líquidos sobre superficies (Dataphysics Contact Angle System SCA 20).
- Medida de cuatro puntos de conductividad eléctrica en superficies y láminas delgadas (Fuente de corriente Keithley 617 y voltímetro Keithley 2400).
- Medidas eléctricas en capas delgadas en función de la temperatura y la atmósfera
- Microscopio de Fuerzas atómicas (AFM) para la caracterización de superficies (Cervantes de Nanotec).
- Microscopio de efecto túnel (STM) con posibilidad de trabajar desde nitrógeno líquido hasta 600 °C (VT-STM de Omicrom).
- Técnicas de caracterización de plasmas: sonda de Langmuir (Plasma Consult single and double sound), espectroscopía de emisión óptica (Avantes 200-900 nm resolución 1 nm) y espectrometría de masas (Hyden)
- Espectrómetro FT-IR con celda DRIFT (Pelkin elmer Spectrum One)
- Sistema de medida de porosidades en capas delgadas.
- Sistema de desorción térmica programada dotado con espectroscopía Auger (VG-8047).
- Espectrómetro de XPS (espectrómetro VSW) con sistema REELS de alta resolución (Kimball Physics EGPS-1022B) y fuente de átomos incorporada (Oxford Scientific Osprey plasma Source).
- Sistema de tratamiento con plasmas Diener.
- Tres cámaras de deposición por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación total de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables, calentables y “biased”.
- Material básico de laboratorio químico: PHmetro, agitadores, calefactores, estufa de secado a vacío, centrífuga.
- Reactores y material de vidrio convencional para síntesis de nanopartículas y catalizadores por via química.
- Rotavapor, sistemas de filtrado.
- Cámara seca MBRAUN.
- Dos campanas extractoras.
- Reactores catalíticos de lecho fijo para catálisis heterogénea sólido-gas.
- Reactores catalíticos para catálisis heterogénea sólido-líquido.
- Sistemas controladores de flujo másico (gases y líquidos evaporados), bombas peristálticas para líquidos.
- Tres cromatógrafos de gases, columnas y detectors FID y TCD.
- Sistema de medida de espectroscopía de impedancia compleja, formado por un impedancímetro Agilent modelo 4294ª, un horno Hobersal ST115020, y una celda de medida hermética para la realización de medidas en atmósfera.
- Buretas automáticas para medición de gases.
- Sistema de preparación de muestras en película delgada TXP de Leica.
- Pulidora, trípode y microscopio óptico.
- TEM de 300kV Tecnai F30 dotado de modo STEM, detector HAADF, analizador EDX Oxford Max80 y filtro de energías GIF Quantum.
- Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.

- Tribómetro de alta temperatura (hasta 800 °C) –Microtest
- Calotest para medida de espesores y evaluación del desgaste.
- Equipo de rayado (Scratch-test) hasta 200N (Tribotechnic).
- Perfilómetro-rugosímetro (Mahr) de tipo táctil y resolución vertical nanométrica.
- Cámara de deposición PVD-Magnetron sputtering (2 magnetron).
- Fuentes DC-Pulsada (ENI) ; RF (Trumpf); HiPIMS (Solvix)
- Horno tubular 1500 °C (Carbolite)
- Pulidora
- Ultrasonidos
- Microscopio óptico (Leica)
- Microscopio óptico Interferométrico 3D (Sensofar)

MATERIALES ÓPTICOS
OPTICAL MATERIALS

GRUPOS DE INVESTIGACIÓN

Materiales Coloidales
Colloidal Materials [642011]
http://colmat.icmse.csic.es

Materiales Ópticos Multifuncionales
Multifunctional Optical Materials [642013]
http://mom.icmse.csic.es

PERSONAL / PERSONNEL

Profesores de Investigación	Dr. Hernán R. Míguez García Dr. Manuel Ocaña Jurado
Científicos Titulares	Dra. Ana Isabel Becerro Nieto Dr. Mauricio E. Calvo Roggiani Dr. Gabriel Lozano Barbero Dra. Nuria Ofelia Núñez Álvarez
Investigadores Distinguidos	Dr. Juan F. Galisteo López
Contratados Juan de la Cierva	Dra. Laura Caliò
Doctores Contratados	Dra. Roxana Marisol Calderón Olvera Dr. Victor Castaing Dr. Daniel González Mancebo Dr. Ngo Thi Tuyen
Personal Investigador en Formación	Gda. Encarnación Arroyo Porriño Gda. Clara Bujalance Aguilera Gda. Elena Cabello Olmo Gda. Beatriz De Sola Báez Gda. Elisabet Gómez González Gda. María Morán Pedroso Gdo. Carlos Romero Pérez Gdo. David Otto Tiede Gdo. José María Viaña Jorge
JAE Intro	D. Roberto Prieto García D. Manuel Romero Aguilar
Personal Técnico Contratado	Lda. Lucía T. Castillo Flores Lda. M. Carmen Gutiérrez Lázaro

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Materiales ópticos basados en nanofósforos para la próxima generación de dispositivos emisores de luz | NANOPHOM	**Nanophosphor-based photonic materials for next generation light-emitting devices | NANOPHOM**
Código/Code:	H2020-ERC-STG/0259
Periodo/Period:	01-04-2017 / 31-03-2023
Organismo Financiador/Financial source:	Comisión Europea
Importe total/Total amount:	1.499.739 €
Investigador responsable/Research head:	Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El desarrollo de fuentes de luz eficientes y respetuosas con el medio ambiente constituye una parte esencial de la estrategia mundial para reducir el consumo mundial de electricidad. Los diodos emisores de luz (LED de sus siglas en inglés) emergen como la alternativa a la iluminación convencional, debido a su alta eficiencia de conversión de energía, larga vida útil, conmutación rápida, robustez y tamaño compacto. Sin embargo, su implementación en la industria electrónica de consumo se ve obstaculizada por el control limitado sobre el brillo, la calidad del color y la direccionalidad de la emisión de LED que proporcionan los elementos ópticos convencionales cuyo funcionamiento se basa en la óptica geométrica.

Este proyecto explora nuevas formas de controlar las propiedades de emisión de nanomateriales luminiscentes, superando los límites impuestos por la óptica convencional, mediante el uso de arquitecturas nanofotónicas. El desarrollo de materiales ópticos fiables y escalables basados en nanofósforos permitirá un control espectral y angular fino sobre la emisión de luz, abordando las deficiencias que los LED actuales presentan. El nuevo diseño óptico de estos dispositivos estará basado en la integración de multicapas ópticas, texturas superficiales, y nano dispersores de composición, tamaño y forma controlados, para obtener materiales que posean propiedades ópticas que faciliten un control preciso de la radiación visible.

Nanophom permitirá mejorar nuestra comprensión sobre fenómenos fundamentales como la formación de modos fotónicos en medios ópticos complejos a los que se puede acoplar la luz, así como avanzar en el desarrollo de dispositivos de iluminación de estado sólido de alta eficiencia.

Energy-efficient and environmentally friendly light sources are an essential part of the global strategy to reduce the worldwide electricity consumption. Light-emitting diodes (LEDs) emerge as a key alternative to conventional lighting, due to their high power-conversion efficiency, long lifetime, fast switching, robustness, and compact size. Nonetheless, their implementation in the consumer electronic industry is hampered by the limited control over brightness, colour quality and directionality of LED emission that conventional optical elements relying on geometrical optics provide.

This project exploits new ways of controlling the emission characteristics of nanophosphors, surpassing the limits imposed by conventional optics, through the use of nanophotonic concepts. The development of reliable and scalable nanophosphor-based photonic materials will allow ultimate spectral and angular control over the light emission properties, addressing the critical shortcomings of current LEDs. The new optical design of these devices will be based on multilayers, surface textures and nano-scatterers of controlled composition, size and shape, to attain large-area materials possessing photonic properties that will enable a precise management of the visible radiation.

Nanophom will significantly advance our comprehension of fundamental phenomena like the formation of photonic modes in complex optical media to which light can couple, as well as advancing the state of the art of high-efficiency solid-state lighting devices.

PERovskite SEmiconductors for PHOtoNics	**PERovskite SEmiconductors for PHOtoNics**
Código/Code:	H2020-MSCA-ITN-ETN/0748 Comisión Europea MSCA-ITN
Periodo/Period:	01-03-2021 / 28-02-2025
Organismo Financiador/Financial source:	Comisión Europea
Importe total/Total amount:	250.904,88 €
Investigador responsable/Research head:	Hernán R. Míguez García

ABSTRACT

Funded by the Marie Skłodowska-Curie programme, PERSEPHONe is a coordinated training network that aims to equip young researchers with new skills and knowledge regarding the development of a novel photonics technological platform based on metal-halide perovskite semiconductors. These materials present unrivalled optoelectronic properties and can be engineered to achieve a large set of desirable

functionalities which may change the roadmap of currently established photonic technologies. They also show great promise for their integration with silicon photonics and silicon-oxynitride-based photonics. The programme will expose 14 early-stage researchers to a wide spectrum of research activities including material synthesis, photonic (and optoelectronic) device and integrated circuit fabrication, characterisation, modelling, upscaling and manufacturing. PERSEPHONe will lay the foundation for a novel photonic technology, strengthening Europe's position in the field.

Diseño óptico optimizado de dispositivos optoelectrónicos basados en puntos cuánticos de perovskita sin ligandos	**Optimized photonic design of ligand-free perovskite quantum dot based optoelectronic devices**
Código/Code:	PID2020-116593RB-I00
Periodo/Period:	01-09-2021 / 31-08-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	302.500 €
Investigador responsable/Research head:	Hernán R. Míguez García y Mauricio E. Calvo Roggiani
Componentes/Research group:	Gabriel S. Lozano Barbero, Juan F. Galisteo López

RESUMEN / ABSTRACT

La motivación del proyecto FreeDot es triple. Primero, proponer soluciones a los inconvenientes específicos que obstaculizan un mayor desarrollo de la tecnología optoelectrónica de perovskita (inestabilidad, durabilidad, sensibilidad ambiental, etc.). La aproximación propuesta se basa en el desarrollo de células solares nanoestructuradas y LED basados en nuevas matrices porosas que permiten la síntesis de ensamblados de nanocristales sin ligandos con buenas propiedades de transporte de carga y, simultáneamente, minimizan su exposición a entornos degradantes. En segundo lugar, demostrar que es posible, también en el caso de dispositivos basados en puntos cuánticos, mejorar la eficiencia de conversión de energía en células solares y de extracción de luz en LEDs mediante la optimización del diseño óptico. Por último, la posibilidad de obtener nanocristales libres de ligandos abre la posibilidad de estudiar las propiedades fotofísicas fundamentales de los puntos cuánticos, lo que normalmente es obstaculizado por la presencia de capas orgánicas en el caso de puntos cuánticos coloidales.

The motivation of the FreeDot project is three-fold. First, to propose solutions to the specific drawbacks hindering further development of perovskite optoelectronic technology (instability, durability, environmental sensitivity, etc.) by developing nanostructured solar cells and LEDs based on novel porous scaffolds that permit the synthesis of ligand-free nanocrystal assemblies, which show dot-to-dot charge transport while, simultaneously, minimizing their exposure to degrading environments. Second, to prove that improved power conversion efficiency, in the case of solar cells, and enhanced outcoupling and control over the spectral and directional properties of the emitted light, in the case of LEDs, are achievable through the optimization of the optical design also for quantum dot based devices. Finally, the synthesis of ligand-free nanocrystals opens the possibility to study fundamental photophysical properties of quantum dots, which are hindered by the presence of organic cappings in colloidal nanocrystals.

Nanopartículas multifuncionales para la obtención de bioimágenes mediante luminiscencia, resonancia magnética y tomografía computerizada de Rayos X	**Multifunctional nanoparticles for luminescent, magnetic resonance and X-ray computed tomography bioimaging**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	RTI2018-094426-B-I00
Periodo/Period:	01-01-2019 / 30-09-2022
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Manuel Ocaña Jurado y Ana Isabel Becerro Nieto
Componentes/Research group:	Nuria O. Núñez Alvarez

RESUMEN / ABSTRACT

El proyecto persigue la preparación de nanopartículas (NPs) multifuncionales con propiedades mejoradas y características (tamaño, estabilidad coloidal y toxicidad) adecuadas que puedan emplearse en más de una modalidad de obtención de imágenes de órganos, tejidos y células, cuyo principal interés radica en que mediante un único tipo de sonda se podría obtener información complementaria esencial para un diagnóstico clínico más riguroso. En concreto, se estudiarán sondas bifuncionales para la obtención de imágenes mediante luminiscencia y resonancia magnética (MRI) o tomografía computarizada de rayos X (CT), y sondas trifuncionales con utilidad para las tres modalidades mencionadas. Se abordarán dos tipos de biosondas luminiscentes. Por una

parte, se diseñarán NPs luminiscentes constituidas por matrices singulares dopadas con cationes lantánidos (Nd^{3+} o Er^{3+}:Yb^{3+} o Tm^{3+}:Yb^{3+}), cuya excitación y emisión tiene lugar en la región del infrarrojo cercano (NIR) conocida como ventana biológica (650-1800 nm), en la que las radiaciones no son dañinas para los tejidos y tienen alto poder de penetración. Por otra parte, se persigue la obtención de NPs cuya luminiscencia persiste después de eliminar la excitación, evitándose así los posibles efectos no deseados de ésta (autofluorescencia de los tejidos, radiaciones dañinas). En el primer caso, se pretende conseguir una mayor estabilidad química y térmica de las sondas mediante la selección de matrices tipo oxifluoruro más estables que las de tipo fluoruro propuestas hasta ahora, En el segundo caso, se abordarán sistemas con luminiscencia persistente en el NIR con composición $ZnGa_20_4$:Cr^{3+} y $Y_3Al_2Ga_30_{12}$:Ce^{3+},Cr^{3+},Nd^{3+}, para los que es prioritario desarrollar nuevos métodos de síntesis que permitan la obtención de NPs uniformes, necesarias para este tipo de aplicaciones. Respecto a la modalidad MRI y en respuesta a la necesidad de desarrollar agentes de contraste para las más modernas técnicas que operan a campos magnéticos altos para aumentar la resolución de las imágenes y así obtener un diagnóstico más preciso, se planea desarrollar NPs constituidas por compuestos (oxifluoruros, vanadatos, fosfatos) de Dy y Ho. Por último, debido al alto número atómico de los elementos constituyentes de las sondas anteriores, es de esperar que éstas también tengan una alta capacidad de atenuación de rayos X, siendo por tanto también potencialmente útiles como agentes de contraste para CT. En este caso, el empleo de las NPs objeto de estudio aportará importantes ventajas respecto a los agentes comerciales utilizados en la actualidad que se traducen en un mayor control del tiempo de residencia en el organismo y de su biodistribución y, por tanto, en la posibilidad de disminuir las dosis utilizadas reportando así un beneficio para el paciente. El proyecto contempla tanto la fabricación de las sondas optimizadas como la exploración de su aplicabilidad al campo del diagnóstico clínico mediante la obtención de imágenes in vivo en ratones. El equipo investigador posee gran experiencia en la síntesis de NPs inorgánicas basadas en tierras raras y dispone de la mayoría de los medios necesarios para su caracterización. Además, dicho equipo cuenta con el apoyo de investigadores de otras instituciones, expertos en diversos aspectos del proyecto, que colaborarán en el desarrollo de algunas tareas del mismo como vienen haciendo desde hace varios años, lo que garantiza el correcto desarrollo de la propuesta.

The project pursues the preparation of multifunctional nanoparticles (NPs) with improved properties and suitable characteristics (size, colloidal stability and toxicity) that can be used to get images of cells, tissues and organs by means of more than one bioimaging technique, thus providing complementary information essential for a more reliable medical diagnosis. Specifically, we shall study bifunctional probes for both, luminescence and magnetic resonance (MRI) or luminescence and X-ray computed tomography (CT), and trifunctional probes that are useful for the three imaging techniques. Two types of luminescent probes will be addressed. On the one hand, luminescent NPs will be designed consisting of single matrices doped with lanthanide cations (Nd^{3+} o Er^{3+}:Yb^{3+} o Tm^{3+}:Yb^{3+}), whose excitation and emission takes place in the near-infrared (NIR) region known as the biological window (650-1800 nm), in which radiation is not harmful to tissues and has a high penetration power. On the other hand, nanoprobes whose luminescence persists after ceasing the excitation will be also developed, thus avoiding the possible undesirable effects of the excitation radiation on the tissues. In the first case, our aim is to achieve greater chemical and thermal stability of the probes by selecting oxifluoride-type matrices, more stable than the fluoride-type matrices proposed so far. In the second case, the aim of the project resides in the exploration of new synthetic routes to obtain nanoparticulated $ZnGa_{2}0_{4}:Cr^{3+}$ and $Y_{3}Al_{2}Ga_{3}0_{12}:Ce^{3+}$, Cr^{3+}, Nd^{3+}, with uniform size and shape, which are essential for bioapplications. Regarding MRI technique, this project aims at developing NPs made up of Dy- and Ho-based oxifluorides, vanadates and phosphates in response to the need of new contrast agents that work at high magnetic fields, which are increasingly being used in clinics to improve image resolution. Finally, due to the high atomic number of the constituent elements of the selected probes, it is expected that they show a high X-ray attenuation capacity, being therefore also useful as CT contrast agents. The advantage of the NPs proposed in this research with respect to the CT CAs currently used in clinics is the longer circulation time of the former, which will allow decreasing considerably the dosage to be given to the patient. The project contemplates both the manufacture of optimised probes and the exploration of their applicability to the field of medical diagnosis by obtaining "in vivo" images in mice. The research team has long experience in the synthesis of rare earths-based inorganic NPs and has most of the necessary equipment for their characterisation. The participation in the work plan of researchers from other institutions, with long expertise on various aspects of the project, who have successfully collaborated with the research team, gives further support to the viability of the proposal.

Materiales ópticos avanzados para dispositivos optoelectrónicos más eficientes | MODO	**Advanced optical materials for more efficient optoelectronic devices | MODO**
MINISTERIO DE CIENCIA E INNOVACIÓN UNIÓN EUROPEA FONDO EUROPEO DE DESARROLLO REGIONAL "Una manera de hacer Europa" AGENCIA ESTATAL DE INVESTIGACIÓN	
Código/Code:	MAT2017-88584-R
Periodo/Period:	01-01-2018 / 30-09-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Hernán Míguez García, Mauricio E. Calvo Roggiani
Componentes/Research group:	Juan F. Galisteo López, Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El proyecto MODO busca optimizar el diseño óptico de dispositivos optoelectrónicos, ya sean fotovoltaicos o emisores de luz, con el objetivo de aumentar su eficiencia o dotarles de nueva funcionalidad. La hipótesis en la que se basa este proyecto es que este objetivo principal puede lograrse mediante la integración de materiales ópticos que permitan controlar la interacción radiación-materia en las láminas absorbentes u ópticamente activas del dispositivo. La estrategia propuesta consiste en realizar, secuencialmente, el diseño, preparación, caracterización e integración en prototipos de estructuras fotónicas de diverso tipo (cristales fotónicos, partículas metálicas, medios ópticos desordenados, superficies corrugadas), empleando principalmente técnicas de procesado en solución escalables y compatibles con las usualmente empleadas para la fabricación de los dispositivos objeto de estudio.

La tecnología optoelectrónica basada en perovskitas ha despertado en los últimos años un interés extraordinario debido a las altas eficiencias de conversión de energía lumínica en eléctrica, superiores al 20%, que se han alcanzado en un tiempo relativamente corto comparado con la evolución de otras tecnologías fotovoltaicas. Por otro lado, presentan elevados rendimientos cuánticos de fotoemisión en el verde y el rojo, lo que también las convierte en candidatas a recubrimientos emisores de luz. Sin embargo, estas expectativas se ven parcialmente ensombrecidas debido a los problemas de estabilidad y potencial impacto ambiental que estas perovskitas presentan. Es uno de los objetivos prioritarios de este proyecto proponer soluciones a estos problemas concretos a través de diseños ópticos que permitan reducir tanto la cantidad de material empleado como la exposición de la lámina activa a ambientes que induzcan su degradación. Se pretende ahondar en la comprensión de los fenómenos que intervienen en la degradación fotoinducida de estos materiales en entornos de distinto tipo, lo que nos permitirá proponer soluciones concretas para desarrollar láminas de perovskita más estables y eficientes. Por otra parte, el proyecto MODO explorará la mejora de una nueva tecnología fotovoltaica, propuesta muy recientemente, basada en nanocristales semiconductores de $AgBiS_2$, de muy bajo impacto ambiental pero con una absorción óptica aún muy inferior al resto de celdas solares emergentes.

Simultáneamente, se aplicarán a dispositivos emisores de luz, basados en nanocristales semiconductores y compuestos orgánicos foto- y electroluminiscentes, conceptos orientados a la amplificación direccional de la luminiscencia en rangos espectrales seleccionados a través del control de la densidad local de estados fotónicos. Se explorará la posibilidad de controlar la dinámica de decaimiento de estados excitados a escala macroscópica y de observar la emisión láser. En todos los casos, el rendimiento energético de los dispositivos objeto de estudio no ha sido optimizado anteriormente desde el punto de vista del diseño óptico.

La propuesta se enmarca dentro del Reto Social denominado "Energía segura, eficiente y limpia" y tiene como objetivo desarrollar tecnología fotónica empleando herramientas de la nanotecnología y del campo de materiales avanzados, todas ellas identificadas como Tecnologías Facilitadoras Esenciales en el programa de H2020 e incluidas en la Estrategia Española de Ciencia y Tecnología.

The MODO project is focused on the optimization of the optical design of optoelectronic devices, be they photovoltaic or light emitting ones, with the aim of increasing their efficiency or endow them with new functionalities. The hypothesis on which it is based is that this goal can be reached by means of the integration of optical materials that allow controlling the radiation-matter interaction in the absorbing or optically active layers of the device. The strategy herein proposed is based on the sequential realization of design, preparation, characterization and integration of devices of diverse photonic structures (photonic crystals, metallic particles, disordered optical media, corrugated surfaces) employing mainly solution processing techniques fully compatible with those used to fabricate the targeted devices. Optoelectronic technology based on perovskites has attracted a great deal of interest in the last years as a result of the high solar to electric power conversion efficiency, above 20%, that have been reached in a relatively short time compared to other photovoltaic technologies. At the same time, they present high photoemission quantum yields in the green and the red, which make them also good candidates as color converter layers for LEDs. However, these expectations are partially threatened by both the stability problems and potentially toxic environmental effects they present. It is one of the main goals of this project to propose solutions to specific drawbacks present in the optoelectronic technology based on hybrid perovskites through the implementation of optical designs that gives rise to a reduction of both the amount of material employed as well as the exposure to environments that typically degrade them. We seek to deepen our understanding of phenomena that give rise to the photoinduced degradation of these materials when exposed to diverse environments, which will allow us to propose specific solutions to develop more stable and efficient perovskite layers. Simultaneously, concepts based on the strict control over the local density of photon states and oriented to the directional amplification of luminescence at selected spectral ranges will be applied to light emitting devices based on semiconductor nanocrystals as well as to photo- and electro-luminescent organic compounds. Full control over the excited state decay dynamics over large areas and observation of laser emission will also be sought after. In all cases, the energy efficiency of the targeted devices has not been optimized before from the point of view of the optical design.

The proposal is included in the framework of the Societal Challenge called "Secure, clean and efficient energy" and aims to develop photonic technology using nanotechnology tools and in the advanced materials field, all identified as Key Enabling Technologies KETs in the Spanish Strategy on Science and Technology, aligned with the European Program H2020.

Verificación de la existencia de fuerzas de Casimir repulsivas en la macroescala en láminas delgadas suspendidas y autosoportadas | VERSUS	**Verification of the existence of macroscale repulsive Casimir forces in suspended self-standing films | VERSUS**
Código/Code:	FIS2017-91018-EXP "Explora"
Periodo/Period:	01-11-2018 / 30-04-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	36.300 €
Investigador responsable/Research head:	Hernán Ruy Míguez García
Componentes/Research group:	Mauricio E. Calvo Roggiani, Juan F. Galisteo López

RESUMEN / ABSTRACT

El proyecto VERSUS tiene como objetivo principal realizar la primera observación de fuerzas de Casimir-Lifshitz repulsivas en sistemas macroscópicos plano-paralelos. Para esto se centrará en el diseño, fabricación y caracterización de materiales ópticos que permitan controlar la intensidad y naturaleza de la fuerza de Casimir-Lifshitz, de forma que puedan observarse y caracterizarse fenómenos de levitación debido al balance de ésta y la fuerza gravitatoria. Esta propuesta, radicalmente novedosa, hace uso de técnicas de espectroscopía óptica (basadas en interferencia óptica entre los haces parcialmente reflejados y transmitido en las intercaras del sistema plano-paralelo) para caracterizar la distancia de equilibrio a la que el sistema levita sobre un sustrato. Para ello, se parte de diseños de materiales cuyas propiedades ópticas y densidades son tales que sumergidas en distintos fluidos leviten sobre ciertos sustratos como resultado del balance de esas fuerzas, como recientemente ha sido demostrado de forma teórica por el equipo solicitante. Nuestro grupo ha demostrado teóricamente que existen combinaciones de materiales que fabricados en forma de láminas delgadas (< 1 micra) pueden levitar a distancias del orden de las pocas decenas o centenas de nanómetros sobre un sustrato adecuado. En particular, láminas de teflón, poliestireno y sílice inmersas en glicerol levitan sobre una oblea de silicio, siendo las distancias de equilibrio controlables y sintonizables a través del grosor de las láminas delgadas y la temperatura del sistema. Las láminas delgadas autosoportadas deben ser compactas, mecánicamente estables, de superficies planas, grosor controlado y químicamente afines al fluido en el que están sumergidas. La observación macroscópica de fuerzas repulsivas de Casimir-Lifshitz, nunca reportada anteriormente, mediante medidas de espectroscopía óptica constituiría un logro sin precedentes en el campo del estudio de las interacciones fundamentales de la materia.

The ultimate goal of the VERSUS project is the first observation of repulsive Casimir-Lifshitz forces in macroscopic plane-parallel systems. To this end, it will focus on the design, fabrication, and characterization of optical materials that allow controlling the intensity and nature of the Casimir-Lifshitz force, so that levitation phenomena can be observed and characterized due to the balance between the latter and gravity force. This radically new approach makes use of optical spectroscopic techniques (based on optical interferometry between the partially reflected and transmitted light at the interfaces of the plane-parallel system) for characterizing the equilibrium distance at which the system levitates over a substrate. According to very recent results attained by the applicant group, it is possible to find materials whose optical constants and densities are such that when they are immersed in a fluid they can levitate over a substrate as a result of the aforementioned force balance. Our group has recently demonstrated theoretically that there is a number of materials that prepared in this films (<1 micrometer) can levitate several tens or hundreds of nanometers over a carefully selected substrate. Specifically, thin layers made of teflon, polystyrene or silicon dioxide immersed in glycerol are expected to levitate over a silicon wafer, being possible to tune the equilibrium distances at which such layers will be suspended through their thicknesses and temperature of the system. The devised self-standing thin films (in single layers or multilayer arrangements) must be compact, mechanically stable, of smooth surfaces, of controlled thickness, and chemically compatible with the fluid in which they are immersed. The macroscopic observation of repulsive Casimir-Lifshitz forces, never reported before, through optical spectroscopic measurements would constitute an unprecedented milestone in the field of fundamental matter interactions.

Nuevos agentes de contraste multimodales para el diagnóstico médico por imagen	**New multimodal contrast agents for medical diagnostic imaging**
Código/Code:	P20_00182
Periodo/Period:	05-10-2021 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	82.050 €
Investigador responsable/Research head:	Ana Isabel Becerro Nieto
Componentes/Research group:	Manuel Ocaña Jurado, Nuria O. Núñez Álvarez, María Luisa García Martín (IIBM)

RESUMEN / ABSTRACT

El proyecto persigue el diseño de agentes de contraste (CAs) multimodales para el registro de imágenes para diagnóstico médico. Estos CAs estarán constituidos por nanopartículas inorgánicas basadas en lantánidos con propiedades adecuadas para el registro de imágenes mediante técnicas complementarias, con objeto de obtener información esencial para un diagnóstico médico más riguroso sin necesidad de inyectar al paciente CAs específicos para cada técnica. Una ventaja adicional de las sondas propuestas respecto a los CAs comerciales es que permiten controlar el tiempo de residencia en el organismo y su biodistribución y, por tanto, disminuir las dosis necesarias, resultando en un claro beneficio para el paciente. En concreto, se desarrollarán agentes de contraste para resonancia magnética (MRI) dual con funcionalidad adicional como agentes de contraste para tomografía computarizada de rayos X (CT) e imagen luminiscente en la región del infrarrojo cercano (NIR) conocida como ventana biológica (650-1800 nm), en la que las radiaciones no son dañinas para los tejidos y tienen alto poder de penetración en los mismos. Se ensayarán varias composiciones: fosfatos, vanadatos, molibdatos y volframatos de elementos lantánidos tales como el Gd, Dy y Ho, que aportarán la funcionalidad magnética y cuyo alto número atómico es óptimo para CT. El dopado de todas ellas con Nd^{3+} permitirá la obtención de imágenes luminiscentes en el NIR. La exploración de la aplicabilidad de dichas sondas al campo del diagnóstico médico por imagen se llevará a cabo mediante la obtención de imagen “in vivo” en ratones.

The project aims to design multimodal contrast agents (CAs) for medical diagnostic imaging. The CAs will consist of lanthanide-based inorganic nanoparticles with properties suitable for different bioimaging techniques. The CAs developed will allow obtaining a more rigorous medical diagnosis without the need to inject the patient with several technique-specific CAs. An additional advantage of the proposed probes over commercial CAs is that they allow control of the residence time in the body and their biodistribution, and thus reduce the doses needed, resulting in a clear benefit for the patient. Specifically, dual magnetic resonance imaging (MRI) CAs will be developed with additional functionality as contrast agents for X-ray computed tomography (CT) and luminescence imaging in the near-infrared (NIR) region known as the biological window (650-1800 nm), where radiation is not harmful to tissues and has high tissue penetration power. Several compositions will be tested: phosphates, vanadates, molybdates, and wolframates of lanthanide elements such as Gd, Dy, and Ho, which will provide the magnetic functionality and whose high atomic number is optimal for CT. Doping all of them with Nd^{3+} will allow luminescent imaging in the NIR. The applicability of these probes to medical imaging will be explored by in vivo imaging in mice.

Desarrollo de Dispositivos Emisores de Luz basados en Perovskita Nanoestructurada |Nano-ABX LED	**Development of light emitting devices based on nanostructured perovskite | Nano-ABX LED**
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P18-RT-2291
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	122.968 €
Investigador responsable/Research head:	Hernán R. Míguez García
Componentes/Research group:	Juan Francisco Galisteo López, Mauricio E. Calvo Roggiani, Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El proyecto Nano-ABX LED se centra en encontrar vías de solución a los principales retos que enfrenta el campo de la emisión de luz basada en perovskitas. Estos son la inestabilidad química y térmica de las perovskitas, así como la dificultad de mantener una eficiencia cuántica elevada independientemente del color de emisión, lo que dificulta la obtención tanto de una gama de colores variada como de distintos tonos de blanco (i.e., distintas temperaturas de color).

El proyecto Nano-ABX LED surge con la motivación de encontrar soluciones a estos problemas. Partiendo de resultados preliminares recientes del grupo de Materiales Ópticos Multifuncionales, se intentará demostrar que la integración de nanocristales de perovskita híbrida en el interior de matrices con porosidad controlada mejora extraordinariamente la estabilidad ambiental de estos materiales, un aspecto que el grupo solicitante de esta propuesta ha estudiado en profundidad, así como permite aumentar el rendimiento cuántico luminiscente a longitudes de onda de emisión controladas. En otra vertiente del proyecto, se explorará el aumento de eficiencia y prestaciones (direccionalidad, control espectral) de los dispositivos a través de la integración de distintas estructuras fotónicas, tomando como punto de partida.

The Nano-ABX LED project focuses on finding ways to solve the main challenges facing the field of perovskite-based light emission. These are the chemical and thermal instability of perovskites, as well as the difficulty of maintaining a high quantum efficiency regardless of the emission color, which makes it difficult to obtain both a varied color range and different shades of white (i.e., different temperatures color).

The Nano-ABX LED project arises with the motivation to find solutions to these problems. Based on recent preliminary results of the Multifunctional Optical Materials

Group, an attempt will be made to demonstrate that the integration of hybrid perovskite nanocrystals inside matrices with controlled porosity dramatically improves the environmental stability of these materials, an aspect that the group requesting this proposal has studied in depth, as well as it allows to increase the luminescent quantum efficiency at controlled emission wavelengths. In another aspect of the project, the increase in efficiency and performance (directionality, spectral control) of the devices will be explored through the integration of different photonic structures, taking as a starting point.

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de Dispositivos Emisores de Luz basados en Perovskita Nanoestructurada

Código/Code: 201960E090
Periodo/Period: 01-09-2019 / 31-08-2024
Organismo Financiador/Financial source: CSIC (Intramural)
Importe total/Total amount: 187.789 €
Investigador responsable/Research head: Hernán Míguez García

Energía Solar y nanociencia. Kit demostrativo

Código/Code: 201560E056
Periodo/Period: 01-01-2020 / 31-09-2021
Organismo Financiador/Financial source: Fundación General CSIC
Importe total/Total amount: 2.300 €
Investigador responsable/Research head: Mauricio Calvo Roggiani

Ayudas extraordinarias para la preparación de proyectos | MAT2017-88584

Código/Code: 2020AEP094
Periodo/Period: 01-01-2021 / 31-08-2021
Organismo Financiador/Financial source: CSIC
Importe total/Total amount: 13.039 €
Investigador responsable/Research head: Hernán Míguez García

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Sistema de caracterización óptica avanzada en atmósfera inerte (EQC2019-005556-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 121.917,11 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Materiales Ópticos Multifuncionales"

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Diseño y preparación de filtros dicroicos para su integración en módulos fotovoltaicos

Periodo/Period:	01-01-2021 / 31-12-2022
Organismo Financiador/Financial source:	BLUESOLAR FILTERS, S.L.
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Hernán Míguez García

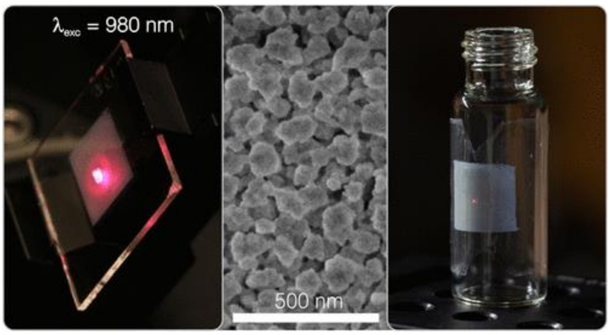
PATENTES / PATENTS

Ceramic material with a garnet structure showing a nonstoichiometry, synthesis and uses thereof
Inventores: Ana Isabel Becerro Nieto
Tipo de Patente: Internacional
Número de Solicitud: 21305159
Fecha Solicitud: 5 de febrero de 2021
Entidad Titular: CNRS, Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Highly Versatile Upconverting Oxyfluoride-Based Nanophosphor Films

Ngo, TT; Cabello-Olmo, E; Arroyo, E; Becerro, AI; Ocaña, M; Lozano, G; Míguez, H
ACS Applied Materials & Interfaces, **13** (2021) 30051-30060
Junio 2021 | **DOI:** 10.1021/acsami.1c07012



Fluoride-based compounds doped with rare-earth cations are the preferred choice of materials to achieve efficient upconversion, of interest for a plethora of applications ranging from bioimaging to energy harvesting. Herein, we demonstrate a simple route to fabricate bright upconverting films that are transparent, self-standing, flexible, and emit different colors. Starting from the solvothermal synthesis of uniform and colloidally stable yttrium fluoride nanoparticles doped with Yb^{3+} and Er^{3+}, Ho^{3+}, or Tm^{3+}, we find the experimental conditions to process the nanophosphors as optical quality films of controlled thickness between few hundreds of nanometers and several micrometers. A thorough analysis of both structural and photophysical properties of films annealed at different temperatures reveals a tradeoff between the oxidation of the matrix, which transitions through an oxyfluoride crystal phase, and the efficiency of the upconversion photoluminescence process. It represents a significant step forward in the understanding of the fundamental properties of upconverting materials and can be leveraged for the optimization of upconversion systems in general. We prove bright multicolor upconversion photoluminescence in oxyfluoride-based phosphor transparent films upon excitation with a 980 nm laser for both rigid and flexible versions of the layers, being possible to use the latter to coat surfaces of arbitrary shape. Our results pave the way toward the development of upconverting coatings that can be conveniently integrated in applications that demand a large degree of versatility.

Enhanced Directional Light Extraction from Patterned Rare-Earth Phosphor Films

Cabello-Olmo, E; Molet, P; Mihi, A; Lozano, G; Míguez, H
Advanced Optical Materials, **9** (2021) 2001611
Enero 2021 | **DOI:** 10.1002/adom.202001611

The combination of light-emitting diodes (LEDs) and rare earth (RE) phosphors as color-converting layers comprises the basis of solid-state lighting. Indeed, most LED lamps include a photoluminescent coating made of phosphor material, i.e., crystalline matrix suitably doped with RE elements, to produce white light from a blue or ultraviolet LED chip. Transparent phosphor-based films constitute starting materials for new refined emitters that allow different photonic designs to be implemented. Among the different photonic strategies typically employed to tune or enhance emission, surface texturing has proved its versatility and feasibility in a wide range of materials and devices. However, most of the nanofabrication techniques cannot be applied to RE phosphors directly because of their chemical stability or because of their cost. The first monolithic patterned structure of down-shifting nanophosphors with square arrays of nanoholes with different lattice parameters is reported in this study. It is shown that a low-cost soft-nanolithography procedure can be applied to red-emitting nanophosphors ($GdVO_4$:Eu^{3+} nanocrystals) to tune their emission properties, attaining a twofold directional enhancement of the emitted light at predesigned emission wavelengths in specific directions.

The Complex Interplay of Lead Halide Perovskites with Their Surroundings

Galisteo-López, JF; Calvo, ME; Míguez, H
Advanced Optical Materials, **9** (2021) 2100133
Marzo 2021 | **DOI:** 10.1002/adom.202100133

Photoexcitation of lead halide perovskites induces a restructuration of the material that simultaneously enhances its emission properties and triggers its degradation. These concomitant processes are strongly dependent on the surroundings of the perovskite, both while and after being processed, underlining the relevance the environment and the interfacial design have in the stability and performance of these materials and the devices based on them. This shocking observation reveals that when subjected to external illumination, lead halide perovskites undergo a number of photophysical processes that strongly modify their structure and thus their optoelectronic properties. Such photoinduced instability stems from a defective structure directly linked to the low-temperature and solution-processed fabrication routes generally employed to build perovskite solar cells with efficiencies comparable to state-of-the-art values. On the other hand, these same inexpensive and unsophisticated procedures make this material a promising component in energy conversion devices. Here, an analysis is provided regarding the different impact on the perovskite structure, hence on its optoelectronic performance, that the interaction with its surroundings has, providing specific examples that highlight this interplay, describing the kind of modification it induces, and listing the related effects on the optoelectronic properties that should be accounted for when characterizing them.

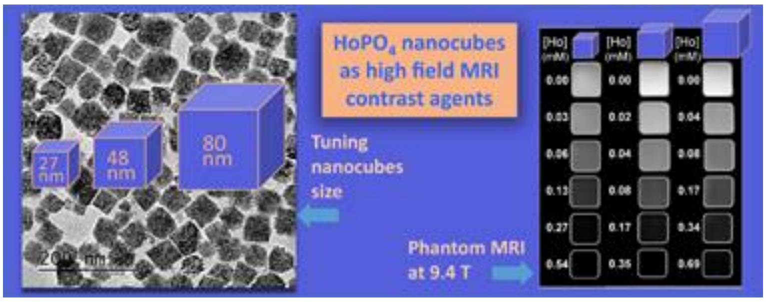
The Role of the Atmosphere on the Photophysics of Ligand-Free Lead-Halide Perovskite Nanocrystals

Morán-Pedroso, M; Rubino, A; Calvo, ME; Espinós, JP; Galisteo-López, JF; Míguez, H
Advanced Optical Materials, **9** (2021) 2100605
Septiembre 2021 | **DOI:** 10.1002/adom.202100605

Lead halide perovskite (LHP) nanocrystals (NCs) have gained attention over the past decade due to their outstanding optoelectronic properties, making them a suitable material for efficient photovoltaic and light emitting devices. Due to its soft nature, these nanostructures undergo strong structural changes upon irradiation, where these light-induced processes are strongly influenced by the environment. Since most processing routes for LHP NCs are based on colloidal approaches, the role of factors such as stabilizing ligands or solvents is usually hard to disentangle from the interaction of external radiation with the perovskite material. Employing a recently proposed synthetic approach, where ligand-free NCs can be grown within metal-oxide-based insulating nanoporous matrices, it has been feasible to perform a clean study of the effect of the surrounding atmosphere on the photophysical properties of perovskite NCs, avoiding the interference of protective capping layers or solvents. Simultaneous light-induced photo-activation and darkening processes are monitored and disentangled, and their relation with bulk and surface processes, respectively, demonstrated.

Holmium phosphate nanoparticles as negative contrast agents for high-field magnetic resonance imaging: Synthesis, magnetic relaxivity study and in vivo evaluation

Gómez-González, E; Caro, C; Martínez-Gutiérrez, D; García-Martín, ML; Ocaña, M; Becerro, AI
Journal of Colloid and Interface Science, **587** (2021) 131-140
Abril 2021 | **DOI:** 10.1016/j.jcis.2020.11.119



The increasing use of high magnetic fields in magnetic resonance imaging (MRI) scanners demands new contrast agents, since those used in low field instruments are not effective at high fields. In this paper, we report the synthesis of a negative MRI contrast agent consisting of $HoPO_4$ nanoparticles (NPs). Three different sizes (27 nm, 48 nm and 80 nm) of cube-shaped NPs were obtained by homogeneous precipitation in polyol medium and then coated with poly(acrylic) acid (PAA) to obtain stable colloidal suspensions of $HoPO_4$@PAA NPs in physiological medium (PBS). The transverse relaxivity (r_2) of aqueous suspensions of the resulting NPs was evaluated at both 1.44 T and 9.4 T. A positive correlation between r2 values and field strength as well as between r_2 values and particle size at both magnetic field strengths was found although this correlation failed for the biggest NPs at 9.4 T, likely due to certain particles aggregation inside the magnet. The highest r_2 value (489.91 $mM^{-1}s^{-1}$) was found for the 48 nm NPs at 9.4 T. Toxicity studies demonstrated that the latter NPs exhibited low toxicity to living systems. Finally, in vivo studies demonstrated that $HoPO_4$@PAA NPs could be a great platform for next-generation T_2-weighted MRI contrast agents at high magnetic field.

Light-Harvesting Properties of a Subphthalocyanine Solar Absorber Coupled to an Optical Cavity

Esteso, V; Caliò, L; Espinós, H; Lavarda, G; Torres, T; Feist, J; García-Vidal, FJ; Bottari, G; Míguez, H
SOLAR RRL, **5** (2021) 2100308
Julio 2021 | **DOI:** 10.1016/j.jcis.2020.11.119

Herein, both from the experimental and theoretical point of view, the optical absorption properties of a subphthalocyanine (SubPc), an organic macrocycle commonly used as a sunlight harvester, coupled to metallic optical cavities are analyzed. How different electronic transitions characteristic of this compound and specifically those that give rise to excitonic (Q band) and charge transfer (CT band) transitions couple to optical cavity modes is investigated. It is observed that whereas the CT band couples weakly to the cavity, the Q band transitions show evidence of hybridization with the photon eigenstates of the resonator, a distinctive trait of the strong coupling regime. As a result of the different coupling regimes of the two electronic transitions, very different spectral and directional light-harvesting features are observed, which for the weakly coupled CT transitions are mainly determined by the highly dispersive cavity modes and for the strongly coupled Q band by the less angle-dependent exciton-polariton bands. Modeling also allows discriminating parasitic from productive absorption in each case, enabling the estimation of the expected losses in a solar cell acting as an optical resonator.

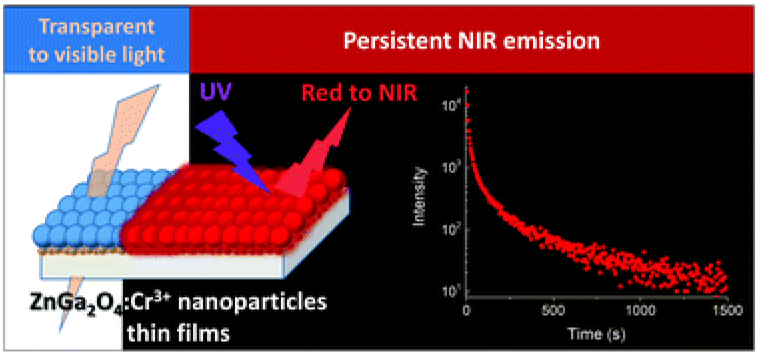
Ligand-Free $MAPbI_3$ Quantum Dot Solar Cells Based on Nanostructured Insulating Matrices

Rubino, A; Caliò, L; Calvo, ME; Míguez, H
SOLAR RRL, **5** (2021) 2100204
Julio 2021 | **DOI:** 10.1002/solr.202100204

The stability, either chemical or thermal, and performance of colloidal quantum dot (CQD) devices are typically limited by the presence of surface-bonded organic ligands required to stabilize the nanocrystals. In addition, optimization of charge transport implies lengthy ligand exchange processing. Herein, evidence of efficient charge transport through a network of ligand-free perovskite quantum dots (PQDs) embedded in an insulating porous matrix made of monodisperse SiO_2 nanoparticles is shown. Methylammonium lead iodide ($CH_3NH_3PbI_3$ or $MAPbI_3$) QDs are prepared in situ by infiltration of precursors within the matrix pores, which act both as nanoreactors for the synthetic reaction and as supporting scaffolds, hence reducing the number of synthetic and postprocessing steps usually required in CQD solar cells. Above a certain nanocrystal load, charge percolation is reached and dot-to-dot transport achieved without compromising quantum confinement effects. Solar cells based on $MAPbI_3$ QDs prepared in this way present a 9.3% efficiency, the highest reported for a scaffold-supported PQD solar cell, and significantly improved stability under solar illumination with respect to their bulk counterparts. Therefore, adequately designed networks of ligand-free PQDs can be used as both light harvesters and photocarrier conductors, in an alternative configuration to that used in previously developed QD solar cells.

Persistent luminescence of transparent $ZnGa_2O_4:Cr^{3+}$ thin films from colloidal nanoparticles of tunable size

Arroyo, E; Medrán, B; Castaing, V; Lozano, G; Ocaña, M; Becerro, AI
Journal of Materials Chemistry C, **9** (2021) 4474-4485
Marzo 2021 | **DOI:** 10.1039/d1tc00258a

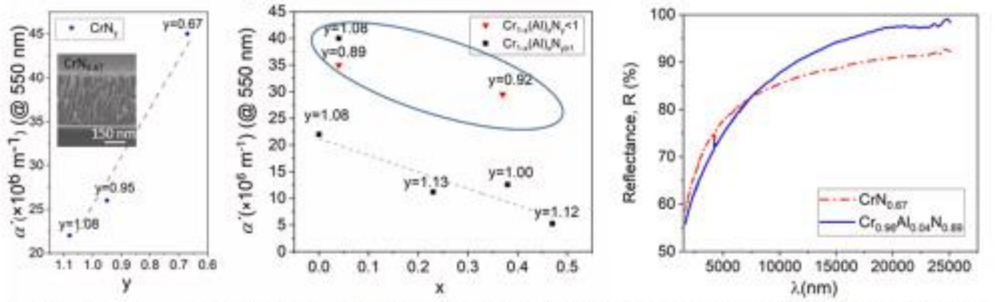


We report on the fabrication of $ZnGa_2O_4:Cr^{3+}$ transparent thin films and the evaluation, for the first time in the literature, of their persistent red to NIR emission. For this purpose, we have used a simple and economic global strategy based on wet processing methods from colloidal nanospheres with uniform size. A microwave-assisted hydrothermal method was first developed for the synthesis of precursor particles, which allows size tuning from 300 nm to 30 nm through simple modification of the Zn^{2+} precursor and the Cr^{3+} content of the starting solutions. $ZnGa_2O_4:Cr^{3+}$ transparent thin films over quartz substrates were then easily fabricated by spin coating, and their structural and optical characteristics were analyzed in detail after annealing at high temperature to elucidate the effect of processing temperature and particle size on the properties of the films. Indeed, our results indicate that high temperature annealing does not compromise the transparency of the films but improves their photoluminescence. In addition, the analysis reveals that persistence luminescence in our films is rather independent of the size of the precursor nanoparticles. Due to their transparency and persistent emission properties, films fabricated from colloidal suspensions of $ZnGa_2O_4:Cr^{3+}$ nanoparticles show great potential for application in the fields of chemical sensing, information storage, labelling, and anti-counterfeiting technology.

High-temperature solar-selective coatings based on Cr(Al)N. Part 1: Microstructure and optical properties of CrN_y and $Cr_{1-x}Al_xN_y$ films prepared by DC/HiPIMS

Rojas, TC; Caro, A; Lozano, G.; Sánchez-López, JC
Solar Energy Materials and Solar Cells, **223** (2021) 110951
Mayo 2021 | **DOI:** 10.1016/j.solmat.2020.110951

Tuneable $Cr_x(Al)_{1-x}N_y$ optical properties by controlling N vacancies and Al contents



- $Cr_{0.96}Al_{0.04}N_{1.08}$ and $Cr_x(Al)_{1-x}N_{y<1}$ as absorber layers in multilayer-based solar selective coatings (SSC)
- $CrN_{0.67}$ and $Cr_{0.96}Al_{0.04}N_{0.89}$ films as IR-reflector/absorber layers for SSC

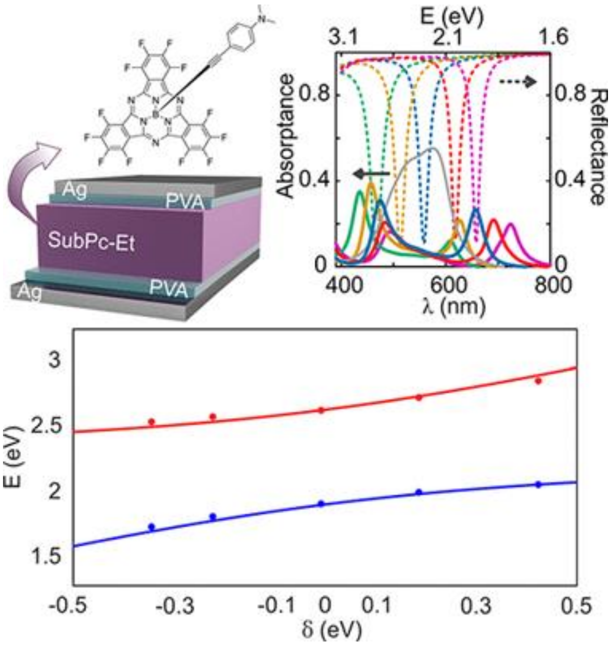
In order to explore the potentialities of $Cr_{1-x}(Al)_xN_y$ materials in multilayer-based solar selective coatings (SSC) for high temperature applications (T > 500 °C), the optical behavior of $Cr_{1-x}(Al)_xN_y$ films is studied in this work. Two sets of layers (CrN_y and $Cr_{1-x}Al_xN_y$) were prepared by direct current (DC) and high-power impulse magnetron sputtering (HiPIMS) technology. The deposition parameters: N_2 flux, HiPIMS frequency and aluminum sputtering power, were modified to get a wide variety of stoichiometries. The composition, morphology, phases and electronic structure of the films were characterized in depth. The optical behavior was determined by UV–Vis–NIR and FTIR spectroscopies, and the optical constants were obtained from the measured transmittance and reflectance spectra based on appropriate dielectric function models. Our results indicate that small changes in the layer composition influence the optical constants. In particular, a metallic-like behavior was obtained for CrN_y layers with N vacancies ($CrN_{0.95}$ and $CrN_{0.67}$ films) while a semiconductor-like behavior was observed for $CrN_{1.08}$. Thus, the CrN_y films can be used as effective absorber layer in multilayer-based SSC, and namely, the $CrN_{0.67}$ film as an IR reflector/absorber layer too. Likewise, the optical properties of $Cr_{1-x}Al_xN_y$ layers can also be tuned from metallic to semiconductor-like behavior depending on the chemical composition. Indeed, the absorption coefficients of $Cr_{1-x}Al_xN_y$ films with optimized Al content and N-vacancies are comparable to those reported for state-of-the-art materials such as TiAlN or TiAlCrN. In addition, a $Cr_{0.96}Al_{0.04}N_{0.89}$ film was found to be a suitable IR reflector/absorber layer.

Ultrastrong Exciton-Photon Coupling in Broadband Solar Absorbers

Bujalance, C; Esteso, V; Caliò, L; Lavarda, G; Torres, T; Feist, J; García-Vidal, FJ; Bottari, G; Míguez, H

Journal of Physical Chemistry Letters, **12** (2021) 10706-10712
Noviembre 2021 | **DOI:** 10.1021/acs.jpclett.1c02898

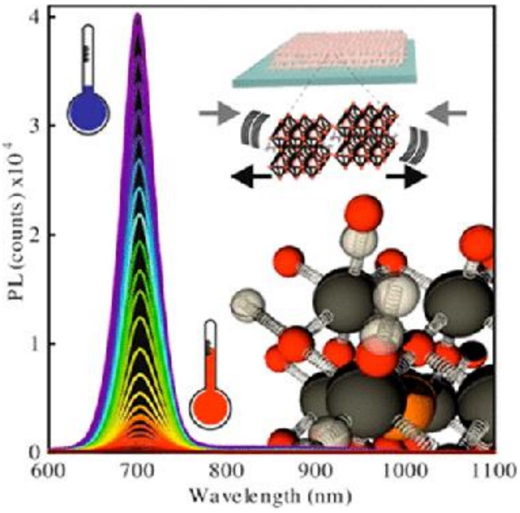
The recent development of organic polaritonic solar cells, in which sunlight absorbers and photon modes of a resonator are hybridized as a result of their strong coupling, has revealed the potential this interaction offers to control and enhance the performance of these devices. In this approach, the photovoltaic cell is built in such a way that it also behaves as an optical cavity supporting spectrally well-defined resonances, which match the broad absorption bands of the dyes employed. Herein we focus on the experimental and theoretical analysis of the specific spectral and angular optical absorption characteristics of a broadband light harvester, namely a subphthalocyanine, when operating in the ultrastrong coupling regime. We discuss the implications of having a broad distribution of oscillator strengths and demonstrate that rational



design of the layered structure is needed to optimize both the spectral and the angular response of the sunlight harvester dye.

Disentangling Electron–Phonon Coupling and Thermal Expansion Effects in the Band Gap Renormalization of Perovskite Nanocrystals

Rubino, A; Francisco-López, A.; Baker, A.J., Petrozza, A.; Calvo, M.E.; Goñi, A.R.; Míguez, H.
Journal of Physical Chemistry Letters, **12** (2021) 569-575
Enero 2021 | **DOI:** 10.1021/acs.jpclett.0c03042



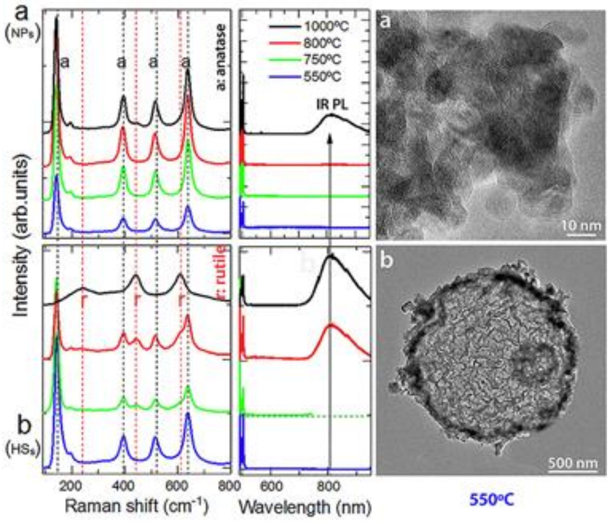
The complex electron–phonon interaction occurring in bulk lead halide perovskites gives rise to anomalous temperature dependences, like the widening of the electronic band gap as temperature increases. However, possible confinement effects on the electron–phonon coupling in the nanocrystalline version of these materials remain unexplored. Herein, we study the temperature (ranging from 80 K to ambient) and hydrostatic pressure (from atmospheric to 0.6 GPa) dependence of the photoluminescence of ligand-free methylammonium lead triiodide nanocrystals with controlled sizes embedded in a porous silica matrix. This analysis allowed us to disentangle the effects of thermal expansion and electron–phonon interaction. As the crystallite size decreases, the electron–phonon contribution to the gap renormalization gains in importance. We provide a plausible explanation for this observation in terms of quantum confinement effects, showing that neither thermal expansion nor electron–phonon coupling effects may be disregarded when analyzing the temperature dependence of the optoelectronic properties of perovskite lead halide nanocrystals.

Impact of Tb^{4+} and morphology on the thermal evolution of Tb-doped TiO_2 nanostructured hollow spheres and nanoparticles

Colomer, MT; Rodríguez, E; Morán-Pedroso, M; Vattier, F; de Andrés, A

Journal of Alloys and Compounds, **853** (2021) 156973

Febrero 2021 | **DOI:** 10.1016/j.jallcom.2020.156973



Tb-doped TiO_2 hollow spheres (HSs) in the range 0.0-2.0 at.% have been synthesized by the first time to the best of our knowledge. The HSs are compared with nanoparticles (NPs) to evaluate the impact of morphology on their physicochemical and photoluminescence (PL) behavior upon increasing calcination temperature. After calcination at 550 ºC, the particles are anatase with a primary average size of 10.0 ± 0.2 nm for the NPs and 12.0 ± 0.2 nm for those that form the micron sized hollow spheres of 1.8 ± 0.2 μm diameter and ca. 64 nm shell thickness. The temperature of the anataseerutile transition is found to be strongly dependent on the presence of Tb as well as on morphology. Contrarily to the usual stabilization of anatase when doping with trivalent rare-earth ions, the transition temperature is reduced when doping with Tb. The rutile phase is further favored for the HSs compared to the NPs probably related to the low density of the HSs and/or a more efficient packing density and/or a bigger crystal size of the nanoparticles that form those spheres with respect to the packing and the size of the NPs and/or the crystal size of the nanoparticles of the HSs with respect to the size of the NPs. Only a slight unit-cell volume increase for the anatase structure is observed upon Tb doping, in both the NPs and in the HSs, contrary to the expected increment due to the larger ionic radius of Tb^{3+} compared to Ti^{4+}. In addition, the intensity of the characteristic f-f Tb^{3+} emission bands is extremely weak both in the anatase and rutile phases. The transition is accompanied with the emergence of an infrared emission band centered at 810 nm related to the formation of defects during the structural transformation providing deep levels in the gap that partly quench the f-f emissions in the rutile phase. The results are consistent with the presence of Tb in both +3 and +4 valence states. XPS measurements confirmed the presence of Tb^{3+} as well as of Tb^{4+} in both HSs and NPs. The large fraction of Tb^{4+} present in the samples originates the weak f-f emission intensity, an only slight increase of the cell parameters and the destabilization of the anatase phase.

Self-preserving ice layers on CO_2 clathrate particles: Implications for Enceladus, Pluto, and similar ocean worlds

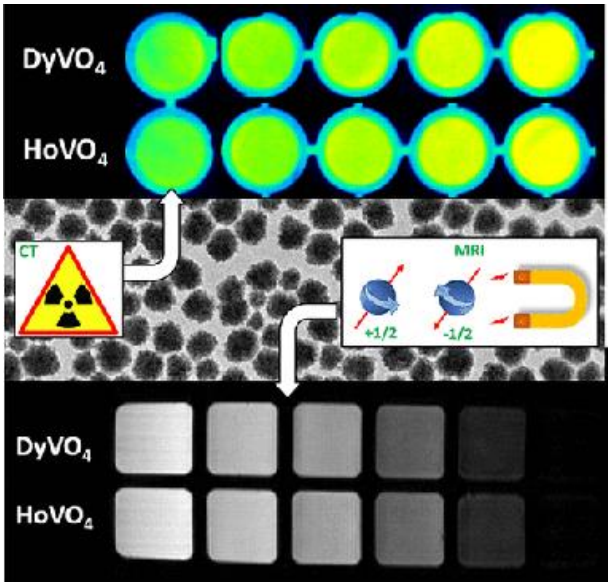
Bostrom, M; Esteso, V; Fiedler, J; Brevik, I; Buhmann, SY; Persson, C; Carretero-Palacios, S; Parsons, DF; Corkey, RW

Astronomy & Astrophysics, **650** (2021) A54
Junio 2021 | **DOI:** 10.1051/0004-6361/202040181

Context. Gas hydrates can be stabilised outside their window of thermodynamic stability by the formation of an ice layer – a phenomenon termed self-preservation. This can lead to a positive buoyancy for clathrate particles containing CO_2 that would otherwise sink in the oceans of Enceladus, Pluto, and similar oceanic worlds.
Aims. Here we investigate the implications of Lifshitz forces and low occupancy surface regions on type I clathrate structures for their self-preservation through ice layer formation, presenting a plausible model based on multi-layer interactions through dispersion forces.
Methods. We used optical data and theoretical models for the dielectric response for water, ice, and gas hydrates with a different occupancy. Taking this together with the thermodynamic Lifshitz free energy, we modelled the energy minima essential for the formation of ice layers at the interface between gas hydrate and liquid water.
Results. We predict the growth of an ice layer between 0.01 and 0.2 μm thick on CO, CH_4, and CO_2 hydrate surfaces, depending on the presence of surface regions depleted in gas molecules. Effective hydrate particle density is estimated, delimiting a range of particle size and compositions that would be buoyant in different oceans. Over geological time, the deposition of floating hydrate particles could result in the accumulation of kilometre-thick gas hydrate layers above liquid water reservoirs and below the water ice crusts of their respective ocean worlds. On Enceladus, the destabilisation of near-surface hydrate deposits could lead to increased gas pressures that both drive plumes and entrain stabilised hydrate particles. Furthermore, on ocean worlds, such as Enceladus and particularly Pluto, the accumulation of thick CO_2 or mixed gas hydrate deposits could insulate its ocean against freezing. In preventing freezing of liquid water reservoirs in ocean worlds, the presence of CO_2-containing hydrate layers could enhance the habitability of ocean worlds in our Solar System and on the exoplanets and exomoons beyond.

Dysprosium and Holmium Vanadate Nanoprobes as High-Performance Contrast Agents for High-Field Magnetic Resonance and Computed Tomography Imaging

Gómez-González, E; Núñez, NO; Caro, C; García-Martín, ML; Fernández-Afonso, Y; de la Fuente, JM; Balcerzyk, M; Ocaña, M
Inorganic Chemistry, **60** (2021) 152-160
Enero 2021 | **DOI:** 10.1021/acs.inorgchem.0c02601

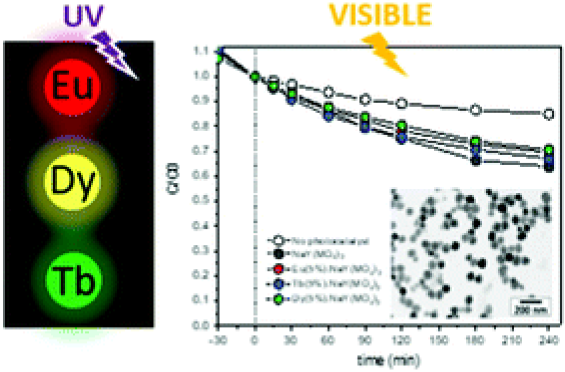


We describe a wet chemical method for the synthesis of uniform and well-dispersed dysprosium vanadate ($DyVO_4$) and holmium vanadate ($HoVO_4$) nanoparticles with an almost spherical shape and a mean size of ~60 nm and their functionalization with poly(acrylic acid). The transverse magnetic relaxivity of both systems at 9.4 T is analyzed on the basis of magnetic susceptibility and magnetization measurements in order to evaluate their potential for application as high-field MRI contrast agents. In addition, the X-ray attenuation properties of these systems are also studied to determine their capabilities as computed tomography contrast agent. Finally, the colloidal stability under physiological pH conditions and the cytotoxicity of the functionalized NPs are also addressed to assess their suitability for bioimaging applications.

$NaY(MoO_4)_2$-based nanoparticles: synthesis, luminescence and photocatalytic properties

Núñez, NO; Gómez-González, E; Calderón-Olvera, RM; Becerro, AI; Colón, G; Ocaña, M
Dalton Transactions, **50** (2021) 16539-16547
Noviembre 2021 | **DOI:** 10.1039/d1dt02365a

We report on a novel synthesis method, which produces $NaY(MoO_4)_2$ nanoparticles having an almost spherical shape and hydrophilic character. The procedure is also suitable for the preparation of $NaY(MoO_4)_2$-based nanophosphors by doping this host with lanthanide cations (Eu^{3+}, Tb^{3+} and Dy^{3+}), which, under UV illumination, exhibit intense luminescence whose color is determined by the selected doping cation (red for Eu^{3+}, green for Tb^{3+} and yellow for Dy^{3+}).

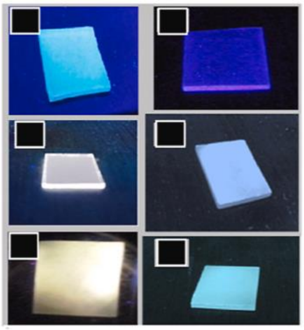


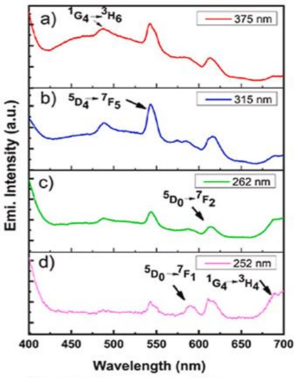
The effects of the cations doping level on the luminescent properties are analyzed in terms of emission intensities and luminescent lifetime, to find the optimum phosphors. Finally, the performance of these nanophosphors and that of the undoped system for the photocatalytic degradation of rhodamine B, used as a model compound, is also analyzed.

White, blue, violet, and other colors from Tm^{3+}/Tb^{3+}/Eu^{3+} co-doped polymorph $SrAl_2O_4$ films, deposited by ultrasonic spray pyrolysis technique

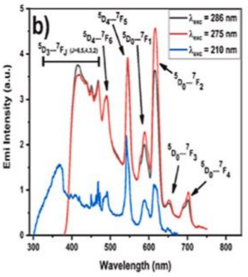
Calderón-Olvera, RM; García-Hipólito, M; Álvarez-Fregoso, O; Álvarez-Pérez, MA; Báez-Rodríguez, A; Ramos-Brito, F; García-Velasco, AC; Falcony, C

Optical Materials, **122** (2021) 111737
Diciembre 2021 | **DOI:** 10.1016/j.optmat.2021.111737





PL emission spectra of the $SrAl_2O_4$:Tb^{3+}, Eu^{3+}, Tm^{3+} film, excited under λ_{exc} = 375 (a), 315 (b), 262 (c), 252 (d), respectively



Emission spectra of the $SrAl_2O_4$:Tb^{3+} (8 at. %), Eu^{3+} (3 at. %) film for the λ_{exc} = 286, 275 and 210 nm.

$SrAl_2O_4$: Tm^{3+}, $SrAl_2O_4$: (Tb^{3+}; Eu^{3+}) and $SrAl_2O_4$: (Tb^{3+}; Eu^{3+}; Tm^{3+}) films were deposited by ultrasonic spray pyrolysis (USP) method at 550 °C and subsequently heat-treated at 800 °C. XRD characterization showed a monoclinic/hexagonal polymorph phase of these films with orthorhombic $Sr_4Al_{14}O_{25}$ as secondary phase. The incorporation of Tm^{3+} ions in strontium aluminate host lattice generated emissions of blue color for photoluminescence and violet color for cathodoluminescence. The violet emission was associated to the electronic transition from $^1I_6$ energy level of Tm^{3+}. Photoluminescence of the $SrAl_2O_4$: (Tb^{3+}; Eu^{3+}) films resulted in two different colors, white emission was observed when excited with 210 nm and bluish-white emission was achieved by exciting with 275, and 286 nm. When three dopant ions (Tm^{3+}; Tb^{3+}; Eu^{3+}) were incorporated inside strontium aluminate host lattice, it was observed (exciting under 252 nm) white photoluminescence emission (x = 0.3377, y = 0.3294); for excitation wavelengths (λ_{exc}) = 262, 315 and 375 nm, emissions in different shades of blue-green were achieved. Quantum efficiencies between 48 and 57% were obtained.

Si sputtering yield amplification: a study of the collisions cascade and species in the sputtering plasma

Cruz, J; Sanginés, S; Soto-Valle; Muhl; Sierra, I; De Lucio-Morales, O; Mitrani, A; Calderón-Olvera, RM; Mendoza-Pérez, R; Machorro-Mejía, R
Journal of Physics D-Applied Physics, **54** (2021) 375201
Septiembre 2021 | **DOI:** 10.1088/1361-6463/ac0c4e

The sputtering yield amplification (SYA) is a phenomenon based on doping a sputtering target with atoms of higher atomic mass. This doping changes the depth and the direction of the collision cascade in the target surface promoting a higher ejection of target atoms. In this work, we present a new way of generating the SYA phenomenon without the need of expensive and complex deposition systems. This was accomplished by increasing the working pressure and adding small pieces of W, as dopant element, on the racetrack of a Si target. The physical phenomena necessary to promote the SYA, for our experimental parameters, were analysed in two different deposition chambers and two sizes of sputtering targets. Based on the collisions in the gas phase, a calculation on the number of W atoms returning to the racetrack area was made, considering the number of atoms deposited on the thin films, to determine their effect on the cascade of collisions. In addition, calculations with the simulation of metal transport code were developed to determine the location on the racetrack zone the returning atoms were redeposited. By using reference samples placed on the racetrack of the Si target, we found that the percentage of SYA depends on the number of dopant atoms redeposited as well as the depth distribution these atoms had in the racetrack surface.

Persistent luminescent nanoparticles: Challenges and opportunities for a shimmering future

Castaing, V.; Arroyo, E.; Becerro, A.I.; Ocaña, M.; Lozano, G.; Míguez, H.
Journal of Applied Physics, **130** (2021) 080902
Agosto 2021 | **DOI:** 10.1063/5.0053283

Persistent phosphors are luminescent sources based on crystalline materials doped with rare-earth or transition metal cations able to produce light after the excitation source vanishes. Although known for centuries, these materials gained renewed interest after the discovery of Eu^{2+},RE^{3+} co-doped aluminates and silicates in the late 1990s due to their unprecedented afterglow properties. In contrast, persistent nanophosphors have emerged only recently as a nanoscale alternative to their bulk counterparts, offering exciting opportunities of particular relevance for *in vivo* imaging, optical data storage, or unconventional light generation. However, taking advantage of the avenues opened by nanoscience demands developing new synthetic strategies that allow precise control of the morphology, surface, and defect chemistry of the nanomaterials, along with a profound understanding of the physical mechanisms occurring in the nanoscale. Besides, advanced physicochemical characterization is required to assess persistent luminescence in a quantitative manner, which allows strict comparison among different persistent nanophosphors, aiming to propel their applicability. Herein, we revisit the main phenomena that determine the emission properties of persistent nanoparticles, discuss the most promising preparation and characterization protocols, highlight recent achievements, and elaborate on the challenges ahead.

Nanophotonics for current and future white light-emitting devices

Galisteo-López, JF; Lozano, G
Journal of Applied Physics, **130** (2021) 200901
Noviembre 2021 | **DOI:** 10.1063/5.0065825

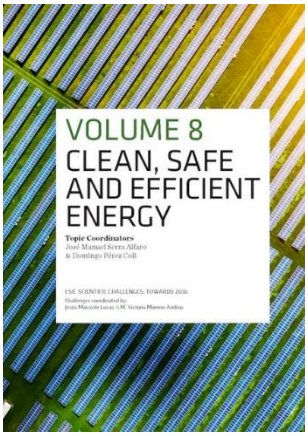
Photonic nanostructures have proven useful to enhance the performance of a wide variety of materials and devices for sensing, catalysis, light harvesting, or light conversion. Herein, we discuss the role of nanophotonics in current and next-generation designs of white light-emitting diodes (LEDs). We discuss recent developments on luminescent materials designed as alternatives to rare earth-doped inorganic microcrystals, i.e., phosphors, for color conversion in LEDs, which has opened the door to the integration of resonant photonic architectures. Nanophotonics enables the devised light-matter interaction with luminescent materials in the nanoscale, which allows providing emitting devices with both enhanced performance and novel functionalities to tackle technological challenges.

Toward Commercialization of Stable Devices: An Overview on Encapsulation of Hybrid Organic-Inorganic Perovskite Solar Cells

Aranda, CA; Caliò, L; Salado, M
Crystals, **11** (2021) 519
Mayo 2021 | **DOI:** 10.3390/cryst11050519

Perovskite solar cells (PSCs) represent a promising technology for energy harvesting due to high power conversion efficiencies up to 26%, easy manufacturing, and convenient deposition techniques, leading to added advantages over other contemporary competitors. In order to promote this technology toward commercialization though, stability issues need to be addressed. Lately, many researchers have explored several techniques to improve the stability of the environmentally-sensitive perovskite solar devices. Challenges posed by environmental factors like moisture, oxygen, temperature, and UV-light exposure, could be overcome by device encapsulation. This review focuses the attention on the different materials, methods, and requirements for suitable encapsulated perovskite solar cells. A depth analysis on the current stability tests is also included, since accurate and reliable testing conditions are needed in order to reduce mismatching involved in reporting the efficiencies of PSC.

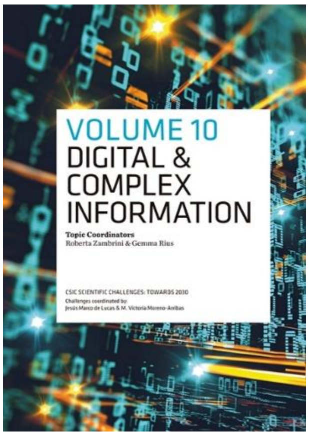
LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS



Clean, safe and efficient energy Volumen 8
CSIC Scientific Challenges : Towards 2030
Consejo Superior de Investigaciones Científicas
2021
ISBN Vol. 8: 978-84-00-10752-9

Challenge 1: Renewable Energy Production
Hernán Míguez, Mariano Campoy Quiles, Luis A. Pérez-Maqueda, Agustín R. González-Elipe, Xavier Granados, Víctor Vilarrasa, Xavier Odradors, Teresa Puig, José María Ripalda, Mariona Coll, Josep Fontcuberta, Alejandro Goñi, Mónica Lira Cantú, María Bernechea, Pedro Atiénzar, Mauricio Calvo
páginas 15-43

Challengue 3: Energy Efficiency and Harvesting
Marisol Martín González, José Francisco Fernández, José Antonio Tenorio, Agustín R. González-Elipe, Gabriel Lozano, Ana I. Borrás, Olga Caballero, Luis Fonseca, Mariano Campoy, Neus Sabaté
páginas 73-95



Digital & Complex Information Volumen 10
CSIC Scientific Challenges : Towards 2030
Consejo Superior de Investigaciones Científicas
2021
ISBN: 978-84-00-10756-7

Challengue 2: Advanced Photonics
Miguel Cornelles, Javier Aizpurua, Juan Diego Aina, Gaspar Armelles, Alfonso Cebollada, Gabriel Cristóbal, Alicia de Andrés, Rebeca de Nalda, Carlos Domínguez, Ingo Fischer, Antonio García, Vicenzo Giannini, Alejandro González, Gervasi Herranz, Laura Lechuga, Bernabé Linares, Cefe López, Luis Martín-Moreno, Hernán Míguez, Agustin Mihi, Aurora Nogales, Luis Pesquera, Pablo Aitor, Ana Quirce, Esther Rebollar, José A. Sánchez, Teresa Serrano, Javier Solis, Tobías Stauer, Angel Valle, Roberta Zambrini

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

2021 Spring Meeting of the European Materials Research Society (E-MRS)
31 mayo – 3 junio [Online]

Transparent multi-colored up-converting oxyfluoride-based nanophosphor thin films
E. Arroyo, T. Tuyen Ngo, E. Cabello, H. Míguez, M. Ocaña, G. Lozano, A.I. Becerro
Póster

Applied Light-Matter Interactions in Perovskite Semiconductors - ALMI-PS2021
2 – 4 junio [Online]

Relevance of Optical Design for Optolectronic Devices based on Lead Halide Perovskites
H. Míguez
Conferencia Invitada

Template-assisted synthesis of optically active $CsPbI_3$ Q-dots
C. Romero Pérez
Comunicación oral

The Role of the Atmosphere on the Photophysics of Ligand-Free Lead-Halide Perovskite Nanocrystals
M. Morán-Pedroso, A. Rubino, M.E. Calvo, J.P. Espinós, J.F. Galisteo-López, H. Míguez
Póster

nanoGe Fall Meeting
18 – 22 octubre [Online]

Ligand-free MAPbI3 quantum dots solar cells
A. Rubino, L. Caliò, C. Romero Pérez, H. Míguez, M. Calvo
Comunicación oral

Tailoring the photophysical properties of a molecular absorber coupled to an optical cavity in strong light-matter regime
V. Esteso, L. Caliò, H. Espinós, G. Lavarda, T. Torres, J. Feist, F.J. García-Vidal, G. Bottari, H. Míguez
Comunicación oral

Role of the Atmosphere on the Photophysics of Ligand-Free Lead Halide Perovskite nanocrystals
J. Galisteo
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

4th Spanish Conference on Biomedical Applications of Nanomaterials (SBAN)
2 – 4 junio [Online, España]

Lanthanide vanadate based nanoparticles as contrast agents for high-field magnetic
resonance imaging and X-ray computed tomography
E. Gómez-González, N.O. Núñez, C. Caro, M.L. García-Martín, Y. Fernández-Alonso, J.M. De la Fuente, M. Balcerzyk, M. Ocaña
Póster

1st Meeting Excellence Network – Red Mode Fotovoltaica
30 junio [Online, España]

Light Harvesting Properties of a Subphthalocyanine Solar Absorber Coupled to an Optical Cavity
V. Esteso, L. Caliò, H. Espinós, G. Lavarda, T. Torres, J. Feist, F.J. García-Vidal, G. Bottari, H. Míguez
Comunicación oral

Conferencia Española de Nanofotónica CEN2021
20-22 septiembre [Online, España]

Nanophosphor Photonics for Efficient Light Conversion
G. Lozano
Conferencia invitada

Uniform $ZnGa_2O_4$:Cr^{3+} bioprobes with long persistent luminescence in the first biological window
E. Arroyo, M. Ocaña, A.I. Becerro
Comunicación oral

Multifuntional up-converting oxyfluoride-based nanophosphor films
T. Tuyen Ngo, E. Cabello-Olmo, E. Arroyo, A.I. Becerro, M. Ocaña, G. Lozano, H. Míguez
Comunicación oral

Modified Optical Properties of a Subphthalocyanine Solar Absorber Embedded into a Fabry-Pérot Cavity
V. Esteso, L. Caliò, H. Espinós, G. Lavarda, T. Torres, J. Feist, F.J. García-Vidal, G. Bottari, H. Míguez
Comunicación oral

Monolithic patterned nanophosphors: light emission control from periodic surface textures
E. Cabello-Olmo, P. Molet, A. Mihi, G. Lozano, H. Míguez
Comunicación oral

The Role of the Atmosphere on the Photophysics of Ligand-Free Lead-Halide Perovskite Nanocrystals
M. Morán-Pedroso, A. Rubino, M.E. Calvo, J.P. Espinós, J.F. Galisteo-López, H. Míguez
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Study of the Casimir-Lifshitz force in the plane parallel geometry**
Autor:	Victoria Esteso Carrizo
Directores:	Sol Carretero Palacios y Hernán R. Míguez García
Centro:	Universidad de Sevilla
Fecha Defensa:	24 de mayo de 2021

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Fabricación y caracterización de celdas solares de sulfuro de plata y bismuto**
Autor:	José Bermejo Vizcaíno
Tutores:	Laura Caliò, Hernán R. Míguez García
Grado:	Trabajo Fin de Máster
Fecha Defensa:	1 marzo 2021

Título: **Optimización de recubrimientos de perovskita para dispositivos optoelectrónicos**
Autor: Raúl Mulero Guerrero
Tutores: Laura Caliò, Hernán R. Míguez García
Grado: Trabajo Fin de Máster
Fecha Defensa: 1 marzo 2021

Título: **Síntesis de nanopartículas de $NaDy(WO_4)_2$ con aplicaciones en diagnóstico por imagen**
Autor: José Aceituno Cruz
Tutores: Nuria O. Núñez Álvarez
Grado: Trabajo Fin de Grado
Fecha Defensa: 23 julio 2021

DOCENCIA / TEACHING

OSA Student Chapter at Yachay Tech (OSA-YT)
24 abril [Online, Ecuador]

Nanophosphor Photonics for Efficient Light Conversion
Gabriel Lozano
Conferencia Invitada

Apolo Summer School SEMINARIO
14 – 16 julio [Online, España]

Light Harvesting Properties of a Subphthalocyanine Solar Absorber Coupled to an Optical Cavity
Gabriel Lozano
Conferencia Invitada

Seminarios del Departamento de Química Inorgánica, Analítica y Química Física. Facultad de Ciencias Exactas y Naturales
14 junio [Universidad de Buenos Aires, Argentina]

Perovskitas ABX3 desde el confinamiento: historia de un encuentro productivo entre quantum dots y materiales porosos
Mauricio Calvo
Conferencia Invitada

Investigadores de esta unidad participan en el Máster Interuniversitario "Láser, Plasma y Tecnología de Superficies" (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Potenciostato y sistema electroquímico
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- Liofilizador de altas prestaciones Epsilon 2-4 (CHRIST)
- Estufa de desecación de 90 litros (RAYPA)
- Cámara Incubadora Opaq + Orbital Maxi (OVAN)
- pH & Ion-metro GLP 22+ (CRISON A)
- Espectrómetro visible-UV CARY-100. Medidas de coeficiente de absorción con luz normal y polarizada.
- Fluorímetro espectroscópico (HORYBA Jobin Yvon Fluorolog) con accesorio para la determinación de tiempos de vida. Microscopio de fluorescencia (HORYBA Jobin Yvon sigle photon controller: FluoroHub).
- Sistema de medida de porosidades en capas delgadas.
- Vis-NIR FTIR espectrofotómetro Bruker GmbH Fuente de excitación continua normal y angular. Specular Reflectance Attached Microscope.
- Tunable Supercontinuum White Laser Source. Fianium LTD 4W total output 400nm – 2400nm range Acousto-Optic Tunable Filter
- Perfilómetro mecánico DektakXT en su versión automática (platina XY motorizada y giro de 360° motorizado) y que incluye soporte de muestras cerámico para muestras flexibles.
- Fluorímetro Edinburgh FLS1000 con accesorios (esfera integradora y criostato).

UNIDAD EXTERNA DE INVESTIGACIÓN: FÍSICA DE MATERIALES

EXTERNAL UNIT: PHYSICS OF MATERIALS

PERSONAL / PERSONNEL

Catedráticos	Dra. Clara F. Conde Amiano
	Dr. Alberto Criado Vega
	Dr. Luis Esquivias Fedriani
	Dr. Victorino Franco García
	Dr. Antonio Muñoz Bernabé
	Dr. Javier S. Blázquez Gámez
Profesores Titulares	Dra. Josefa María Borrego Moro
	Dra. M. del Carmen Gallardo Cruz
	Dra. Ángela Gallardo López
	Dr. Felipe Gutiérrez Mora
	Dr. Jhon Jairo Ipus Bados
	Dr. José María Martín Olalla
	Dr. Víctor Morales Flórez
	Dra. Ana Morales Rodríguez
	Dr. Francisco Javier Romero Landa
Profesores Contratados Doctores	Dr. Rafael Caballero Flores
	Dra. Rocío del Carmen Moriche Tirado
Doctores Contratados	Dr. Luis M. Moreno Ramírez
	Dra. Jia Yan Law
	Dra. Bibi Malmal Moshtaghion Entezari
Personal Investigador en Formación	Gdo. Álvaro Díaz García
	Gda. Carmen Muñoz Ferreiro
	Gdo. Pedro Rivero Antúnez
	Gdo. Antonio Vidal Crespo
Investigadores Honorarios	Dr. Jaime del Cerro González
	Dr. Arturo Domínguez Rodríguez
	Dr. Justo Jiménez Fernández

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Abordando las Limitaciones de Materiales Magnetocalóricos para su Implementación en Aplicaciones Energéticamente Eficientes	
Código/Code:	PID2019-105720RB-I00
Periodo/Period:	01-06-2020 / 31-05-2024
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Investigador responsable/Research head:	Victorino Franco García
Componentes/Research group:	Josefa María Borrego Moro, Jhon Jairo Ipus Bados

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales	
Código/Code:	PGC2018-101377-B-I00
Periodo/Period:	01-01-2019 / 31-12-2022
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Ángela Gallardo López, Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora

Refuerzo Intragranular de Cerámicas con Fases de Baja Dimensionalidad

MINISTERIO
DE CIENCIA
E INNOVACIÓN

AGENCIA
ESTATAL DE
INVESTIGACIÓN

Código/Code:	PGC2018-094952-B-I00
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	96.800 €
Investigador responsable/Research head:	Víctor Morales Flórez
Componentes/Research group:	Luis Esquivias Fedriani, Francisco de Paula Jiménez Morales, Florentino Sánchez Bajo

A la Búsqueda de Nuevos Cerámicos Ultraduros a Base de Boro para Aplicaciones Estructurales en la Nueva Generación de Aviones y uso Seguro y Eficiente de la Energía

MINISTERIO
DE CIENCIA
E INNOVACIÓN

AGENCIA
ESTATAL DE
INVESTIGACIÓN

Código/Code:	PID2019-103847RJ-I00
Periodo/Period:	01-10-2020 / 30-09-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Investigador responsable/Research head:	Bibi Malmal Moshtaghion Entezari
Componentes/Research group:	Francisco Luis Cumbreras Hernández, Arturo Domínguez Rodríguez, Diego Gómez García

Influencia de excitaciones múltiples sobre transiciones de fase termomagnéticas para aplicaciones energéticas

Código/Code:	US-1260179
Periodo/Period:	01-02-2020 / 30-04-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Victorino Franco García
Componentes/Research group:	Javier Sebastián Blázquez Gámez, Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon Jairo Ipus Bados

Transiciones de fase termo-magnéticas para un uso eficiente de la energía y de los recursos

Código/Code:	P18-RT-746
Periodo/Period:	01-01-2020 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Victorino Franco García
Componentes/Research group:	Javier Sebastián Blázquez Gámez, Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon Jairo Ipus Bados

Fabricación y caracterización microestructural y mecánica de cerámicas de carburo de boro con estequiometría controlada (BORONCARB)	
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P18-RTJ-1972
Periodo/Period:	01-11-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Francisco Javier Martínez Vázquez, Bibi Malmal Moshtaghion
Componentes/Research group:	Francisco Luis Cumbreras Hernández

Fundamentos y potencialidades del refuerzo de compuestos de matriz cerámica con alótropos de carbono (FRAC)	
Junta de Andalucía Unión Europea Fondo Europeo de Desarrollo Regional Andalucía se mueve con Europa	
Código/Code:	P20_01121
Periodo/Period:	05-10-2021 / 30-06-2023
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Víctor Morales Flórez
Componentes/Research group:	Arturo Domínguez Rodríguez, Luis María Esquivias Fedriani, Francisco de Paula Jiménez Morales, María del Carmen Lemos Fernández

Desarrollo de cerámicas avanzadas con nanomateriales 2D para su aplicación en sistemas de propulsión y frenado en la industria aeroespacial (AEROCER-2D)

Código/Code:	P20_01024
Periodo/Period:	05-10-2021 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Ángela Gallardo López
Componentes/Research group:	Felipe Gutiérrez Mora, Ana Morales Rodríguez, Antonio Muñoz Bernabé, Rosalía Poyato Galán, Rocío del Carmen Moriche Tirado

Otros Proyectos / Other Projects

Hysteresis and frequency response as limiting factors for efficient thermomagnetic energy conversion

Código/Code:	FA8655-21-1-7044
Periodo/Period:	30-09-2021 / 29-09-2024
Organismo Financiador/Financial source:	Air Force Office of Scientific Research
Investigador responsable/Research head:	Victorino Franco García
Componentes/Research group:	Jia Yan Law, Luis Miguel Moreno Ramírez

Diseño y puesta en funcionamiento de una planta piloto para el reciclado de fosfoyesos

Código/Code:	AT17_5282_USE
Periodo/Period:	01-02-2020 / 31-07-2021
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Luís María Esquivias Fedriani
Componentes/Research group:	Pedro García Haro, Alberto Gómez Barea, Víctor Morales Flórez

Optimization of Magnetocaloric Materials Towards Energy Efficient Applications

Código/Code:	W911NF1920212
Periodo/Period:	16-09-2019 / 15-09-2021
Organismo Financiador/Financial source:	US Army Research Laboratory
Investigador responsable/Research head:	Victorino Franco García
Componentes/Research group:	Javier Sebastián Blázquez Gámez, Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon Jairo Ipus Bados, Jia Yan Law, Álvaro Díaz García

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

- Colaboración con grupo de Jerôme Chevalier y Helen Reveron, INSA Lyon, Mateis. Carmen Muñoz Ferreiro (tesis en cotutela). Financiación: Ayuda de la Univ. De Lyon.
- Colaboración con grupo de Frank Kern – Andrea Gommeringer, Universitat Stuttgart, IFKB (Institut für Fertigungstechnologie keramischer Bauteile)
- Colaboración con Katalin Balazsi, Head of Thin Film Physics Department, Hungarian Academy of Sciences, Centre for Energy Research, Budapest, Hungría

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Design of Fe-containing GdTbCoAl high-entropy-metallic-glass composite microwires with tunable Curie temperatures and enhanced cooling efficiency

Yin, HBC; Law, JY; Huang, YJ; Franco, V; Shen, HX; Jiang, SD; Bao, Y; Sun, JF
Materials & Design, **206** (2021) 109824
Agosto, 2021 | **DOI:** 10.1016/j.matdes.2021.109824

Through designing the composition and processing approach, the non-equiatomic $(Gd_{36}Tb_{20}Co_{20}Al_{24})_{100-x}Fe_x$ (x = 0, 1, 2 and 3 at.%) high-entropy-metallic-glass (HE-MG) alloy microwires were successfully fabricated by melt-extraction technique. The microstructure and magnetocaloric properties of the micro wires were systematically investigated. The microwires possess tunable Curie temperatures, i.e. 81- 108 K, above the typical rare-earth (RE) containing HE-MG reports. The high Curie temperatures are attributed to the designed composition.

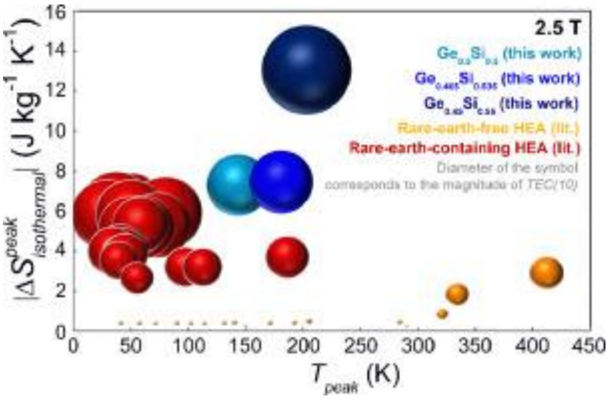
Magnetocaloric response peak values of Fe-containing GdTbCoAl alloy microwires range 7.6-8.9 J kg^{-1} K^{-1} (5 T), which are comparable to those of many outstanding RE-containing magnetocaloric HE-MGs. The characteristics of the melt-extraction method, combining with compositional effects, favor the formation of amorphous and nanocrystalline phases. The increase in the cooling efficiency for microwires with higher Fe content can be attributed to the broadening of the Curie temperature distribution induced by the composition difference between nanocrystalline phase and amorphous matrix. The designed composition and the melt-extraction processing approach for Fe-containing GdTbCoAl alloys can tune their Curie temperatures towards a temperature range of natural gas liquefaction and improve their magnetocaloric properties. This demonstrates that Fe-containing GdTbCoAl HE-MG composite microwires have great potential as high-performance magnetic refrigerants.

Increased magnetocaloric response of FeMnNiGeSi high-entropy alloys

Law, JY; Diaz-Garcia, A; Moreno-Ramirez, LM; Franco, V

Acta Materialia, **212** (2021) 116931

Junio, 2021 | **DOI:** 10.1016/j.actamat.2021.116931



The search for high-entropy alloys (HEAs), a new class of materials, with high magnetocaloric performance can address the open question about their potential in functional applications. HEAs exist in a vast compositional space but magnetocaloric HEAs typically exhibit modest magnetocaloric response as they undergo second-order magnetic phase transitions. In this work, through a property-directed search approach, FeMnNiGeSi HEAs with varying Ge/Si ratio are found in the large HEA space to exhibit magnetostructural first-order phase transition. Isothermal entropy change as large as 13 J kg^{-1}K^{-1} (for 2.5T) is achieved, which is the largest reported to date for magnetocaloric HEAs. When compared to conventional high-performance magnetocaloric materials, our work is observed to be comparable to many of the conventional systems, closing the gap between magnetocaloric HEAs versus conventional magnetocaloric materials for the first time. This opens a new path for the search of functional materials with the optimal mechanical properties of HEAs.

Pushing the limits of magnetocaloric high-entropy alloys

Law, JY; Franco, V

APL Materials, **9** (2021) 080702

Agosto, 2021 | **DOI:** 10.1063/5.0058388

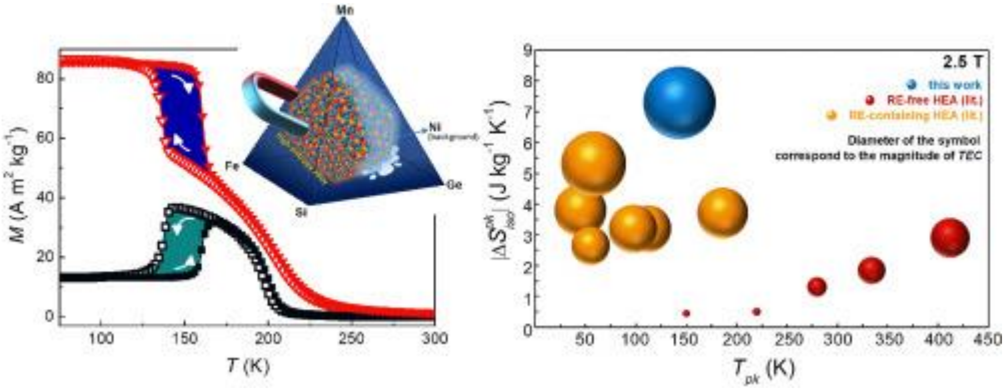
High-entropy alloys (HEAs) have become a topic of high research interest due to the excellent mechanical properties that can be found in this new type of materials. However, their functional properties are usually modest when compared to conventional materials. The discovery of high-entropy alloys with an optimal combination of mechanical and functional properties would be a leap forward in the reliability of devices that use them as functional elements. This Research Update focuses on magnetocaloric HEAs, showing that a directed search strategy allows us to improve their performance in a significant way, closing the pre-existing gap between magnetocaloric HEAs and high-performance magnetocaloric materials. Further challenges that remain in this line of research are highlighted.

MnFeNiGeSi high-entropy alloy with large magnetocaloric effect

Law, JY; Moreno-Ramirez, LM; Diaz-Garcia, A; Martin-Cid, A; Kobayashi, S; Kawaguchi, S; Nakamura, T; Franco, V

Journal of Alloys and Compounds, **855** (2021) 157424

Febrero, 2021 | **DOI:** 10.1016/j.jallcom.2020.157424



The magnetocaloric effect of high-entropy alloys (HEAs) due to a second-order phase transition exhibits limited response. A significant improvement is obtained in $Mn_{22.3}Fe_{22.2}Ni_{22.2}Ge_{16.65}Si_{16.65}$ HEA as it exhibits a magneto-structural first-order phase transition. Its isothermal entropy change of 7.3 J kg^{-1}K^{-1} (for 2.5 T) is the largest reported to date for rare earth-free HEAs. The alloy composition was found by a directed search that starts from a medium entropy alloy with the desired transformation combined with isostructural substitutions. Our findings demonstrate the existence of HEA with magneto-structural phase transition and that HEAs have promising potential as high-performance magnetocaloric materials.

Characterization of thermal hysteresis in magnetocaloric NiMnIn Heusler alloys by Temperature First Order Reversal Curves (TFORC)

Diaz-Garcia, A; Moreno-Ramirez, LM; Law, JY; Albertini, F; Frabbrici, S; Franco, V
Journal of Alloys and Compounds, **867** (2021) 159184
Junio, 2021 | **DOI:** 10.1016/j.jallcom.2021.159184

The temperature variant of the First-Order Reversal Curves (TFORC) technique has been applied to study the thermal hysteresis of two magnetocaloric Heusler alloys with slightly different compositions that are close to the stoichiometry $Ni_{50}Mn_{34}In_{16}$. The similarity of the compositions causes the samples to have similar Curie temperature but different martensitic transition temperature and thermal hysteresis, leading to different features when analyzing the TFORC distributions due to the different relative distance between the two transitions. The asymmetry of the martensitic transformation upon cooling and heating has relevant effects on the TFORC distributions. The experimental results have been compared to the predictions of a model of the transformation, which allows to separate the contributions of the magneto-structural transformation and the thermomagnetic behavior of both martensitic and austenitic phases, allowing to determine the origin of the different features of the distributions.

Deconvolution of overlapping first and second order phase transitions in a NiMnIn Heusler alloy using the scaling laws of the magnetocaloric effect

Diaz-Garca, A; Law, JY; Moreno-Ramirez, LM; Giri, AK; Franco, V
Journal of Alloys and Compounds, **871** (2021) 159621
Agosto, 2021 | **DOI:** 10.1016/j.jallcom.2021.159621

Magnetocaloric materials with a first order phase transition may exhibit additional thermomagnetic phase transitions in the temperature range of interest due to the second-order magnetic phase transitions of the phases. In the case of Heusler alloys exhibiting magneto-structural transformation (martensitic type), this first-order phase transition is usually accompanied by a temperature dependence of magnetization of the different magnetic structures. The temperature of the martensitic transition in this type of materials can be tuned by altering the composition, making possible a close proximity between this transition and the Curie transitions (second-order type) of the structures. This can result in a tight overlap between the two types of magnetocaloric effects, usually of different signs. In this work, the magnetocaloric response of overlapping first and second-order phase transitions of a $Ni_{48.1}Mn_{36.5}In_{15.4}$ Heusler alloy has been deconvoluted by applying the scaling laws of the magneto caloric effect. This deconvolution allows the prediction of the magnetocaloric response of the pure first order phase transition, helping to gauge if it is worth synthesizing other alloys of the same family for which the Curie transition would be much more distant. The deconvoluted signal confirms that the anomalous experimental decrease of ΔS_{iso} with increasing the field above ~ 3 T is due to the competing contribution of the overlapping second-order phase transition.

Thermo-magnetic characterization of phase transitions in a Ni-Mn-In metamagnetic shape memory alloy

Romero, FJ; Martin-Olalla, JM; Blazquez, JS; Gallardo, MC; Soto-Parra, D; Vives, E; Planes, A
Journal of Alloys and Compounds, **887** (2021) 161395
Diciembre, 2021 | **DOI:** 10.1016/j.jallcom.2021.161395

The partially overlapped ferroelastic/martensitic and para-ferromagnetic phase transitions of a $Ni_{50.53}Mn_{33.65}In_{15.82}$ metamagnetic shape memory alloy have been studied from calorimetric, magnetic and acoustic emission measurement. We have taken advantage of the existence of thermal hysteresis of the first order ferroelastic/martensitic phase transition (~2.5 K) to discriminate the latent heat contribution $\Delta H_t = 7.21(15)kJkg^{-1}$ and the specific heat contribution $\Delta H_c = 216(1)Jkg^{-1}$ to the total excess enthalpy of the phase transition. The specific heat was found to follow a step-like behavior at this phase transition. The intermittent dynamics of the ferroelastic/martensitic transition has been characterized as a series of avalaches detected both from acoustic emission and calorimetric measurements. The energy distribution of these avalanche events was found to follow a power law with a characteristic energy exponent $\varepsilon \cong 2$ which is in agreement with the expected value for a system undergoing a symmetry change from cubic to monoclinic. Finally, the critical behavior of the para-ferromagnetic austenite phase transition that takes place at ~311 K has been studied from the behavior of the specific heat. A critical exponent $\alpha \cong 0.09$ has been obtained, which has been shown to be in agreement with previous values reported for Ni-Mn-Ga alloys but different from the critical divergence reported for pure Ni.

Comparative study of structural and magnetic properties of ribbon and bulk $Ni_{55}Fe_{19}Ga_{26}$ Heusler alloy

Manchon-Gordon, AF; Ipus, JJ; Kowalczyk, M; Blazquez, JS; Conde, CF; Svec, P; Kulik, T; Conde, A
Journal of Alloys and Compounds, **889** (2021) 161819
Diciembre, 2021 | **DOI:** 10.1016/j.jallcom.2021.161819

The influence of the fabrication method on the magnetostructural properties of a $Ni_{55}Fe_{19}Ga_{26}$ Heusler alloy, obtained both as a ribbon, by melt-spinning, and as a pellet, by arc-melting, has been analyzed. It has been found that, while the arc-melting technique leads to the precipitation of the gamma phase and to a non-modulated martensite structure, the alloy prepared by melt-spinning presents a fully 14M modulated martensitic structure at room temperature. The tetragonal non-modulated martensite in the arc-melted bulk sample transforms into the 14M structure after a long thermal treatment (at 1073 K for 24 h) and subsequent quenching. Characteristic temperatures of the martensitic transformation are higher for melt-spun ribbons than for bulk sample, due to the precipitation of the gamma phase and consequent different martensite composition. However, while the martensitic transformation temperature is practically constant in the case of the bulk sample, it changes by similar to 150 K in the case of the ribbon sample submitted to the same thermal treatment applied to bulk samples. Finally, it was found that the martensitic transformation occurs in the paramagnetic regime of both types of samples.

Reactive SPS for sol-gel alumina samples: Structure, sintering behavior, and mechanical properties

Rivero-Antunez, P; Cano-Crespo, R; Sanchez-Bajo, F; Dominguez-Rodriguez, A; Morales-Florez, V
Journal of the European Ceramic Society, **41** (2021) 5548-5557
Septiembre, 2021 | **DOI:** 10.1016/j.jeurceramsoc.2021.04.060

This work presents a fast and direct controlled routine for the fabrication of fully dense alumina based on the reactive spark plasma sintering (reactive-SPS) of boehmite (γ-AlOOH) nano-powders obtained by the sol-gel technique. The evolution of the transition aluminas during sintering has been studied. Some boehmite powders were seeded with α-Al_2O_3 particles prior to the gelation. Boehmite seeded powders exhibited a direct transition to α-Al_2O_3 at 1070 °C, enhancing the transformation kinetics and lowering the required temperature by more than 100 °C. For comparison, other samples were prepared by previously annealing the seeded and unseeded boehmite powders. Thus, α-Al_2O_3 powders were obtained and were sintered by standard-SPS. A detailed structural and mechanical characterization is presented, comparing the hardness and indentation fracture resistance for different grain sizes and porosities. Both the reactive-SPSed samples and the standard-SPSed samples showed a high hardness (18-20 GPa), whereas the reactive-SPSed samples exhibited a lower indentation fracture resistance due to a large grain size (~10 μm). Improvements of this procedure for obtaining smaller grain size are discussed. In summary, the presented technique brings a revolutionary fast method for the fabrication of fully dense alumina, as this process reduces the time and temperature required for alumina densification.

Titanium carbonitride fabricated by spark plasma sintering: Is it a ceramic model of carbon-induced Friedel-Fleisher strengthening effect?

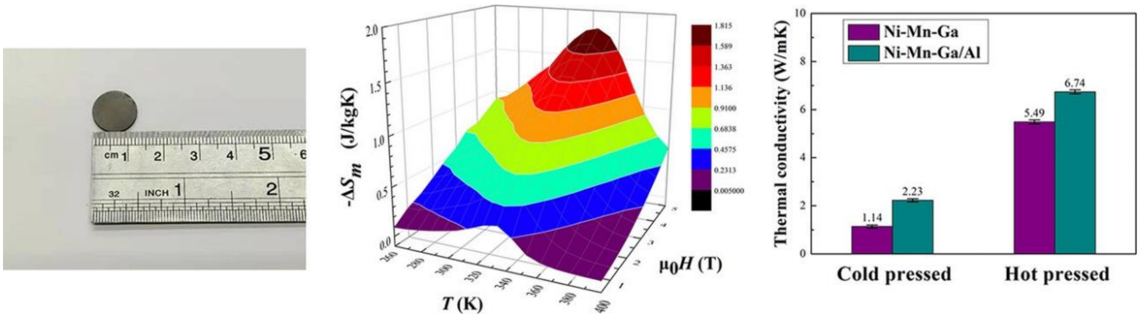
Moshtaghioun, BM; Morgado-Chávez, JM; Cumbrera, FL; Gómez-García, D
Journal of the European Ceramic Society, **41** (2021) 6275-6280
Octubre, 2021 | **DOI:** 10.1016/j.jeurceramsoc.2021.06.039

This work is devoted to the analysis of the composition, microstructure and room-temperature mechanical properties of a carbonitride ceramics: the case of Ti(C,N) ceramics fabricated by spark plasma sintering (SPS). Particular attention has been given to know whether there is a possible composition change under electric field, finding all the computed lattice parameters of SPS-ed $TiN_{1-x}C_x$ ceramics lay within tiny interval (~ $TiC_{0.43}N_{0.57}$), hence following Vegard's law. The analysis of the strengthening effect of substitutional carbon, making use of our own results and those available in literature shows that a classical $x^{1/2}$ dependence could account for experimental data. This fact reveals that a Friedel-Fleisher mode of strong localized carbon pinning points might describe the hardening effect of those impurities. However, the large scattering of experimental data and the fact that tiny deviations of stoichiometry might play a relevant role on the hardening force us to leave this as an open problem.

Structure and magnetic study of Ni-Mn-Ga/Al composite with modified magnetocaloric properties and enhanced thermal conductivity

Zhang, YC; Franco, V; Peng, HX; Qin, FX
Scripta Materialia, **201** (2021) 113956
Agosto, 2021 | **DOI:** 10.1016/j.scriptamat.2021.113956

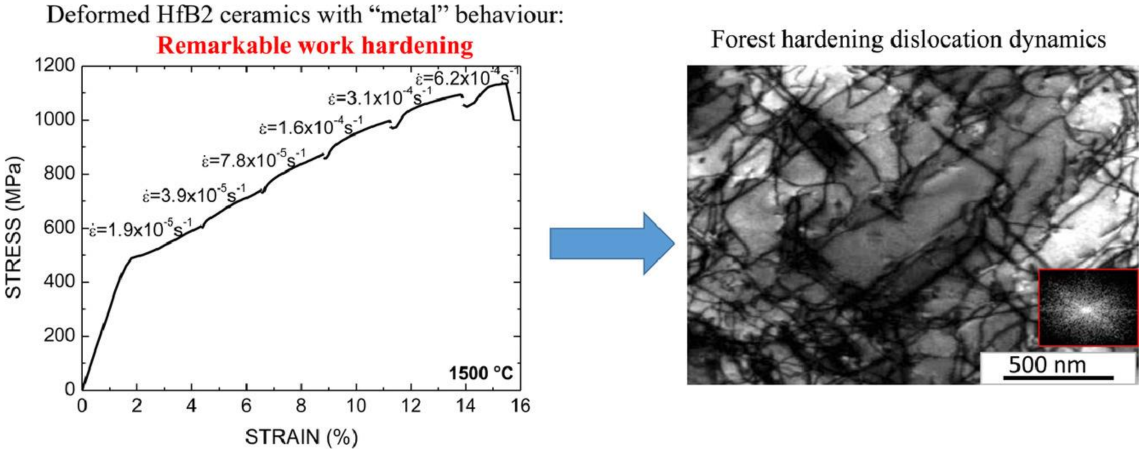
Magnetocaloric materials undergoing first order phase transition are important candidates for solid-state refrigeration but their narrow temperature range and noteworthy hysteresis are barriers to practical applications. To address these issues, we used hot pressing technique to prepare a metal matrix composite by choosing ball milled and heat-treated Ni-Mn-Ga particles together with Al powders (25 vol.%). Mi-crostructure, morphology, structural transformation,



thermal conductivity, magnetic and magnetocaloric properties of the prepared Ni-Mn-Ga/Al composite were systematically characterized and studied. Un -like the pure Ni-Mn-Ga bulk alloy counterparts, the composite exhibited broadened working temperature range of 120 K. Meanwhile, it possessed largely reduced hysteresis during thermal and magnetic cycling due to second order phase transition revealed by Banerjee criterion and field dependent exponent n. The composite also exhibited improved thermal conductivity of 6.7 W/mK due to decreased interface thermal resistance between Ni-Mn-Ga particles and Al matrix.

HfB_2 ceramic polycrystals: A low-temperature metal-like ceramic at high temperatures?

Zapata-Solvas, E; Moshtaghioun, BM; Gomez-Garcia, D; Dominguez-Rodriguez, A; Lee, WE
Scripta Materialia, **203** (2021) 114037
Octubre, 2021 | **DOI:** 10.1016/j.scriptamat.2021.114037



Hafnium diboride (HfB_2) is a highly refractory (melting above 3000 °C) ceramic with many potential applications at high temperatures. To enable its use at temperature for extended periods its high-temperature plasticity must be known. This paper examines the mechanical response at temperatures between 900 °C and 2000 °C in air and in a reducing atmosphere, interpreting the data in the frame of classical models for the plasticity of compact-packed metals at low temperatures. In particular, the Friedel law and the principle of similitude for dislocation patterning are assessed. This reveals that HfB_2 is a singular example of a ceramic material with "metal" mechanical behaviour.

Cation-driven electrical conductivity in Ta-doped orthorhombic zirconia ceramics

Moshtaghioun, BM; Laguna-Bercero, MA; Pena, JI; Gomez-Garcia, D; Dominguez-Rodriguez, A
Ceramics International, **47** (2021) 7248-7252
Marzo, 2021 | **DOI:** 10.1016/j.ceramint.2020.10.227

This paper is devoted to the study of the electrical conductivity of tantalum-doped zirconia ceramics prepared by spark plasma sintering. In this study, the temperature dependence of conductivity in as-prepared specimens and in those previously annealed in air is determined and compared. A semi-empirical model, which is based on the oxidation states of the cations, has been developed and successfully assessed. According to this, the conductivity is basically controlled by the diffusion of tetravalent zirconium cations in both cases, although the concentration of these species varies drastically with the amount of induced oxygen vacancies. This is a quite unexpected fact, since conductivity is normally controlled by anionic diffusion in zirconia ceramics. This option is forbidden here due to the presence of substitutional pentavalent cations. Therefore, conductivity values are much lower than those reported in trivalent or divalent substitutional cation doped zirconia ceramics.

Analysis of the magnetic field dependence of the isothermal entropy change of inverse magnetocaloric materials

Moreno-Ramirez, LM; Law, JY; Pramana, SS; Giri, AK; Franco, V
Results in Physics, **22** (2021) 103933
Marzo, 2021 | **DOI:** 10.1016/j.rinp.2021.103933

In this work, the magnetic field dependence of the inverse magnetocaloric (MC) effect is analyzed using a mean field approach for describing antiferromagnetic to ferromagnetic magnetoelastic transitions. The model is able to describe both second- and first-order transition through the introduction of a magnetovolume energy term. The power law dependence for the field dependence of the isothermal magnetic entropy change ($\Delta S_{iso} \propto \Delta H^n$), has an exponent n with an overshoot above 2 for first-order transitions, while it is not present for the second-order case. This is in excellent agreement with previous phenomenological observations, supporting the validity of recently proposed criterion to distinguish between first- and second-order thermomagnetic transitions. A main difference with respect to direct MC effect is that negative values of the exponent n are obtained at temperatures close to the transition. This is ascribed to the reduction of the inverse MC response due to the influence of the unavoidable ferromagnetic to paramagnetic transition at higher temperatures. The obtained features are qualitatively compared to those of $GdBaCo_2O_6$ (antiferromagnetic to ferromagnetic magnetoelastic transition), showing a good agreement between both experiments and the model. The obtained information is extrapolated to understand the behavior of the exponent n for a $Ni_{49}Mn_{36}In^{15}$ sample (magnetostructural transition).

Hysteresis, latent heat and cycling effects on the magnetocaloric response of $(NiMnSi)_{0.66}(Fe_2Ge)_{0.34}$ alloy

Moreno-Ramirez, LM; Diaz-Garcia, A; Law, JY; Giri, AK; Franco, V
Intermetallics, **131** (2021) 107083
Abril, 2021 | **DOI:** 10.1016/j.intermet.2020.107083

In this work, we further analyze the promising magnetocaloric $(NiMnSi)_{0.66}(Fe_2Ge)_{0.34}$ alloy, which presents isothermal entropy change values as large as 29 J kg^{-1} K^{-1} for 2 T at room temperature. It undergoes a magneto-structural transition accompanied by large thermal/magnetic hysteresis, which remains up to high magnetic fields (corroborated by the elaboration of an experimental magnetic phase diagram). We illustrate that this huge hysteretic behavior (i.e. different responses when magnetizing or demagnetizing) can lead to erroneous interpretation of the order of the phase transition by applying the conventional Banerjee's criterion or the recently developed field dependence analysis of the magnetocaloric effect. Additionally, the cyclability of the response is characterized, showing that after several measurements the magnetocaloric response is significantly reduced (by $\approx$ 40%) due to sample breaking. These dependences together with the large latent heat of the transition (15.0 kJ kg^{-1}) lead to relatively small values of the adiabatic temperature change for powdered samples at moderate field changes, reaching around 0.6 K for 1.75 T by direct measurement methods.

Possible half-metallic behavior of $Co_{2-x}Cr_xFeGe$ Heusler alloys: Theory and experiment

Mahat, R; Kc, S; Karki, U; Law, JY; Franco, V; Galanakis, I; Gupta, A; LeClair, P
Physical Review B, **104** (2021) 014430
Julio, 2021 | **DOI:** 10.1103/PhysRevB.104.014430

This paper reports a combined experimental and theoretical study of structural, electronic, magnetic, and mechanical properties of quaternary Heusler alloys $Co_{2-x}Cr_xFeGe$ prepared by arc-melting with Cr concentrations $0 \leq x \leq 1$. Single-phase microstructures are observed for Cr compositions from $x = 0.25$ to $x = 1$. Lower Cr concentrations are multiphased. X-ray diffraction patterns at room temperature reveal a face-centered cubic crystal structure in all single-phase samples. The low-temperature saturation magnetic moments, as determined from magnetization measurements, agree fairly well with our theoretical results and also obey the Slater-Pauling rule for half-metals, a prerequisite for half-metallicity. All alloys are observed to have high Curie temperatures that scale linearly with the saturation magnetic moments. Relatively high mechanical hardness values are also observed. First-principles calculations also predict a finite band gap in the minority spin channel of the alloys, increasing in size with increasing Cr concentration. Cr substitution brings the Fermi level toward the center of this gap while also increasing the majority spin density of states near the Fermi level. As a whole, $Co_{2-x}Cr_xFeGe$ shows great promise as a half-metal with 100% spin polarization.

The Possible Detriment of Oxygen in Creep of Alumina and Zirconia Ceramic Composites Reinforced with Graphene

Cano-Crespo, R; Rivero-Antunez, P; Gomez-Garcia, D; Moreno, R; Dominguez-Rodriguez, A
Material, **14** (2021) 984
Febrero, 2021 | **DOI:** 10.3390/ma14040984

This paper aims to give an answer to the following question: is the oxidation of graphene a critical issue for high-temperature plasticity in graphene-reinforced ceramics? To give a convincing reply, we will focus on two very different graphene-based ceramic composites: reduced graphene oxide (rGO)-reinforced alumina (α-Al_2O_3) and reduced graphene oxide (rGO)-reinforced yttria tetragonal zirconia (t-ZrO_2). The processing of the powders has been made using a colloidal route, and after that, a spark plasma sintering process was performed in order to densify the samples. Creep tests were performed at temperatures between 1200-1250 °C in an argon atmosphere. The microstructure obtained by SEM of the sintered and tested specimens was characterized quantitatively to elucidate the deformation mechanism. Raman spectroscopy was carried out to check the integrity of the graphene. The average grain size was in the order of 1 μm and the shape factor was 0.7 for all the studied materials. The integrity of the graphene was checked before and after the creep experiments. The careful analysis of the creep tests shows that graphene oxide or its reduced version are not efficient phases for creep resistance improvement in general, contrary to what is reported elsewhere. However, the results permit the suggestion of a creep improvement in nanocomposites at a very high temperature regime due to an enhanced reactivity of oxygen between carbon and alumina interfaces. In the case of zirconia, the results give us the conclusion that the oxidation of graphene is a highly detrimental issue regarding the improvement of high-temperature plasticity.

Combined kinetic and Bean-Rodbell approach for describing field-induced transitions in $LaFe_{11.6}Si_{1.4}$ alloys

Moreno-Ramirez, LM; Blazquez, JS; Radulov, IA; Skokov, KP; Gutfleisch, O; Franco, V; Conde, A
Journal of Physics D-Applied Physics, **54** (2021) 135003
Abril, 2021 | **DOI:** 10.1088/1361-6463/abd583

We propose a combination of the Kolmogorov-Johnson-Mehl-Avrami nucleation and growth theory and the Bean-Rodbell model to describe the field-induced transition in $LaFe_{11.6}Si_{1.4}$ alloys. The approach is applied to a set of bulk samples undergoing first-order transitions produced by different routes and including doping effects. The kinetic analysis of both magnetization and demagnetization processes reveals a nucleation and three-dimensional interface-controlled growth for these alloys. Introducing the kinetic process between the metastable and stable solutions of the Bean-Rodbell model, the field dependence of the magnetization/demagnetization processes, including magnetic hysteresis for different magnetic field sweeping rates, is better reproduced than with the pure model.

Experimental method to determine specific heat capacity and transition enthalpy at a first-order phase transition: Fundamentals and application to a Ni-Mn-In Heusler alloy

Romero, FJ; Gallardo, MC; Martin-Olalla, JM; del Cerro, J
Thermochimica Acta, **706** (2021) 179053
Diciembre, 2021 | **DOI:** 10.1016/j.tca.2021.179053

A new method that characterizes thermal properties during a first-order phase transition is described. The technique consists in exciting the sample by a series of constant frequency thermal pulses which one in every *N* pulses -*N* is a small number like four-being exceedingly large in amplitude. This pulse induces phase transformation which is inhibited during the following smaller pulses due to thermal hysteresis. That way the specific heat capacity for a given mixture of phases can be determined. The results obtained are independent of experimental parameters like the rate and the amplitude of the pulses, unlike what happens in other calorimetric techniques. The

method also provides the enthalpy excess by analysing the energy balance between the dissipated heat and the heat flowing during each pulse of measurement.
The protocol is tested to analyse the phase transitions of a Heusler alloy $Ni_{50.53}Mn_{33.65}In_{15.82}$. The paramagnetic-ferromagnetic transition for the austenite phase is continuous and the specific heat capacity shows a lambda anomaly. The martensitic phase transition shows a first-order character and the specific heat capacity follows a step-like behaviour.

Reversibility of the Magnetocaloric Effect in the Bean-Rodbell Model

Moreno-Ramirez, LM; Franco, V
Magnetochemistry, **7** (2021) 60
Mayo, 2021 | **DOI:** 10.3390/magnetochemistry7050060

The applicability of magnetocaloric materials is limited by irreversibility. In this work, we evaluate the reversible magnetocaloric response associated with magnetoelastic transitions in the framework of the Bean-Rodbell model. This model allows the description of both second- and first-order magnetoelastic transitions by the modification of the eta parameter ($\eta < 1$ for second-order and $\eta > 1$ for first-order ones). The response is quantified via the Temperature-averaged Entropy Change (*TEC*), which has been shown to be an easy and effective figure of merit for magnetocaloric materials. A strong magnetic field dependence of *TEC* is found for first-order transitions, having a significant increase when the magnetic field is large enough to overcome the thermal hysteresis of the material observed at zero field. This field value, as well as the magnetic field evolution of the transition temperature, strongly depend on the atomic magnetic moment of the material. For a moderate magnetic field change of 2 T, first-order transitions with $\eta \approx 1.3$-1.8 have better *TEC* than those corresponding to stronger first-order transitions and even second-order ones.

Influence of Cr-substitution on the structural, magnetic, electron transport, and mechanical properties of $Fe_{3-x}Cr_xGe$ Heusler alloys

Mahat, R; Shambhu, KC; Wines, D; Regmi, S; Karki, U; Li, Z; Ersan, F; Law, J; Ataca, C; Franco, V; Gupta, A; LeClair, P
Journal of Magnetism and Magnetic Materials, **521** (2021) 167398
Marzo, 2021 | **DOI:** 10.1016/j.jmmm.2020.167398

We performed combined experimental and theoretical studies of the effect of Cr substitution for Fe on the structural, magnetic, transport, electronic, and mechanical properties of $Fe_{3-x}Cr_xGe$ ($0 \leqslant x \leqslant 1$) intermetallic alloys. Single phase microstructures are observed for $\times \leqslant 0.70$. Higher Cr concentrations $x > 0.70$ are multi-phased. A hexagonal $D0_{19}$ structure is found for all Cr concentrations, with the lattice parameters increasing systematically with an increasing Cr content. All the alloys in the series are found to be ferromagnets with large magnetization values of about 6 $\mu_B/f.u.$ and high Curie temperature above room temperature. The low-temperature saturation magnetic moments agree fairly well with our theoretical results and also obey the Slater-Pauling rule. The density functional theory calculation reveals that Cr substitution energetically favours one of the Fe sites in Fe_3Ge. The electrical resistivity measured over the temperature range from 5 K to 400 K shows metallic behavior, with a residual resistivity ratio that decreases with Cr content. Vicker's hardness values are observed to increase with increasing Cr content to approximately 5 GPa.

Structural, electronic, magnetic, transport and mechanical properties of the half-metal-type quaternary Heusler alloy Co2Fe1-xVxGe

Mahat, R; Shambhu, KC; Karki, U; Regmi, S; Law, JY; Franco, V; Galanakis, I; Gupta, A; LeClair, P
Journal of Magnetism and Magnetic Materials, **539** (2021) 168352
Diciembre, 2021 | **DOI:** 10.1016/j.jmmm.2021.168352

We report on the bulk properties of the Heusler alloy system $Co_2Fe_{1-x}V_xGe$ with $0 \leqslant x \leqslant 1$ in steps of 0.125. We find single-phase alloys only for x = 0.25 and x = 0.375, both of which exhibit an $L2_1$ crystal structure. The alloys are found to be soft ferromagnets with high Curie temperatures (~ 800 K). Magnetization measurement shows the saturation magnetization to be 5.21 $\mu_B/f.u.$ and 4.78 $\mu_B/f.u.$ for $Co_2Fe_{0.75}V_{0.25}Ge$ and $Co_2Fe_{0.625}V_{0.375}Ge$ respectively, in good agreement with the values expected from a Slater-Pauling rule for half metals. In zero applied magnetic field, the resistivity versus temperature of these alloys does not display the usual T-2 dependence at low temperatures indicative of electron-magnon scattering, another indirect suggestion of half metallicity. Our ab initio calculations also predict half-metallic character in the alloys after V substitution. The large exchange splitting between the occupied majority-and unoccupied minority-spin states leads to the Fermi level almost intersecting a peak in the V majority density of states. This leads to a markedly higher spin polarization upon V doping, making V an ideal dopant for achieving half-metallic behavior in Co_2FeGe. Relatively high mechanical hardness values are also observed.

Kinetic Analysis of the Transformation from 14M Martensite to L2(1) Austenite in Ni-Fe-Ga Melt Spun Ribbons

Manchon-Gordon, AF; Lopez-Martin, R; Ipus, JJ; Blazquez, JS; Svec, P; Conde, CF; Conde, A
Metals, **11** (2021) 849
Junio, 2021 | **DOI:** 10.3390/met11060849

In this study, the non-isothermal kinetics of the martensitic transition from 14M modulated martensite to austenite phase in $Ni_{55}Fe_{19}Ga_{26}$ ribbons obtained by melt-spinning has been analyzed. The proximity of the martensitic transition to room temperature makes it very sensitive to pressure and subtle differences for different pieces of the ribbon (ascribed to stresses stored in the ribbon during its rapid solidification process). Despite the dispersion in the characteristic parameters of the transition, a general behavior is observed with a decreasing activation energy as the heating rate increases due to the nucleation driven character of the transition. It has been shown that a first-order autocatalysis can describe the temperature evolution of the austenite fraction using only two experimental temperatures. Predicted curves are in good agreement with experimental data.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

First- and second-order phase transitions in RE6Co2Ga (RE = Ho, Dy or Gd) cryogenic magnetocaloric materials
D. Guo, L.M. Moreno-Ramírez, C. Romero-Muñiz, Y. Zhang, J.Y. Law, V.Franco, J. Wang, Z. Ren
Science China Materials, 64 (2021) 2846-2857

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

Digital Workshop "Materials for Energy"
25-26 febrero [Bochum, Alemania]

Magnetic materials for energy efficient refrigeration
V. Franco
Conferencia Invitada

APS March Meeting 2021
15-19 marzo [Virtual]

Influence of mixing the low-valent transition metal atoms Y = Sc, Ti, V, Cr, Mn and Fe on the properties of possible half-metallic Heusler compounds Co1.5Y0.5FeSi
R. Mahat, S. KC, U. Karki, S. Regmi, J.Y. Law, V. Franco, I. Galanakis, A. Gupta, P. LeClair
Comunicación oral

Workshop on i-Caloric Effects 2021" (WiCE 2021)
30-31 marzo [Río de Janeiro, Brasil]

Characterizing the thermal hysteresis of caloric materials with T-FORC
V. Franco
Conferencia Invitada

International eWorkshop on Science and Technology of Emerging Materials (eSTEM-21)
19-21 abril [Karur, Tamil Nadu, India]

Magnetocaloric Effect: From Energy Efficient Refrigeration to Fundamental Studies of Phase Transitions
V. Franco
Conferencia Invitada

Intermag 2021
26-30 abril [Virtual]

Overcoming the Limitations of Magnetocaloric Rare-Earth-Free High-Entropy Alloys
J.Y. Law, Á. Díaz-García, L. M. Moreno-Ramírez, V. Franco
Conferencia Invitada

Deconvolution of first and second order phase transitions using the scaling laws of the magnetocaloric effect
Á. Díaz-García, J. Y. Law, A. K. Giri, V. Franco
Conferencia Invitada

T-FORC as a tool for the characterization of magnetocaloric materials: from experiments to models
V. Franco, Á. Díaz-García, L.M. Moreno-Ramírez, J.Y. Law, S. Fabbrici, F. Albertini
Conferencia Invitada

Analysis of the Magnetic Field Dependence of the Isothermal Entropy Change of Inverse Magnetocaloric Materials
L. M. Moreno-Ramírez, J.Y. Law, S. S. Pramana, A.K. Giri, V. Franco
Conferencia Invitada

Effect of Mixing the low-Valent Transition Metal Atoms Y = Sc, Ti, v, Cr, Mn and Fe on the Properties of Quaternary Heusler Compounds Co2-xYxFeSi ($0 \leq x \leq 1$)
R. Mahat, S. KC, U. Karki, J.Y. Law, V. Franco, I. Galanakis, A. Gupta, P
Póster

Workshop GECAT 2021 – Young researchers /Jóvenes investigadores
4 junio [Virtual]

Deconvoluting of the magnetocaloric responses due to concurrent first and second-order phase transitions
Á. Díaz-García, J.Y. Law, L. M. Moreno-Ramírez, A. K. Giri, V. Franco
Comunicación oral

Design of Fe-containing GdTbCoAl high-entropy-metallic-glass composite microwires with tunable Curie temperatures and enhanced cooling efficiencies
H. Yin, J.Y. Law, Y. Huang, V. Franco, H. Shen, S. Jiang, Y Bao, J. Sun
Comunicación oral

Thermomagnetic phase transitions and magnetocaloric effect in RE2T2Ga compounds (T=Ni and Co; RE=Dy, Ho, Er and Tm)
Dan Guo, L.M. Moreno-Ramírez, V.Franco, Yikun Zhang
Comunicación oral

Implementation of a simple demonstrator for elastocaloric effect in shape memory alloys
J. Revuelta, J.Y. Law, V. Franco
Comunicación oral

The 9th International Conference on Fracture Fatigue and Wear FFW2021
2-3 agosto [Virtual]

Influence of graphene-based nanostructures morphology on the tribology properties in zirconia composites
C. Muñoz-Ferreiro, R. Moriche-Tirado, Á. Gallardo-López, R. Poyato, F. Gutiérrez-Mora
Comunicación oral

ACS Fall 2021
22-26 agosto [Atlanta, Georgia, Estados Unidos de América]

Mössbauer studies of Fe and Co-based monocrystalline and multicomponent amorphous alloys
J.M. Borrego, J. S. Blázquez, C.F. Conde, A. Conde, J. M. Greneche
Comunicación oral

2021 AtC-AtG Conference
24-25 agosto [Virtual]

Design of Fe-containing GdTbCoAl high-entropy-metallic-glass composite microwires with tunable Curie temperatures and enhanced cooling efficiency
H. Yin, J.Y. Law, Y. Huang, V. Franco, H. Shen, S. Jiang, Y Bao, J. Sun
Comunicación oral

Design of Fe-containing GdTbCoAl high-entropy-metallic-glass composite microwires with tunable Curie temperatures and enhanced cooling efficiency
H. Yin, J.Y. Law, Y. Huang, V. Franco, H. Shen, S. Jiang, Y Bao, J. Sun
Comunicación oral

First- and Second-Order Phase Transitions in RE6Co2Ga (RE = Ho, Dy or Gd) Cryogenic Magnetocaloric Materials
D. Guo, L. M. Moreno-Ramírez, C. Romero-Muñiz, Y. Zhang, J.Y. Law, V. Franco, J. Wang, Z. Ren
Comunicación oral

European congress and exhibition on advanced materials and processes EUROMAT 2021
13-17 septiembre [Virtual]

Processing and mechanical characterization of few layered graphene / zirconia composites
C. Muñoz-Ferreiro, H. Reveron, S. Cottrino, J. Chevalier, A. Morales-Rodríguez, R. Poyato, Á. Gallardo-López
Comunicación oral

Electrical-discharge machinable zirconia composites with graphene nanostructures: Influence of nanostructure type and processing technique
Á. Gallardo-López, R. Moriche, A. Gommeringer, F. Kern, C. López-Pernía, C. Muñoz-Ferreiro, R. Poyato
Comunicación oral

Mechanical properties and fracture mechanisms of graphene-based nanostructures/yttria-stabilized tetragonal zirconia (3ytzp) and their dependence on nanostructure aspect ratio and processing conditions
R. Moriche, C. Muñoz-Ferreiro, C. López-Pernía, E. Guisado, R. Poyato, Á. Gallardo-López
Comunicación oral

R-Curve behavior of GBN/3YTZP composites
C. López-Pernía, H. Reveron, J. Chevalier, A. Morales-Rodríguez, R. Poyato, Á. Gallardo-López
Comunicación oral

Studying the slow crack growth in a few layered graphene / zirconia composite
C. Muñoz-Ferreiro, Á. Gallardo-López, R. Poyato, A. Morales-Rodríguez, H. Reveron, J. Chevalier
Póster

5th Young Researchers in Magnetism - IEEE Spanish Chapter - CEMAG
10-12 noviembre [Girona, España]

Cryogenic Magnetocaloric performance of RENiX2 compounds (RE = Er, Ho and Dy, X = Al and Ga)
D. Guo, L. M. Moreno-Ramírez, Y. Zhang, J.Y. Law, V. Franco
Comunicación oral

Synthesis and Characterization of Magnetocaloric Ni-Co-Mn-Ti Heusler Alloys
A.N. Khan, L.M. Moreno-Ramírez, J.Y. Law, V. Franco
Póster

Enhancing the magnetocaloric response of high-entropy metallic-glass by icrostructural control
H. Yin, J.Y. Law, Y. Huang, H. Shen, S. Jiang, S. Guo, V. Franco, J. Sun
Póster

2nd World Congress on High-Entropy Alloys (HEA2021)
5-8 diciembre [Charlotte, Estados Unidos de América]

One order of magnitude improvement of the magnetocaloric effect of rare-earth-free HEAs
J.Y. Law, Á. Díaz-García, L.M. Moreno-Ramírez, V. Franco
Comunicación oral

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título: **Preparation and Properties of Micro-/Nano-Structured Ni-Mn-Ga Magnetocaloric Alloys and Their Composites**
Autor: Yi Chi Zhang
Directores: HX Peng, FX Qin, V. Franco
Centro: Zhejiang University
Fecha Defensa: Septiembre 2021

Título: **Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno**
Autor: Cristina López Pernía
Directores: Rosalía Poyato Galán y Ángela Gallardo López
Centro: Universidad de Sevilla
Fecha Defensa: 1 de octubre de 2021

Formación de Graduados / Master Degree Thesis

Título: **Procesado y sinterización de matrices de circona cúbica con nanoestructuras de grafeno/ Processing and sintering of cubic circona matrices with Graphene nanostructures**
Autor: Tomás Espuny Plaza
Tutores: Rocío Moriche y Rosalía Poyato
Grado: Trabajo Fin de Grado del Grado en Ingeniería de Materiales
Centro: Facultad de Química, Universidad de Sevilla

Título: **Influencia de la adición de nanoestructuras de grafeno en el comportamiento de cerámicos avanzados sometidos a flexión**
Autor: Elisa Guisado Arenas
Tutores: Rocío Moriche y Ángela Gallardo López
Grado: Trabajo Fin de Grado del Grado en Ingeniería de Materiales
Centro: Facultad de Física, Universidad de Sevilla

Título: **Procesado y caracterización microestructural de composites cerámicos con nanoláminas de nitruro de boro**
Autor: Ana Castro Chincho
Tutores: A. Morales Rodríguez y Rosalía Poyato
Grado: Trabajo Fin de Grado del Grado en Ingeniería de Materiales
Centro: Facultad de Física, Universidad de Sevilla
Fecha Defensa: 17 septiembre 2021

Título: **Modelos de densidades electrónicas simples para describir los factores de dispersión atómicos**
Autor: Alejandro Zurita Caballero
Tutores: Javier S. Blázquez Gámez
Grado: Trabajo Fin de Grado del Doble Grado en Física e Ingeniería de Materiales
Centro: Facultad de Física, Universidad de Sevilla

Título: **Variaciones del nivel del mar en la costa mediterránea española**
Autor: Ana García García
Tutores: Manuel Vargas Yáñez, Josefa Borrego Moro
Grado: Trabajo Fin de Grado
Centro: Facultad de Biología, Universidad de Sevilla

Título: **Simulación de espectros y recuperación de los parámetros hiperfinos en espectroscopía Mössbauer**
Autor: Javier Sivianes Castaño
Tutores: Javier S. Blázquez Gámez
Grado: Trabajo Fin de Grado del Doble Grado en Física e Ingeniería de Materiales
Centro: Facultad de Física, Universidad de Sevilla

Título: **Modelos de densidades electrónicas simples para describir los factores de dispersión atómicos**
Autor: Alejandro Zurita Caballero
Tutores: Javier S. Blázquez Gámez
Grado: Trabajo Fin de Grado del Doble Grado en Física e Ingeniería de Materiales
Centro: Facultad de Física, Universidad de Sevilla

Título: **Estudio de la resistencia al desgaste de compuestos cerámicos con grafeno de pocas capas**
Autor: Fernando Escobar García
Tutores: Rocío Moriche y Felipe Gutiérrez
Grado: Trabajo Fin de Máster del programa de Ciencia y tecnología de nuevos materiales
Centro: Facultad de Física, Universidad de Sevilla

Título: **Caracterización de superficies electromecanizadas de cerámicas con grafeno**
Autor: Pablo Montero Oliva
Tutores: Ángela Gallardo López y Rosalía Poyato
Grado: Trabajo Fin de Máster del programa de Ciencia y tecnología de nuevos materiales
Centro: Facultad de Física, Universidad de Sevilla
Fecha Defensa: 10 diciembre 2021

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales y en titulaciones de Grado y doble Grado de la Universidad de Sevilla (ver ACTIVIDADES DIVULGATIVAS Y FORMATIVAS)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Calorímetro de barrido diferencial (Perkin-Elmer DSC7)
- Criostato para espectrómetro Mössbauer
- Balanza termogravimétrica (Perkin-Elmer TGA-7)
- Espectrómetro Mössbauer (Wissel MB-500) con horno y criostato.
- Coercímetro (desarrollado en el laboratorio)
- Magnetómetro de muestra vibrante (LakeShore 7000) con horno y criostato.
- Equipo de medida directa de temperatura adiabática (Advanced Magnetic Technologies).
- Molino Planetario (Fritszch Pulverisette Vario 4)
- Equipo de solidificación por enfriamiento ultrarrápido (melt spinning, Bühler)
- Equipo de fusión por arco (MAM1, Bühler)
- Calorímetro de conducción, resolución en la medida del flujo de calor mejor que 0,1 W, fluctuaciones en temperatura del orden de 10^{-6} K, velocidad de barrido menor de 0,01K/h, rango 80-320K, es posible aplicar tensión uniaxial hasta 30 kg/cm^2 y campo eléctrico hasta 800V/cm
- Calorímetro de conducción, rango 80-400K, campo eléctrico 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia
- Adelgazador iónico Gatan. Model 691. Precision ion polishing system.
- Autoclave SanoClav
- Balanzas de precisión: GR.-200; AND EK-300i.
- Centrifugadora Rotofix 32A
- 4 Cortadoras Isomet Low Speed Saw (Buehler)
- Cortadora mediana Isomet 1000. Precision saw.
- Cortadora Isomet 4000. Linear Precision Saw. Buehler.
- Embutidora Pneumet II. Mounting Press. Buehler/Metaserv.
- Embutidora Simplimet 1000. Automatic Mounting Press. Buehler
- Estufa
- 2 Discoplan TS Struers
- 2 Dimple Grinder. Model 656. Gatan.
- Durómetro Wilson VH1150
- Durómetro Duramin Struers
- Hornillo/agitador magnético SBS, Heat-stir “Stuart” , CB302
- Horno de tubo de argón. Termolab, Hornos eléctricos TH1700
- Horno de aire Lenton Furnaces
- Horno mufla
- Limpiadores ultrasonidos Bandelin, Sonorex Digitec; Branson 3510
- Máquina de ensayos de deformación Zwick / Roell Zmart. Pro 1185

- Máquina de ensayos de deformación Instron 5982
- Máquina de fluencia con flujo de gases.
- Máquina de fluencia estanca de argón.
- Microscopio Óptico Confocal Leica DCM3D
- Microscopio Óptico Leica DMRD / Leica DMRE / Leica DFC420
- Molino de agitación PM100 Retsch
- Molino de bolas Molino mezclador MM200, Retsch
- Multímetro Keithley 2000 multimeter.
- Plasma etching Emitech k1050X
- Pulidoras Buehler. Beta. Grinder-Polisher y Motopol 8. Buehler Metaserv
- Prensa Manual Astur Sinter
- Prensa Fluxana, Vaneox, Pressing Technology
- 2 Refrigeradoras. (P. Castro Ibérica)
- Registro Tesatronic (Tesa TT60)
- Registro gráfico (Servogor 122 DC/kipp & Zonen)
- Reómetro Reactor Controller Controller 4836
- Rotavapor R-100. Buehi
- 4 Sondas de Ultrasonidos
- Tribómetro Microtest MT/30/SCM/T
- 2 Ultrasonic Disc Cutter Model 601 Gatan.

SERVICIOS GENERALES
GENERAL SERVICES

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Adquisición de un equipo de medida de Espectroscopía Ultrarrápida de estados transitorios para el Servicio de Espectroscopías del ICMS (EQC2018-004413-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 441.843,34 €
Periodo: 1-1-2018 / 31-03-2021
Cofinanciado por el Grupo de Investigación "Materiales Ópticos Multifuncionales"

Actualización Analizador del Servicio de Análisis de Superficies del ICMS (EQC2019-005712-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 173.653,02 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición de un equipo de difracción de rayos X multicomponente (monocromador Ka1, microdifracción y textura) para el servicio general de difracción de rayos X (IE17-5351)
Financia: Junta de Andalucía
Importe Concedido: 270.000,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición e Instalación de un calorímetro de altas prestaciones (IE17-5798)
Financia: Junta de Andalucía
Importe Concedido: 202.300,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Equipo de Nanoindentación para la evaluación de propiedades mecánicas en la nanoescala (IE17-5311)
Financia: Junta de Andalucía
Importe Concedido: 200.000,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición e instalación de un equipo de medida de espectrometría, eficiencia cuántica y distribución angular de electroluminiscencia, para el Servicio de Espectroscopias del ICMS (IE19_221)
Financia: Junta de Andalucía
Importe Concedido 120.020,00 €
Periodo: 29-12-2020 / 28-12-2022
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

SERVICIO DE ESPECTROSCOPIAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopias incluye las Unidades de Espectroscopía Raman, Espectroscopía Infrarroja y Espectroscopía Ultravioleta-Visible. Este servicio está dedicado a la determinación de la estructura molecular de los compuestos químicos y la caracterización de materiales.

This Service consists of four different spectroscopies: Raman Spectroscopy, Infrared Spectroscopy and Ultraviolet-Visible Spectroscopy. It is devoted to the determination of molecular structure of chemical compounds and materials.

ESPECTROSCOPÍA MICRO-RAMAN / MICRO-RAMAN SPECTROSCOPY

La espectroscopía Raman se basa en un proceso fotónico en el que la radiación incidente es dispersada por la muestra, produciéndose transiciones de tipo vibracional y rotacional. En general, el espectro Raman se interpreta como un espectro vibracional que ofrece información muy similar al espectro de infrarrojo, aunque las vibraciones que se ven reflejadas en el espectro Raman no son siempre las mismas que en aquél. Para que un modo vibracional sea activo en espectroscopía Raman es necesario que se produzcan cambios en la polarizabilidad de los enlaces químicos o la molécula considerada, lo que conlleva la producción de momentos dipolares inducidos. Su campo de aplicación es muy extenso: semiconductores, compuestos del carbono (grafito, diamante, nanotubos, fibras…), catalizadores, pigmentos, etc.

Raman spectroscopy is based on a photonic process in which the incident radiation is dispersed by the sample. This latter is perturbed leading to vibrational and rotational transitions. In general, the Raman spectrum is interpreted like a vibrational one, providing information very similar to the infrared spectroscopy, although the Raman active vibrations are not always the same as those excited with infrared radiation. A Raman vibration mode is active if there is a change of polarizability of the chemical bonds or the considered molecule, which in turn results in the generation of induced dipolar momentum. Its application fields are very broad: semiconductors, carbon compounds (graphite, diamond, nanotubes, fibers…), catalysts, pigments, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- LabRAM Horiba Jobin Yvon dotado de un microscopio confocal y 3 longitudes de excitación (785 cm^{-1} rojo, 532 cm^{-1} verde, y 325 cm^{-1} UV)
  LabRAM Horiba Jobin Yvon equipped with a confocal microscope and 3 excitation lasers (785 cm^{-1} red, 532 cm^{-1} green, and 325 cm^{-1} UV)

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

ESPECTROSCOPÍA INFRARROJA / INFRARED SPECTROSCOPIES

La espectroscopía de Infrarrojos (FT-IR) se basa en la absorción de radiación infrarroja por parte de los materiales. Esta absorción supone un cambio en la energía vibracional de los enlaces, siempre que se produzca un cambio en la polarización de dicho enlace. El resultado obtenido es un espectro en el que se representa la radiación absorbida o transmitida en función del número de onda de la radiación, lo cual permite identificar el enlace correspondiente.
El equipo en el ICMS cubre un rango de número de ondas que va desde 5000 a 250 cm-1 (óptica de CsI) y se puede trabajar con purga o en vacío. Se halla equipado con accesorios para trabajar en los modos de Reflectancia Difusa (DRIFT), Reflectancia Total Atenuada (ATR) y Reflexión Especular. Dispone de un microscopio de Infrarrojos que tiene una resolución lateral de 10 μm.

Infrared spectroscopy (FT-IR) is based on the selective absorption of the infrared radiation by the materials. This absorption means a change in the vibrational energy of the chemical bonds, whenever it occurs a change in the polarization. The result is a spectrum showing the absorbed or transmitted radiation as a function of the wavenumber of the radiation, which can be assigned to the corresponding chemical bound.
The equipment at the ICMS works in a wavenumber range from 5000 to 250 cm^{-1} (CsI optic), and can operate with a gas purge or in vacuum. It is equipped with several accessories to do Diffuse Reflectance (DRIFT), Attenuated Total Reflectance (ATR) or Specular Reflectance. It has got an Infrared Microscope with a lateral resolution of 10 μm.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- JASCO FT/IR-6200 IRT-5000
  JASCO FT/IR-6200 IRT-5000

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

ESPECTROSCOPÍA ÓPTICA EN EL RANGO ULTRAVIOLETA, VISIBLE E INFRARROJO CERCANO / ULTRAVIOLET-VISIBLE-NEAR INFRARED SPECTROSCOPIES

La técnica de espectroscopía en el rango ultravioleta, visible e infrarrojo cercano (UV-Vis-NIR) nos permite conocer como materiales de distinta morfología (principalmente polvos, láminas y partículas o moléculas en suspensión) reflejan y transmiten la luz incidente en el rango comprendido entre 190 nm y 3000 nm. De esta forma, es posible extraer información sobre su eficiencia como filtros ópticos, ya sean especulares o difusores, y/o sobre la luz absorbida por ellos, lo que indirectamente nos permite estimar su gap electrónico (en el caso de dieléctricos), las transiciones electrónicas que tienen lugar (en el caso de moléculas o sistemas dopados con átomos de otra especie), o las resonancias plasmónicas (en el caso de metales).

The Ultraviolet-Visible-Near Infrared Spectroscopy (UV-Vis-NIR) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
The equipment in the laboratory, which works in the wavelength range of 190 nm to 900 nm can operate in the Transmission mode or in Diffuse Reflectance Modes.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Cary 5000 + UMA (Universal Measurement Accesory)
  Cary 5000 + UMA (Universal Measurement Accesory)

- Cary 300
  Cary 300

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

LABORATORIO DE ESPECTROSCOPÍA ULTRA-RÁPIDA / ULTRAFAST EMISSION AND ABSORPTION SPECTROSCOPY

El laboratorio de espectroscopía ultra-rápida permite realizar medidas de absorción y emisión resueltas en el tiempo con una resolución temporal de 190 femtosegundos (fs) y un amplio rango temporal que va de los 190 fs a 1 milisegundo (ms). Las medidas pueden realizarse en el rango espectral 350-850 nm.
The ultra-fast spectroscopy laboratory allows performing time-resolved absorption and emission measurements with a time resolution of 190 femtoseconds (fs) over a broad temporal range (190 fs - 1 millisecond). Measurements can be carried out in the 350-850nm spectral range.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Sistema de excitación láser ultra-rápido formado por un láser pulsado PHAROS (Light Conversion) (longitud de onda de emisión 1030nm, tasa de repetición 1kHz y duración de pulso 190fs) y un amplificador paramétrico (OPA) ORPHEUS (Light Conversion) que produce pulsos de duración y tasa de repetición iguales al PHAROS pero con una longitud de onda sintonizable en el rango 350-2500nm.

- Espectrómetros de absorción para el rango temporal 190fs-8ns (HELIOS, Ultrafast Systems) y 2ns-1ms (EOS, Ultrafast Systems). Ambos sistemas permiten realizar medidas en el rango espectral 350-1100nm con una resolución de 2nm.

- Espectrómetro de emisión para el rango temporal 190fs-5ns (HALCYONE, Ultrafast Systems) oeprativo en el rango espectral 350-1100nm.

- Sistema de time-correlated single-photon counting (TCSPC) para realizar medidas de emisión resuelta en el tiempo en el rango temporal 1ns-1ms y en el rango espectral 200-850nm.

Responsables Científicos/ Scientific Responsibles: Dr. Hernán Míguez García, Dr. Juan F. Galisteo López

SERVICIO DE ESPECTROMETRÍA DE EMISIÓN ATÓMICA / ATOMIC EMISSION SPECTROMETRY SERVICE

La espectrometría de emisión atómica de plasma acoplado inductivamente (ICP-OES) es una técnica analítica que permite la cuantificación de elementos hasta nivel traza en muestras en solución. La muestra a analizar es nebulizada y conducida a un plasma de argón, en donde se produce la desolvatación, vaporización, atomización e ionización de los elementos a analizar. Los átomos e iones excitados por la elevada energía térmica suministrada por el plasma emiten durante el proceso de relajación radiación electromagnética de longitudes de onda características de cada elemento. La intensidad de las distintas líneas de emisión es proporcional a la concentración del analito y con la correspondiente curva de calibración es posible realizar su cuantificación. Esta técnica presenta elevada sensibilidad, excelente límite de detección (en el rango ppb, μg/L), buena precisión, alto rendimiento y capacidad multi-elemental, aunque en determinadas ocasiones se pueden producir interferencias espectrales debido a un alto número de líneas de emisión.

Se pueden suministrar muestras sólidas, realizándose la digestión por parte del servicio, o líquidas en solución acuosa ligeramente ácida. No se admiten muestras en HF. Las muestras líquidas no deben presentar precipitados ni coloides en suspensión y deberán poseer un volumen mínimo de 10 ml. Las muestras se entregarán al técnico encargado del servicio, junto con la solicitud de análisis debidamente cumplimentada que se encuentra disponible en la web del ICMS.

Inductively coupled plasma atomic emission spectrometry (ICP-OES) is an analytical technique that allows the quantification of elements up to the trace level in samples in solution. The sample to be analyzed is nebulized and conducted to an argon plasma, where desolvation, vaporization, atomization and ionization of the elements take place. The atoms and ions reach an excited state by the high thermal energy supplied by the plasma and during the relaxation process electromagnetic radiation is emitted with wavelengths characteristic of each element. The intensity of the different emission lines is proportional to the concentration of the elements, which can be quantified by using appropriate calibration curves. This technique has high detection limits (in the ppb range, µg / L), good reliability, high throughput and multi-elemental capacity, although in some cases spectral interferences can occur due to a high number of emission lines.

Solid samples (digestion will be carried out by the service) or liquid samples in slightly acidic aqueous solution can be supplied. Samples in HF medium are not allowed. Liquid samples must not present precipitates or colloids in suspension and must have a minimum volume of 10 ml. The samples will be delivered to the technician in charge of the service, together with the duly completed analysis request that is available on the ICMS website.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)
  iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)

- Digestor por microondas ETHOS EASY (Milestone)
  Microwave Digestion System ETHOS EASY (Milestone)

Responsables Científicos/ Scientific Responsibles: Dr. Francisco José Gotor Martínez
Personal Técnico/ Technical Assistant: Lda. Belinda Sigüenza Carballo

SERVICIO DE ANÁLISIS TEXTURAL Y TÉRMICO / TEXTURAL AND THERMAL ANALYSIS SERVICE

Este servicio incluye las siguientes Unidades: Análisis Térmico, Fisi-quimisorción, Análisis de Tamaño de Partícula y Potencial Z. Está dedicado a la determinación de la textura, estructura y comportamiento térmico de los materiales.

This Service includes the following units: Thermal Analysis, Physisorption and Chemisorption, Particle Size and Z-potential determination. It is devoted to the characterization of texture, microstructure and thermal behavior of advanced materials.

FISI-QUIMISORCIÓN / PHYSISORPTION-CHEMISORPTION

Este servicio constituye una herramienta básica para la caracterización microestructural de sólidos pulverulentos de distinta naturaleza, en cuanto a porosidad, superficie específica y superficie químicamente activa.

En el servicio se dispone de un analizador de adsorción de gases (Micromeritics, ASAP 2020) que proporciona isotermas de adsorción y desorción, a partir de los cuales se obtienen de ellas la superficie específica y distribución del tamaño de poro y de microporo de estos materiales, incorporando también los accesorios necesarios para medidas de quimisorción.

This service constitutes a basic tool for the microstructural characterization of powdered solids of different natures, regarding to their porosity, specific surface area and chemically active surface.

This service is composed by a phyisisorption analyser (Micromeritics, ASAP 2020) which provides the complete adsorption/desorption isotherms, from which the specific surface area, pore and micropore size distribution and concentration of reactive sites are obtained. The instrument is also equipped for carrying out chemisorption of different reactive molecules, as O_2, H_2, CO, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Analizador científico de fisisorción ASAP2010 (Micromeritics)
  Physisorption analyser ASAP 2010 (Micromeritics)

- Analizador de quimisorción ASAP2010 (Micromeritics)
  Chemisorption analyser ASAP 2010 (Micromeritics)

- Analizador de fisisorción multimuestra TRISTAR II (Micromeritics)
  Multisample physisorption analyser TRISTAR II (Micromeritics)

- Analizador de fisisorción multimuestra TRISTAR II-Kr (Micromeritics)
  Multisample physisorption analyser TRISTAR II-Kr (Micromeritics)

Responsables Científicos/ Scientific Responsibles: Dr. Gerardo Colón Ibáñez y Dr. Alfonso Caballero Martínez
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

ANÁLISIS TÉRMICO / THERMAL ANALYSIS

Las técnicas de análisis térmico permiten estudiar aquellos cambios físicos o químicos que ocurren en los sólidos en función de la temperatura y que conlleven modificaciones en su masa o intercambios de calor con su entorno.
En el servicio se pueden realizar experimentos desde temperatura ambiente hasta 1500 °C, tanto en atmósfera inerte (N_2) como reactiva (aire, O_2,…).
Se dispone de dos técnicas: Análisis Termogravimétrico (TG) y Análisis Térmico Diferencial (ATD).

Thermal analysis techniques allow to studying physical or chemical changes occurring in solid in samples as a function of the temperature. Those changes should involve either a mass change or a heat flow.
The experiments can be performed in the range from room temperature to 1500 °C, both under inert (N_2), or reactive (air, O_2,…) atmospheres.
Two different techniques are available: Thermogravimetry (TG) and Differential Thermal Analysis (DTA)

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Equipo termogravimétrico simultáneo TG/DTA/DSC STA449 F5 Jupiter (NETZSCH)
  STA449 F5 Jupiter (NETZSCH) Simultaneous TG/DTA/DSC Instrument

- Dilatómetro mecánico horizontal DIL 402 Expedis Select (NETZSCH)
  DIL 402 Expedis Select (NETZSCH) Horizontal Dilatometer

- Equipo de análisis térmico simultáneo TG/ATD/CDB TA Instruments Q600
  TA Instruments Q600 Simultaneous TG/DTA/DSC instrument

- Equipo termogravimétrico TG, TA Instruments Q5000
  Thermogravimetric instrument TG, TA Instruments Q5000

- Equipo de calorimetría Calvet, Setaram Sensys
  Calvet Calorimetry Equipment, Setaram Sensys

Responsable Científico/ Scientific Responsible: Dr. Luis A. Pérez Maqueda
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

SERVICIO DE MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SERVICE

El servicio está dedicado a la caracterización química y estructural de muestras sólidas mediante técnicas de microscopía electrónica. Las técnicas de caracterización disponibles en el servicio son la Microscopía Electrónica de Transmisión (TEM) y la Microscopía Electrónica de Barrido (SEM), con el equipamiento anexo de preparación de muestras para TEM y SEM.

This Service is devoted to the chemical and structural characterization of solid samples by means of electron microscopies. The characterization techniques available at ICMS are Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM), with additional equipment for TEM and SEM sample preparation.

MICROSCOPÍA ELECTRÓNICA DE BARRIDO / SCANNING ELECTRON MICROSCOPY

La microscopía electrónica de barrido proporciona información microestructural, morfológica y de composición química en escala microscópica. Se puede aplicar a todo tipo de materiales y problemáticas de estudio en ciencia de materiales: cerámicas, plásticos, metales, minerales, catalizadores, muestras de patrimonio histórico, capas finas, recubrimientos, interfases, nanopartículas, etc. El equipo SEM es un microscopio de emisión de campo de cátodo frío que permite realizar imágenes de la morfología y textura superficial de las muestras con una resolución de 1 nm a 15kV. También permite trabajar a bajo voltaje en muestras sin metalizar y en modo transmisión (STEM-in-SEM) en muestras electrón-transparentes. Acoplado al detector de rayos-X (EDX) permite análisis elementales y mapas composicionales.

The scanning electron microscopy provides information about the microstructure, morphology and chemical composition at the microscopic scale of solid samples. It can be applied to all type of materials including ceramics, polymers, metals, minerals, catalysts, samples from cultural heritage, thin films, coatings, interfaces, nanoparticles, etc. The SEM microscope is a field emission cold cathode equipment which enables images of the surface morphology and texture of samples with a resolution of 1 nm at 15kV. It also allows working at low voltages with non-metalized samples and in transmission mode for electron-transparent samples (STEM-in-SEM). Coupled to the X-ray detector (EDX) enables compositional analysis and elemental mapping.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio SEM, modelo Hitachi S4800 SEM-FEG: cañón de emisión de campo de cátodo frío y voltaje de 0.5-30 kV, resolución de 1 nm a 15kV. Dotado de analizador EDX Bruker-X Flash-4010 con una resolución de 133 eV (en la línea MnKα) y detector con portamuestras para trabajar en modo transmisión (STEM-in-SEM).
  Hitachi S4800 SEM-FEG microscope: cold cathode field emission gun with voltage from 0.5 to 30 kV, resolution of 1nm at 15 kV. Equipped with a Bruker-X Flash-4010 EDX detector with a resolution of 133 eV (at the MnKα line), and a detector with sample holder to work in transmission mode (STEM-in- SEM).

- Equipamiento adicional en el "laboratorio de preparación de muestras para microscopía electrónica"
  Additional equipment in the "electron microscopy samples preparation laboratory"

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho, Dra. Cristina T. Rojas Ruiz
Personal Técnico/ Technical Assistant: Dra. M. Carmen Jiménez de Haro

MICROSCOPÍA ELECTRÓNICA DE TRANSMISIÓN / TRANSMISSION ELECTRON MICROSCOPY

La microscopía electrónica de transmisión es una técnica ampliamente utilizada para la caracterización estructural y química de materiales a escala microscópica y nanoscópica, proporcionando imágenes bidimensionales de la textura de la muestra, forma y tamaño de grano y/o de partícula, grado de homogeneidad a escala microscópica, grado de cristalinidad de la muestra, identificación de fases cristalinas, e imágenes de alta resolución que identifican dominios cristalinos. El equipo está dotado de un analizador EDX para el análisis composicional. Puede aplicarse a todo tipo de materiales y campos de estudio en ciencia y tecnología de materiales trabajando sobre muestras electrón-transparentes preparadas en su caso ad-hoc para este fin. El servicio realiza microscopía en modo transmisión: Imágenes en campo claro y campo oscuro, difracción de electrones de área seleccionada y microscopía electrónica de alta resolución así como análisis elemental de área seleccionada. No se dispone de modo STEM.

The transmission electron microscopy is a widely used technique for the microstructural and chemical characterization at micro and nanoscales, providing two-dimensional images of the sample texture and shape as well as grain and/or particle size, degree of homogeneity at the microscopic scale, degree of crystallinity of the sample, identification of crystalline phases, and high resolution images to identify the crystalline domains. The microscope is equipped with an EDX analyzer for compositional analysis. It can be applied to all type of materials and research topics in materials science and technology working with electron-transparent samples prepared ad-hoc for this end. The service performs transmission electron microscopy: Imaging in bright and dark field, selected area electron diffraction and high resolution electron microscopy, as well as elemental analysis of selected areas. It does not provide STEM mode.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio Philips CM20 (200kV) con una resolución estructural de 0.14 nm entre líneas y 0.23 nm entre puntos, portamuestras de uno y dos giros y de calentamiento. Acoplados al equipo se dispone de un sistema de análisis por Energías Dispersivas de Rayos X (EDX X-Max 80T, Oxford Instruments) y una cámara CCD (Gatan) para registro de imágenes.
  Philips CM200 microscope (200kV) with a structural resolution of 0.14 nm between lines and 0.23 nm between points. Sample holders with one and two angles and heating. It is equipped with a X-ray Energy Dispersive Analyzer (EDX X-Max 80T, Oxford Instruments) and a CCD GATAN camera for image acquisitions.

- Equipamiento adicional en el "laboratorio de preparación de muestras para microscopía electrónica"
  Additional equipment in the "electron microscopy samples preparation laboratory"

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho, Dra. Cristina T. Rojas Ruiz

Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (cicCartuja), Dª María Inmaculada Rosa Cejudo

LABORATORIO DE NANOSCOPÍAS Y ESPECTROSCOPÍAS-LANE / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio LANE cuenta con un microscopio TEM de emisión de campo Tecnai F30, dotado con modo STEM, detectores HAADF y EDX y filtro de energía (GIF). Las técnicas disponibles incluyen: medidas TEM en campo claro y campo oscuro; TEM de alta resolución; difracción de electrones; análisis STEM-HAADF; análisis EDX y STEM-EDX así como EELS y STEM-EELS, incluyendo medidas puntuales, en línea y mapas composicionales; imágenes EFTEM; análisis espectro-imagen y tomografía electrónica.

The LANE laboratory includes a Tecnai F30 field emission TEM microscope, equipped with STEM mode, HAADF and EDX detectors and an energy filter (GIF). Available techniques include: TEM measurements in bright and dark field; high resolution TEM; electron diffraction; STEM-HAADF analysis; EDX and STEM-EDX analysis as well as EELS and STEM-EELS, including point and in-line measurements and compositional maps; EFTEM images; spectrum-image analysis and electronic tomography.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio Tecnai G2 F30 S-TWIN de 300KV con cañón de emisión de campo. Resolución estructural de 0.2 nm entre puntos, portamuestras de uno y dos giros. Acoplados al equipo se dispone de un detector EDX Silicon Drift Detector X-Max 80T (Oxford Instruments) y un filtro de energías Gatan (GIF Quantum SE)
  Tecnai G2 F30 S-TWIN 300KV microscope with field emission gun. Structural resolution of 0.2 nm between points, sample holders with one and two angles. Attached to the equipment are an EDX Silicon Drift Detector X-Max 80T Detector (Oxford Instruments) and a Gatan energy filter (GIF Quantum SE)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho, Dra. Cristina T. Rojas Ruiz

Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (cicCartuja) , Dª María Inmaculada Rosa Cejudo

LABORATORIO DE PREPARACIÓN DE MUESTRAS PARA MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio de preparación de muestras para TEM y SEM dispone de metalizador de oro, evaporador de carbón, metalizador de Cr y carbón, cortadora de disco, pulidora, “disc-grinder”, cortadora ultrasónica, pulidora cóncava (dimple) y adelgazador iónico (Fischione 1010).

The laboratory for TEM and SEM samples preparation has a gold coater, a carbon evaporator, a metallization system for Cr and carbon, a diamond wheel cutter, a grinder with disc-grinder device, an ultrasonic cutter, a concave polishing (dimple) and ion thinning (Fischione 1010).

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistants: Dª María Inmaculada Roja Cejudo, Dª Olga Montes Amorín (cicCartuja) y Dra. M. Carmen Jiménez de Haro

SERVICIO DE PREPARACIÓN Y CARACTERIZACIÓN DE SISTEMAS CATALÍTICOS HETEROGÉNEOS / PREPARATION AND CHARACTERIZATION SERVICES OF HETEROGENEOUS CATALYTIC SYSTEMS

Este Servicio puede suministrar todo tipo de muestras sólidas con actividad catalítica en diversos procesos de interés industrial, energético y medioambiental.
Las muestras se suministran en cualquier etapa de preparación, con o sin pretratamiento o incluso listas para ser utilizadas. Puede incluir su caracterización por diversas técnicas físicas y químicas.

This Service can supply all types of solid samples with catalytic activity in various processes of industrial, energy and environmental interest.
Samples are supplied at any stage of preparation, with or without pretreatment or even ready to use. It can include their characterization by various physical and chemical techniques.

PRESTACIONES DEL SERVICIO / SERVICES PROVIDED

- Preparación de muestras
  Sample preparation

- Tratamientos térmicos y químicos
  Thermal and chemical treatments

- Evaluación de las prestaciones catalíticas
  Evalutaion of catalytic performance

Responsables Científicos/ Scientific Responsibles: Dr. Alfonso Caballero Martínez

SERVICIO DE DIFRACCIÓN DE RAYOS X
X-RAY DIFFRACTION LABORATORY SERVICE

La difracción de rayos-X permite la identificación cualitativa y cuantitativa de sustancias cristalinas y su caracterización microestructural y textural.
El servicio dispone en la actualidad de cuatro difractómetros independientes, configurados específicamente para abordar el análisis de muestras policristalinas de muy distinta naturaleza, en lo referente a su composición, estabilidad química, cristalinidad, etc.
Asimismo, con alguno de ellos se pueden llevar a cabo, además de los análisis rutinarios (θ-2θ), otros varios más avanzados, como pueden ser:

- Seguir las transformaciones de fase “in situ” provocadas por calentamientos en atmósfera inerte (vacío, Ar) o reactiva (H2, O2,..).
- Caracterizar materiales en la nanoescala (1-100 nm) mediante el estudio de la dispersión de rayos-X a ángulos bajos (SAXS).

- Determinar el grosor, densidad y rugosidad de películas delgadas, mediante Reflectometría de rayos-X.
- Obtener la estructura cristalina de materiales inestables a la atmósfera o muy transparentes a los rayos-X, mediante el empleo de capilares.

X-ray diffraction allows the qualitative and quantitative identification of crystalline substances and their microstructural and textural characterization.
At present, four independent diffractometers are available in this service, specifically configured to analyze the composition, chemical stability, crystallinity and many other properties in polycrystalline samples of a varied nature. Besides ordinary analyses (θ-2θ), part of the equipment can perform some advanced studies as:

- Direct monitoring of transformations undergone in materials under heating, such as phase changes, under inert o reactive atmosphere.
- To characterize materials at the nanoscale (1-100 nm) through X-ray scattering at low angles, using the SAXS technique.
- To measure some physical parameters of layers such as density, thickness and surface roughness with the reflectometry setup.
- To obtain the diffraction patterns of samples either sensitive to the atmosphere or highly transparent to X-rays (organic compounds) employing the capillary configuration.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Difractómetro Panalytical X'PERT PRO con cargador automático de muestras
  Diffractometer PANALYTICAL X'PERT PRO with automatic sample charger

- Difractómetro PHILIPS X'PERT PRO con cámara de alta temperatura (1200 °C) ANTON PAAR HTK 1200
  Diffractometer PHILIPS X'PERT PRO with high temperature chamber (1200 °C) ANTON PAAR HTK 1200

- Difractómetro Panalytical X'PERT PRO (reflectometría, SAXS, ángulo rasante y capilares)
  Diffractometer PANALYTICAL X'PERT PRO (reflectometry, SAXS, low angle scattering and capillary)

- Difractómetro de polvo SIEMENS D5000 DUAL (reflexión y transmisión)
  Diffractometer SIEMENS D5000 DUAL (reflection and transmission)

Responsable Científico/ Scientific Responsible: Dra. Concepción Real Pérez
Personal Técnico/Technical Assistant: Dr. José María Martínez Blanes

SERVICIO DE ANÁLISIS DE SUPERFICIE/ SURFACE ANALYSIS SERVICE

El Servicio de Análisis de superficie consta de un espectrómetro de Espectroscopía de Fotoelectrones de Rayos X (XPS). Este servicio está dedicado al análisis químico y electrónico de superficies sólidas. También permiten conocer la composición en profundidad (desde la superficie hacia el interior) de los sólidos.

The surface analysis service consists of an X-ray Photoelectron Spectrometer (XPS). This service is devoted to the electronic and chemical analysis of solid surfaces. It also provides information about the compositional depth profile of solids (from their surface toward their bulk).

ESPECTROSCOPÍA DE FOTOEMISIÓN DE ELECTRONES / X-RAY PHOTOELECTRON SPECTROSCOPY

Las "Espectroscopías de Fotoelectrones" (XPS/ESCA y AES) son unas poderosas técnicas de análisis cuantitativo no destructivo, sensibles exclusivamente a las primeras capas de la superficie de los sólidos (20-30 Å), lo que permite obtener información sobre las propiedades químicas, físicas y electrónicas de las mismas.
El interés técnico de esta información es enorme en campos tales como corrosión, catálisis, tratamientos de superficies, fenómenos de flotación y adherencia, segregación de fases, etc.
La característica más importante de la Espectroscopía de Fotoelectrones (XPS/ESCA) es que permitir diferenciar distintos estados de oxidación y/o situaciones del entorno (coordinación) de los átomos en las muestras sólidas analizadas. El límite de detección es del 0.5% para cada especie química. El servicio dispone actualmente de dos instrumentos independientes.

Typically, "photoelectron spectroscopies" are a powerful set of non-destructive analysis techniques, exclusively sensitive to the more superficial few atomic layers (20-30 Å), allowing to obtain valuable information about their chemical, physical and electronic properties.
The technical interest of the resulting information is huge in fields such as catalysis, corrosion, surface treatments, floating and adhesion phenomena, or segregation processes, among others.
The most remarkable characteristic of X-Ray Photoelectron Spectroscopy (XPS/ESCA) is that it allows to discriminate, for a given element, between different oxidation states or chemical surroundings (coordination).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

Espectrómetro de Fotoelectrones PHOIBOS 100-DLD, compuesto de:
Photoelectron Spectrometer PHOIBOS 100-DLD, consisting on:

- Cámara de análisis, analizador hemiesférico multicanal PHOIBOS 100-DLD, manipulador de cuatro ejes, y fuentes de excitación de rayos X (dual, AlKα y MgKα, acromático), de luz ultravioleta y de haces de electrones, lo que permite realizar análisis superficiales mediante técnicas de XPS, UPS, ISS y REELS, así como estudios angulares.

Analysis Chamber, equipped with a hemispheric multichannel analyser PHOIBOS 100-DLD, a four axis manipulator, a dual X-ray source (achromatic AlKα, Mg Kα), a UV lamp, and a electron gun, allowing to perform surface analysis by XPS, UPS. ISS and REELS, including angular resolved studies.

- Dos Precámaras de tratamientos, con vacío residual de 10^{-8} y 10^{-9} mbar respectivamente, en las que es posible someter a las muestras a tratamientos diversos como: calentamientos a alta temperatura (T<800 °C) bajo atmósfera controlada, desbastado iónico con gases inertes o reactivos, exposición a plasmas, iluminación con laser, deposición de metales, óxidos y compuestos sencillos, exfoliación in situ, etc.
  Two prechambers for different treatments, with ultimate vacuum levels of 10-8 and 10-9 mbar respectively, where samples can be subjected to diverse treatments and transferred to the analysis chamber without exposure to the atmosphere. The possible treatments include heating at high temperature (< 800 °C) under controlled atmosphere, ion sputtering with inert or reactive gases, exposure to plasma, laser treatments, deposition of metals, oxides or simple compounds, exfoliation, etc.

Espectrómetro de Fotoelectrones SPECS, compuesto de:
Photoelectron Spectrometer SPECS, consisting on:

- Cámara de análisis, dotada de analizador hemiesférico multicanal PHOIBOS 100, manipulador de tres ejes, y fuentes de excitación de rayos X (dual, Alkα y MgKα).
  Analysis Chamber, equipped with a hemispheric multichannel analyser PHOIBOS 100, three axis manipulator and dual X-ray source (achromatic Al Kα, Mg Kα).

- Precámara de tratamiento de alta presión y alta temperatura (HPHT Cell). En esta Cámara es posible someter a las muestras a tratamientos térmicos en presencia de gases hasta una presión de 20 atm y 800 °C, tanto en estático como en dinámico (simultáneamente).
  Pre-chamber for High Pressure/High Temperature treatments (HPHT Cell).Samples can be subjected to treatments in the presence of gases up to 20 bar and 800 °C (simultaneously). These treatments can be performed either under static or flowing gas conditions. After treatments, samples can be transferred to the analysis chamber without exposure to the atmosphere.

- Una cámara de inserción rápida dotada de sistema de aparcamiento/ desgasificado, que permite evacuar las muestras a temperatura reducida (T < 150 °C). También es posible la realización de tratamientos de desbatado iónico o la incorporación de otros sistemas (iluminación con luz Uv-Vis, evaporación de metales, u otros compuestos, etc.)
  A Fast entry chamber, equipped with a parking and degassing system, allowing the samples to be evacuated at moderate temperature (T < 150 °C). It is also possible to sputter the samples under an accelerated ion beam (0.5- 5.0 kV) using inert or reactive gases. Incorporation of some other equipment (Visible light illumination, metal evaporators) is also contemplated.

Responsables Científicos/ Scientific Responsibles: Dr. Juan Pedro Espinós Manzorro y Dr. Juan Pedro Holgado Vázquez
Personal Técnico/Technical Assistant: Dra. Florencia Vattier Lagarrigue

SERVICIO DE MECANIZADO/ MACHINING WORKSHOP

Se trata de un servicio horizontal fundamental para el Instituto y unidades externas adscritas al mismo, ya que permite mejorar, modificar y adecuar el material y equipamiento científico a las necesidades de cada investigador y/o investigación en curso, incluso llegando a su fabricación partiendo de una necesidad concreta. Ofrece asesoramiento técnico, diseño y fabricación de todos los elementos anteriormente descritos.
Además brinda la posibilidad de realizar pequeñas reparaciones y parte del mantenimiento general del equipamiento científico y de laboratorio.

This is a service essential for the Institute and external drives attached to the same. Because it allows you to improve, modify and adapt the material and scientific equipment to the needs of each researcher and/or research in progress. Even going to the extent of their manufacture on the basis of a specific need. Offering technical advice, design and manufacture of all elements described above.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

El servicio cuenta con herramientas manuales y herramientas eléctricas para la conformación de materiales muy diversos. Contando con la posibilidad unión de diversos materiales mediante los procesos de soldeo que se pueden realizar en el servicio:

The service account with hand tools and power tools for the formation of very diverse materials. With the possibility union of various materials by welding processes that can be performed in the service:

- Soldadura fuerte con diferentes aportes
  Brazing with different contributions
- Soldadura por arco eléctrico
  Electric arc welding
- Soldadura TIG sobre aceros
  TIG welding on steel

Para los procesos de mecanizado por arranque de viruta se cuenta con las siguientes máquinas-herramientas:

For the processes of machining by chip is has the following machine-tools:

- Centro de mecanizado,HAAS TM 1P
  HAAS CNC milling machine,TM 1P
- Taladro vertical, ERLO TSAR32
  Vertical drill, ERLO TSAR32

- Torno paralelo convencional PINACHO SC200
  Conventional lathe PINACHO SC200

- Torno paralelo semiautomático PINACHO SMART TURN180
  Semi-automatic lathePINACHO SMART TURN180

- Fresadora de torreta Fortex FTX-4-FC VARIO
  Fortex FTX-4-FC VARIO Milling Machine

Personal Técnico/Technical Assistant: D. Juan Carlos Sánchez Martín, D. Adrián Gómez Castaño

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Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.
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Más información en http://www.uco.es/plasma_laser_superficie

Organizado por:

El Máster Interuniversitario Plasma, Láser y Tecnologías de Superficie es un máster conjunto de la Universidad de Córdoba y la Universidad Politécnica de Madrid, en el que intervienen también investigadores del Instituto de Ciencia de Materiales de Sevilla (Centro mixto CSIC – US) y del Instituto de Ciencia de Materiales de Madrid (CSIC)

Fecha de Celebración: Curso Académico 2019-20
Créditos necesarios: 60
Dirigido a: Titulados en Química, Física, así como, los Titulados en Ingeniería afines (Ingenieros Químicos, de Materiales, etc.)
Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.
Mención de Calidad
https://www.uco.es/organiza/departamentos/fisica/es/novedades/112-master-plasma-laser-y-tecnologias-de-superficie

Profesorado ICMS

Principios Básicos
Interaccion de partículas y radiación con la materia. Láseres (Créditos: 4) | Dra. Asunción Fernández Camacho
Materiales e Ingeniería de Superficies (Créditos: 4) | Dra. Ana Isabel Becerro Nieto | Dr. Alfonso Caballero Martínez | Dr. Agustín R. González-Elipe |Dr. Francisco Yubero Valencia

Tecnologías
Plasmas y Tecnología de superficies (Créditos: 4) | Dr. Alberto Palmero | Dr. José Cotrino Bautista
Tecnologías de lámina delgada (Créditos: 5) | Dra. Ana Isabel Borrás Martos | Dr. Agustín R. González-Elipe | Dra. María del Carmen López Santos
Técnicas de caracterización de superficies y láminas delgadas (Créditos: 5) | Dra. T. Cristina Rojas Ruiz | Dr. Francisco Yubero Valencia

Aplicaciones
Nanotecnología de superficies y sus aplicaciones (Créditos: 4) | Dr. Ángel Barranco Quero | Dr. Juan Ramón Sánchez Valencia (US) | Dr. Francisco Javier Aparicio Rebollo
Nuevos materiales para dispositivos (Créditos: 4) | Dra. Ana Isabel Borrás Martos
Funcionalización de superficies (Créditos: 4) | Dr. Juan Carlos Sánchez-López | Dra. María Carmen López Santos | Dr. Francisco Yubero Valencia

Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid, Instituto de Ciencia de Materiales de Sevilla

Asimismo, el personal del ICMS imparte docencia en titulaciones de Grado y doble Grado de la Universidad de Sevilla. La docencia se desarrolla en diversos centros: Facultad de Física, Facultad de Biología, Facultad de Química, Facultad de Farmacia y Escuela Técnica Superior de Ingeniería Informática.

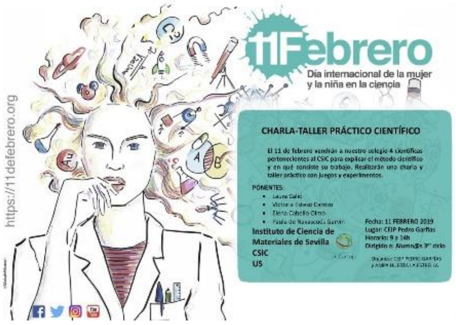
CONFERENCIAS INVITADAS IMPARTIDAS POR PERSONAL DEL ICMS
INVITED CONFERENCES BY PERSONNEL OF THE ICMS

31 marzo | **Designing CO_2 conversion catalysts for gas-phase processes**
Invited Webinar
Tomás Ramírez Reina
Lugar: Loughborough University, UK

23 junio | **Advanced catalysts for low-carbon energy applications**
Invited Webinar
Tomás Ramírez Reina
Lugar: Energy & Bioproducts Research Institute (EBRI) Aston University, UK

DIVULGACIÓN / DISSEMINATION

PARTICIPACION EN EL DÍA INTERNACIONAL DE LA MUJER Y LA NIÑA EN LA CIENCIA/ INTERNATIONAL WOMEN AND GIRL'S DAY IN SCIENCE



El 11 de febrero vendrán a nuestro colegio 4 científicas pertenecientes al CSIC para explicar el método científico y en qué consiste su trabajo. Realizarán una charla y taller práctico con juegos y experimentos.

La ciencia y la igualdad de género son vitales para alcanzar los Objetivos de Desarrollo Sostenible (ODS). Por ello y con el fin de lograr el acceso y la participación plena y equitativa en la ciencia para las mujeres y las niñas, y además para lograr la igualdad de género, la Asamblea General de las Naciones Unidas decidió proclamar en 2016 el 11 de febrero como el Día Internacional de la Mujer y la Niña en la Ciencia. Para la celebración de este día y con el objetivo de dar a conocer el papel de la mujer en la ciencia y fomentar las vocaciones científicas en las niñas, se organizan en España, a través de la plataforma 11F, muchas actividades donde han participado científicos/as y becarios/as del ICMS.

"Mujeres, Luz y Materia". CEIP Pedro Garfias. Ponentes: Laura Caliò, Víctoria Esteso Carrizo. Con la participación de Elena Cabello Olmo, María Morán Pedroso, Clara Bujarance Águilera, Thi Tuyen Ngo.

"Nuevos Materiales, Luz y Color". CEIP Huerta del Carmen. Ponente: Elena Cabello Olmo

"Taller de celdas solares de frutos rojos". CEIP Marie Curie. Ponente: Laura Caliò

"Mujer, Ciencia y Nuevos Materiales". IES Pino Montano. Ponentes: Paula Navascués y Elena Cabello

"Mujer, Ciencia de Materiales y Plasma". IES Federico Mayor Zaragoza. Ponente Paula Navascués

CONCURSO DE NANORELATOS

Este concurso, a nivel de Andalucía, está organizado por el Instituto de Ciencia de Materiales de Sevilla, dentro del marco del VII Festival de la Nanociencia y la Nanotecnología 10alamenos9. El objetivo de este concurso es fomentar en la comunidad educativa el interés por el mundo a escala nanométrica, el denominado nanomundo, que a pesar de su diminuto tamaño es muy diverso e interesante. Hay dos categorías, una para estudiantes de 3 y 4 de la ESO y otra para 1 y 2 bachillerato, dotadas con premios de 200€ y diploma.

Ganadores: María Martínez Rosa, 4° ESO Colegio Santa Ana de Sevilla con su relato "New Blood" (Categoría A) [**https://youtu.be/kTUT7LZCIT4**] y Alonso Yuste Rojas, 2° Bachillerato Colegio San Alberto Magno, Montequinto con su relato "La Nanometa"(Categoría B) [**https://youtu.be/1XU0j--x1DM**]

FESTIVAL DE NANOCIENCÍA Y NANOTECNOLOGÍA 10 A LA MENOS 9 / NANOSCIENCE AND NANOTECHNOLOGY 10 A LA MENOS 9

10ALAMENOS9
Festival de Nanociencia
y Nanotecnología

El VII Festival de Nanociencia, es un festival organizado a nivel nacional, que pretende, de una forma amena, acercar a todos los públicos la escala nanométrica, sus efectos y cómo este conocimiento va a cambiar nuestras vidas a través de innumerables aplicaciones y productos.

NANO-SEMINARIOS

“El gran nanomundo”: Juan Ramón Sánchez Valencia. Seminarios Virtuales con colegios (días 4 y 11 de Mayo 2021) y presencial en la biblioteca municipal de San Jerónimo (12-05-2021)

VERMÚ DE NANOCIENCIA

Up-converting nanophosphor materials based on rare earth elements
Dra. **Thi Tuyen Ngo**
24 de Mayo 2021
https://youtu.be/p20zVvKBJ80

Nanogeneradores: la dinámo para microdispositivos autónomos
Dr. **Francisco Javier Aparicio Rebollo**
25 de Mayo 2021
https://youtu.be/Z5Yc9-Sc7QQ

VI ENCUENTRO NACIONAL DE NANODIVULGACIÓN. I ENCUENTRO IBEROAMERICANO DE NANODIVULGACIÓN

En el marco del VII Festival de la Nanociencia y Nanotecnología, El ICMS organizó una nueva edición del Encuentro Nacional e Iberoamericano de Nanodivulgadores que tuvo lugar el 4 de noviembre. Este encuentro reunió a investigadores y comunicadores tanto del ámbito de la nanotecnología como de la ciencia en general, e incluyó dos sesiones de ponencias en las que se mostraron interesantes iniciativas puestas en marcha en diferentes lugares de España y Latinoamérica. El encuentro tuvo formato mixto presencial/virtual

Programa
15:00-15:10 Presentación/Apertura
15:10-16:15. Sesión I Ponencias de 10 minutos (7 de charla y 3 depreguntas).
Modera: Dra. Rosalía Poyato Galán. Científica y miembro de la comisión de divulgación del ICMS

16:15-16.45 Charla invitada. Francisco R. Villatoro. Profesor de la Universidad de Málaga y divulgador científico

16:45-17:00. Pausa-café
17:00-18:00. Sesión II Ponencias de 10 minutos (7 de charla y 3 de preguntas).
Modera: Dr. Juan Pedro Holgado. Científico y miembro de la comisión de divulgación del ICMS

18.10-18.20 Conclusiones y fin primera parte
18:20-18:30 Pausa-café
18:30-19:30 Consorcio 10alamenos9 Modera: Jordi Diaz –Marcos, Universidad de Barcelona. Divulgador científico

FERIA DE LA CIENCIA / FAIR OF SCIENCE

La 19ª Feria de la Ciencia de Sevilla del 2021, 2º Feria Virtual (del 18 al 21 de mayo de 2021), constituye un punto de encuentro donde se desarrollan actividades de divulgación de la Ciencia y la Tecnología, realizando demostraciones y experimentos para facilitar la comprensión de contenidos científicos.
En esta edición virtual, en el stand del Instituto de Ciencia de Materiales de Sevilla se presentaron: un video institucional, paneles informativos con la investigación que se realiza en las distintas unidades del instituto y una serie de videos "Yo investigo" y "Tú Experimentas" con experimentos demostrativos:

Video presentación del ICMS: Teresa Cristina Rojas

Videos: "**Yo investigo**": Carlos Romero, Clara Bujalance, Elena Cabello, María Morán, Javier Castillo, Xavier García, Paula Navascues, Encarnación Arroyo, Antonio Márquez, Ana Mª Gómez, Manuel Oliva, T. Cristina Rojas, Gerardo Colón, Carmen Muñoz

Videos: "**Tú experimentas**":
Fabricación de una celda solar de colorante: Mauricio Calvo, Laura Caliò, Carlos Romero
Superficies Autolimpiables: Mª Carmen Hidalgo

The Fair of Science (18 to 21 May 2021, in Seville) constitutes a meeting point where many activities for spreading of science and technology were carried out. Demonstrations and experiments where presented to facilitate the understanding of scientific aspects.
In this year, a virtual edition was done, were the ICMS presented several videos and panels.

Participacion en la noche europea / European Researchers’ Night

LA NOCHE EUROPEA
DE L@S INVESTIGADOR@S
MUJERES Y HOMBRES QUE HACEN CIENCIA PARA TI

La Noche Europea de los Investigadores, celebrada el Sevilla 24 septiembre de 2021, es un proyecto de divulgación científica enmarcado en Horizonte 2020, bajo las acciones Marie Skłodowska-Curie. Su principal objetivo es acercar los investigadores a los ciudadanos para que conozcan su trabajo, los beneficios que aportan a la sociedad y su repercusión en la vida cotidiana. Se celebra simultáneamente en 371 ciudades europeas desde 2005.

El ICMS ha participado con las siguientes actividades:

Charla:
Nanogeneradores. Captando energía de nuestros pasos para alimentar nuestros dispositivos inteligentes portátiles. Francisco Javier Aparicio Rebollo

Talleres:
Hidrofobicidad o cómo evitar que tus gafas se empañen por culpa de la mascarilla. Grupo de Nanotecnología de superficies y plasma

Triboelectricidad, o cómo aprovechar la energía del rozamiento. Grupo de Nanotecnología de superficies y plasma

Charlas-talleres:

Plasma, la tecnología ecológica. Ana Gómez, Manuel Oliva

Diamante, grafito y grafeno, ¿Son familia?. Rosalía Poyato

Participacion en el Salon del Estudiante de grado y postgrado y ferisport 2021

10 al 14 de Mayo 2021. El ICMS ha participado en un stand virtual dando a conocer su investigacion mediante una serie de videos realizados por miembros de distintos grupos de investigación

María Morán Pedroso. Video Divulgativo “Mi investigación”
Carlos Romero Pérez. Video Divulgativo “Mi investigación”

Semana de la Ciencia y la Tecnología / Science and Technology Week

La semana de la Ciencia (celebrada del 8 al 11 de noviembre de 2021) es un evento de carácter europeo diseñado para demostrar cómo la ciencia y la tecnología nos afectan y cómo éstas pueden ser utilizadas para mejorar nuestras vidas y el mundo que nos rodea.

Café con Ciencia

El café con Ciencia acerca de forma original y atractiva la ciencia y sus protagonistas. Esta actividad de divulgación genera un punto de contacto entre profesionales de la ciencia y alumnos de secundaria, promoviendo la reflexión sobre diversos asuntos en un entorno cercano y participativo.

En esta edición se compartió un desayuno virtual con estudiantes de bachillerato para dialogar sobre temas concretos de sus estudios y sobre sus respectivas trayectorias profesionales. Esta actividad cumple el triple objetivo de comunicar la ciencia a través de sus propios protagonistas, promover la cultura científica y fomentar vocaciones investigadoras.

Las mesas de encuentro tienen una hora de duración, y se desarrollan con grupos reducidos de quince alumnos.

Mesa. Nanogeneradores. Captando energía de nuestros pasos para alimentar nuestros dispositivos inteligentes portátiles. Francisco Javier Aparicio Rebollo

MATERLAND / MATERLAND

Participantes

El Dr. Mauricio Calvo participó en el grupo de trabajo de divulgación del proyecto Materland (http://materland.sociemat.es/) El grupo de trabajo es un grupo multidisciplinar formado por 100 personas que cubren diferentes ámbitos de la Ciencia e Ingeniería de Materiales y con titulaciones en física, química, biología, ingeniería de materiales, mecánica, industrial, arquitectura, etc. de centros de investigación, universidades e institutos de secundaria

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