ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo Superior de Investigaciones Científicas

Universidad de Sevilla

Junta de Andalucía

2019

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2019

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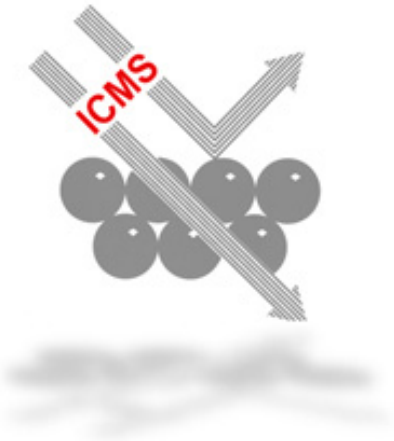

EDITA

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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DISEÑO Y MAQUETACIÓN
José Carlos Rivero Cabello

COMISIÓN MEMORIA-ICMS
Gerardo Colón Ibáñez - Francisco José Gotor Martínez - Hernán R. Míguez García

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El ICMS en 2019
ICMS in 2019

Presentación Presentation

A través de esta Memoria 2019, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo de 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 2019, contaba con 91 empleados, de los cuales 47 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 H2 y otros combustibles limpios y de origen renovable, el aprovechamiento y conversión química del CO2, 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.

Through this Activity Report 2019, 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 Andalucia. 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 91 people, 47 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 H2 and other clean and renewable fuels, the exploitation and chemical conversion of CO2, 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.

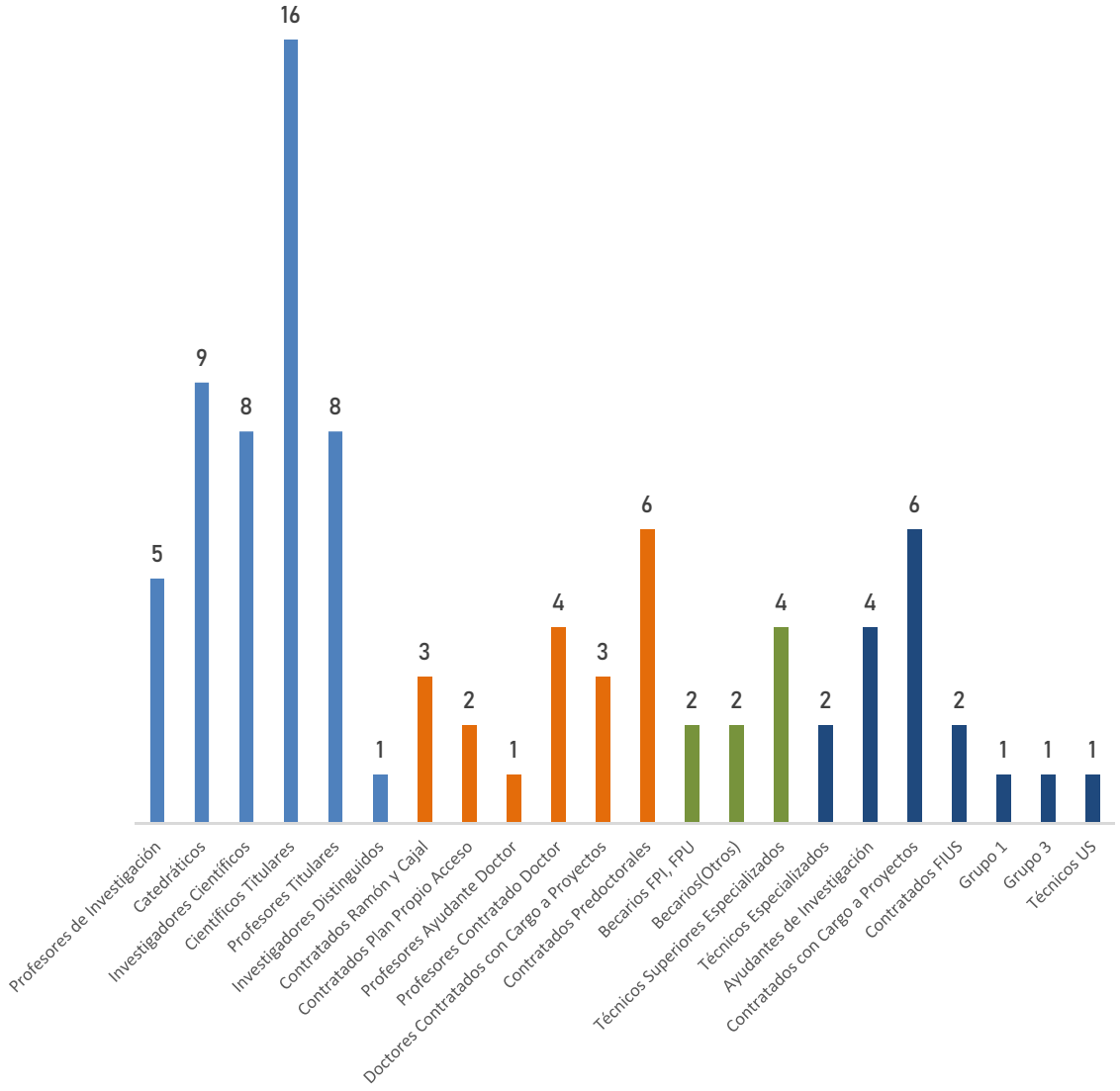
Our main efforts for near future are to cover the most modern and innovative aspects of the current Material Science and Material Engineering, as well as to support in the region of Andalusia, the development of a new economic system, in which scientific and technological basis should become fundamental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

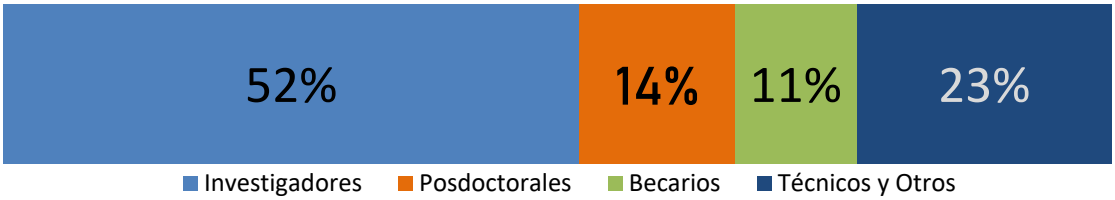
Dr. Juan Pedro Espinós Manzorro
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS
Statistical Data of ICMS

RECURSOS HUMANOS / HUMAN RESOURCES

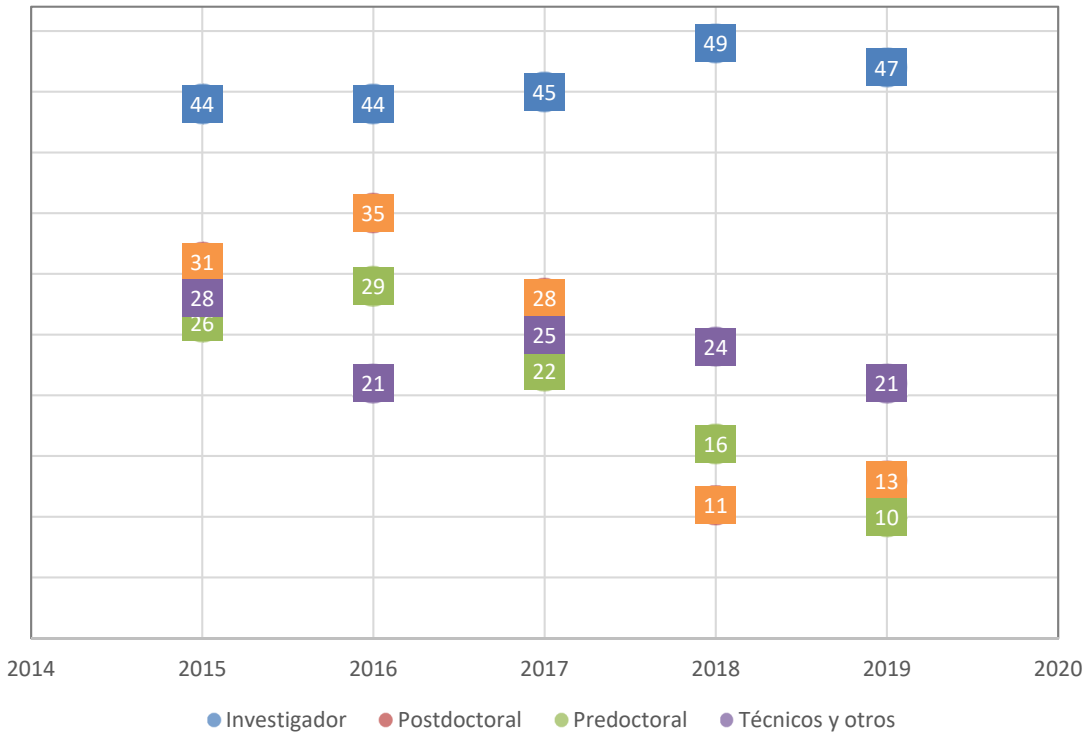
Distribución del personal por categorías – 2019*
Distribution by professional category – 2019



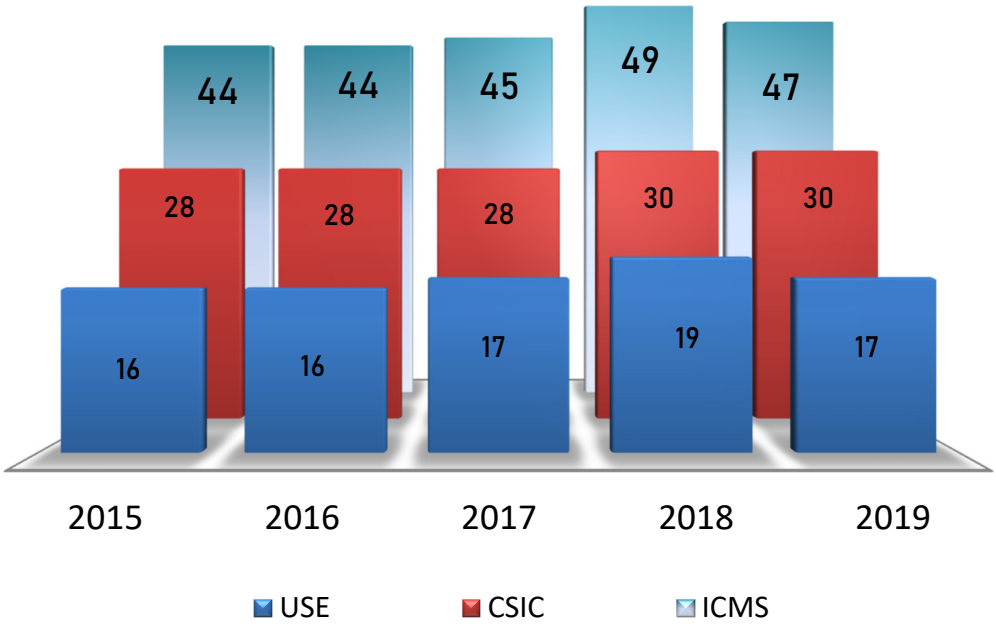


* Datos a 31 de Diciembre de 2019

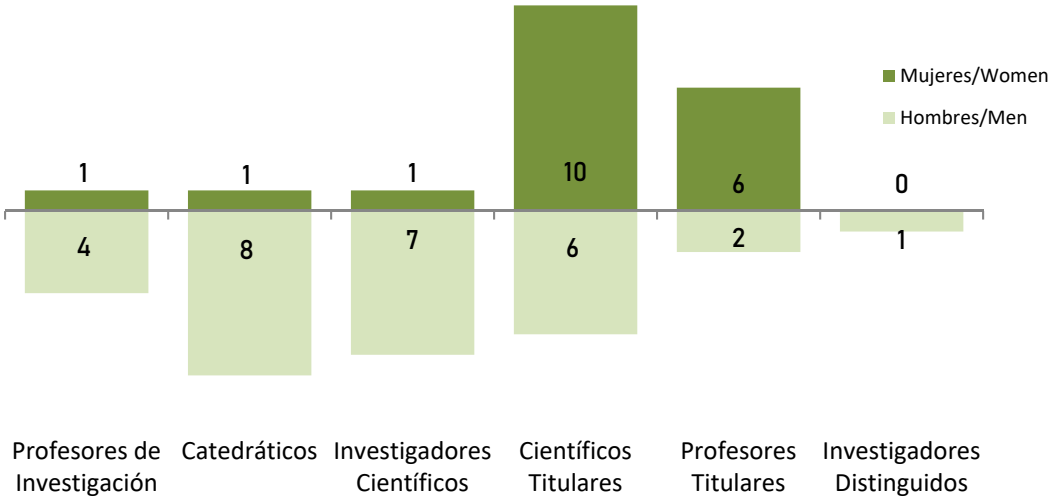
Evolución 2015-2019 del personal
Evolution of Staff

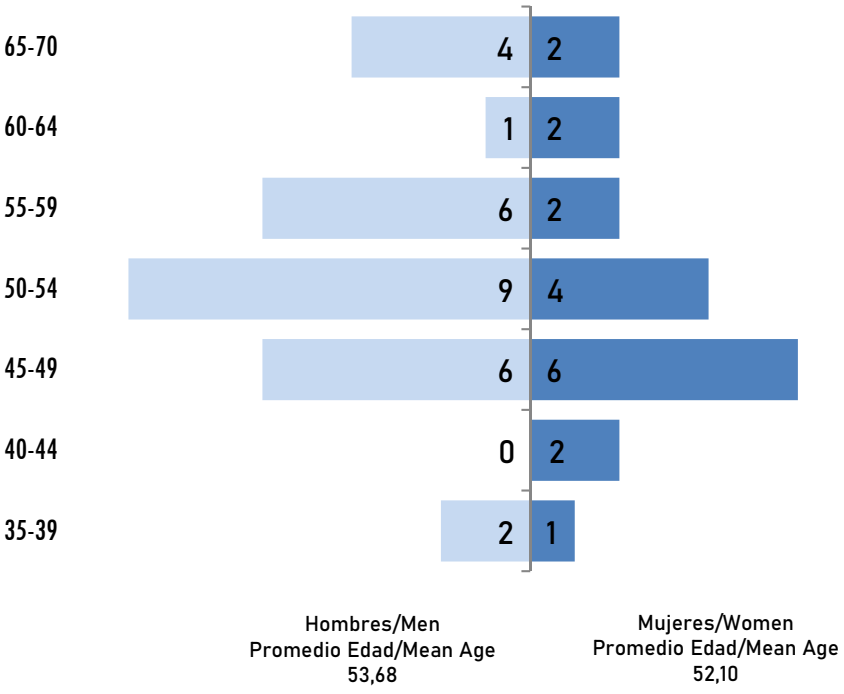


Evolución 2015-2019 del personal investigador
Evolution of Research Staff



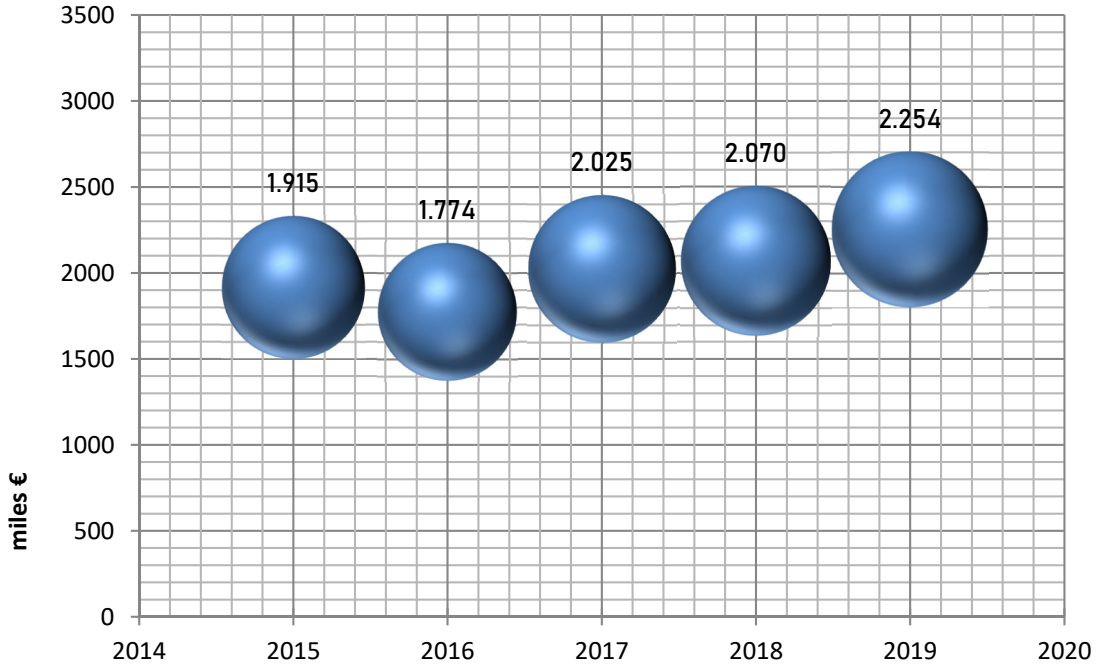
Distribución del personal Investigador (sexo y edad)
Distribution by Scientific staff (gender and age)





FINANCIACIÓN / FUNDING

Evolución 2015-2019 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
ADVANCED FUNCTIONAL MATERIALS	1	16,836	Q1
APPLIED CATALYSIS B-ENVIRONMENTAL	2	16,683	Q1
NANO ENERGY	1	16,602	Q1
ACS CATALYSIS	2	12,350	Q1
RENEWABLE & SUSTAINABLE ENERGY REVIEWS	1	12,110	Q1
CHEMICAL ENGINEERING JOURNAL	2	10,652	Q1
CHEMISTRY OF MATERIALS	1	9,567	Q1
APPLIED ENERGY	1	8,848	Q1
ACS APPLIED MATERIALS & INTERFACES	3	8,758	Q1
JOURNAL OF POWER SOURCES	1	8,247	Q1
JOURNAL OF COLLOID AND INTERFACE SCIENCE	1	7,489	Q1
SENSORS AND ACTUATORS B-CHEMICAL	1	7,100	Q1
JOURNAL OF MATERIALS CHEMISTRY C	1	7,059	Q1
ACS PHOTONICS	1	6,864	Q1
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	1	6,710	Q1
MATERIALS & DESIGN	1	6,289	Q1
RENEWABLE ENERGY	1	6,274	Q1
ELECTROCHIMICA ACTA	1	6,215	Q1
APPLIED SURFACE SCIENCE	6	6,182	Q1
CATALYSIS TODAY	4	5,825	Q1
FUEL	1	5,578	Q1
JOURNAL OF MATERIALS CHEMISTRY B	1	5,344	Q1
JOURNAL OF MATERIALES RESEARCH AND TECHNOLOGY-JMR&T	1	5,289	Q1
SCRIPTA MATERIALIA	1	5,079	Q1
ADVANCED MATERIALS INTERFACES	2	4,948	Q1
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	1	4,939	Q2
CHEMISTRY-A EUROPEAN JOURNAL	1	4,939	Q1
CHEMCATCHEM	3	4,853	Q2

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT
INORGANIC CHEMISTRY	1	4,825	Q1
JOURNAL OF ALLOYS AND COMPOUNDS	6	4,650	Q1
APPLIED CLAY SCIENCE	2	4,605	Q1
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	3	4,495	Q1
ACS APPLIED ENERGY MATERIALS	1	4,473	Q2
CONSTRUCTION AND BUILDING MATERIALS	1	4,419	Q1
NANOMATERIALS	5	4,324	Q2
JOURNAL OF ENVIRONMENTAL CHEMICAL ENGINEERING	1	4,300	Q1
TRIBOLOGY INTERNATIONAL	1	4,271	Q1
TOXICOLOGY	1	4,099	Q1
SCIENTIFIC REPORTS	3	3,998	Q1
COLLOIDS AND SURFACES A-PHYSICOCHEMICAL AND ENGINEERING ASPECTS	1	3,990	Q2
CERAMICS INTERNATIONAL	5	3,830	Q1
SURFACES AND INTERFACES	1	3,724	Q1
FRONTIERS IN CHEMISTRY	4	3,693	Q2
LANGMUIR	4	3,557	Q2
FRONTIERS OF CHEMICAL SCIENCE AND ENGINEERING	1	3,552	Q2
CATALYSTS	5	3,520	Q2
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	3	3,502	Q1
MATERIALS CHEMISTRY AND PHYSICS	1	3,408	Q2
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	1	3,407	Q1
MATERIALS RESEARCH BULLETIN	1	3,355	Q2
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	2	3,306	Q2
JOURNAL OF LUMINESCENCE	1	3,280	Q1
MOLECULES	1	3,267	Q2
SPECTROCIMICA ACTA PART A-MOLECULAR AND BIOMOLECULAR SPECTROSCOPY	1	3,232	Q1
PLASMA SOURCES SCIENCE & TECHNOLOGY	1	3,193	Q1
RSC ADVANCES	1	3,119	Q2
PLASMA PROCESSES AND POLYMERS	1	3,065	Q1
ENVIRONMENTAL SCIENCE AND POLLUTION RESEARCH	1	3,056	Q2
JOURNAL OF NON-CRYSTALLINE SOLIDS	1	2,929	Q1

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT
ACS OMEGA	2	2,870	Q2
JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART B-APPLIED BIOMATERIALS	1	2,831	Q2
METHODS AND APPLICATIONS IN FLUORESCENCE	1	2,800	Q2
PLOS ONE	2	2,740	Q2
JOURNAL OF SOLID STATE CHEMISTRY	1	2,726	Q1
TRANSACTIONS OF NONFERROUS METALS SOCIETY OF CHINA	1	2,615	Q1
COATINGS	1	2,436	Q2
JOURNAL OF ORGANOMETALLIC CHEMISTRY	1	2,304	Q2
RESEARCH ON CHEMICAL INTERMEDIATES	1	2,262	Q3
COMPTES RENDUS CHIMIE	1	2,223	Q3
JOURNAL OF MATERIALS SCIENCE-MATERIALS IN ELECTRONICS	1	2,220	Q2
METALS	1	2,117	Q1
THIN SOLID FILMS	1	2,030	Q3
JOURNAL OF NANOMATERIALS	1	1,980	Q3
ARCHAEOLOGICAL AND ANTHROPOLOGICAL SCIENCES	1	1,978	Q1
JOURNAL OF CULTURAL HERITAGE	1	1,955	Q3
MATERIALS RESEARCH EXPRESS	4	1,929	Q3
SOLID STATE NUCLEAR MAGNETIC RESONANCE	1	1,846	Q3
INTERNATIONAL JOURNAL OF QUANTUM CHEMISTRY	1	1,747	Q2
ARABIAN JOURNAL FOR SCIENCE AND ENGINEERING	1	1,711	Q3
SURFACE AND INTERFACE ANALYSIS	1	1,665	Q4
REVIEW OF PALAEOBOTANY AND PALYNOLOGY	1	1,425	Q3
REVISTA DE METALURGIA	1	0,878	Q3
INTERNATIONAL JOURNAL OF MATERIALS RESEARCH	1	0,653	Q2
Total	**135**	**4,808**	

(*) Factor de Impacto correspondiente al año 2019
Journal Citation Reports of 2019

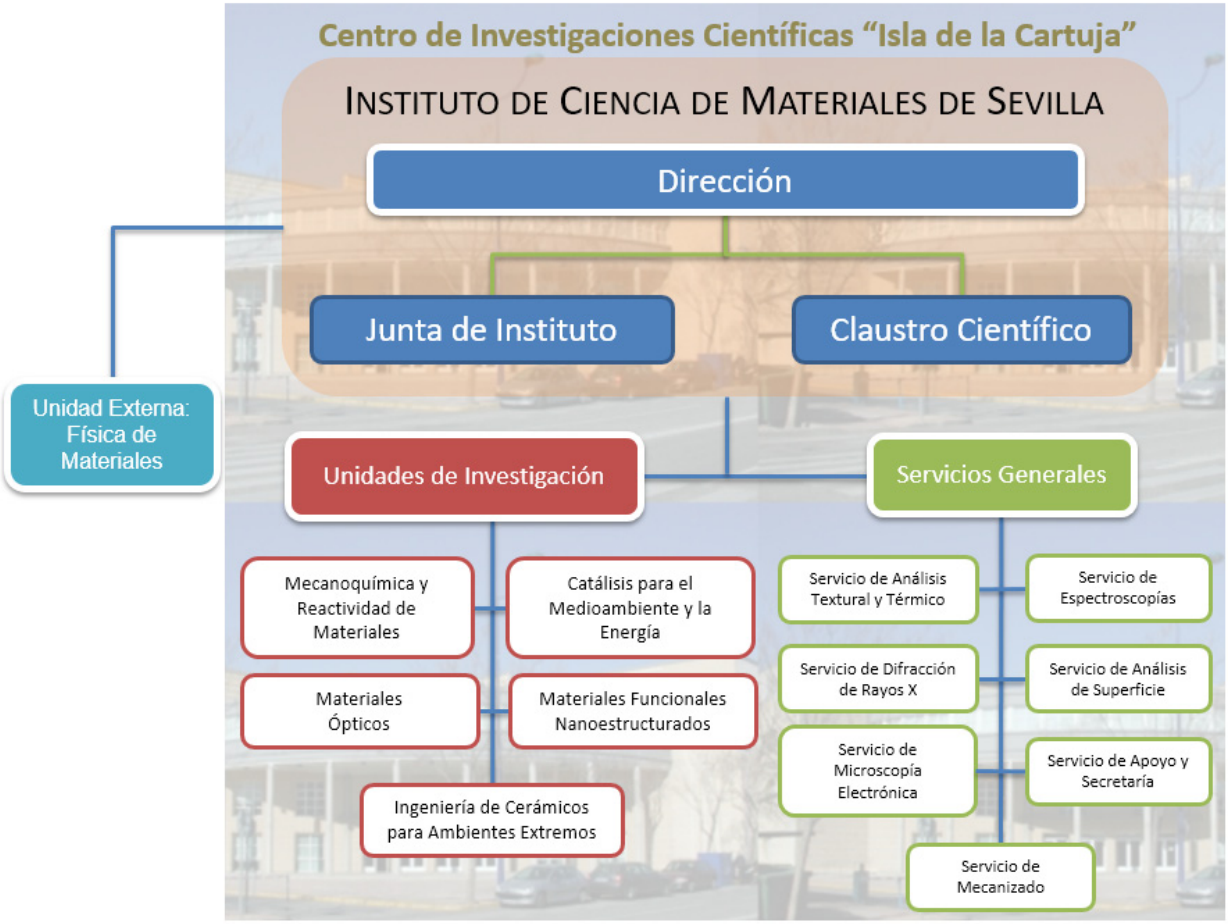
Composición y Estructura
Structure and Organisation

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 Andalucia -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 en 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 Andalucia - 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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Personal Contratado

Ldo. Dirk Hufschmidt

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Gdo. Carlos Romero Pérez
Ldo. Andrea Rubino
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Dr. Victor J. Rico Gavira

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PHYSICS OF MATERIALS (EXTERNAL UNIT)

Catedrático

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Profesor Emérito

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Investigador honorario

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Dr. Justo Jiménez

Unidades de Investigación
Research Units

CATÁLISIS PARA EL MEDIOAMBIENTE Y LA ENERGÍA
CATALYSIS FOR ENVIRONMENT AND ENERGY

GRUPOS DE INVESTIGACIÓN

Fotocatálisis Heterogénea: Aplicaciones
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

PERSONAL / PERSONNEL

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Dr. José Antonio Navío Santos
Dr. José Antonio Odriozola Gordón

Investigadores Científicos

Dr. Miguel Angel Centeno Gallego
Dr. Gerardo Colón Ibáñez

Científicos Titulares

Dra. M. Carmen Hidalgo López
Dr. Juan Pedro Holgado Vázquez

Profesores Titulares

Dra. María Isabel Domínguez Leal
Dra. Svetlana Lyubomirova Ivanova
Dra. Anna Dimitrova Penkova
Dra. Francisca Romero Sarria

Investigadores Contratados

Dr. Luis Bobadilla Baladrón

Profesores Contratado Doctor

Dra. Leidy Marcela Martínez Tejada
Dra. Rosa Pereñíguez Rodríguez

Becarios Predoctorales

Ldo. Lola de las Aguas Azancot Luque
Ldo. Felipe Rubén Puga Martínez
Ldo. José Luis Santos Muñoz (hasta abril)

Personal Contratado

Lda. Laura Blandón Évora (hasta julio)
Ldo. Gabriel Delgado Martín
Lda. Ángeles María López Martín
Ldo. Juan Carlos Navarro
Ldo. Francisco Jesús Platero Moreno

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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)

Código/Code:	RTI2018-096294-B-C33
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
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 Unions 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, SO_x, 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.

Desarrollo de nuevos materiales nanoestructurados para la valorización de metano a hidrógeno y olefinas C2-C4 Development of new nanostructured materials for methane valorization to C2-C4 olefins

Código/Code:	ENE2017-82451-C3-3-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	205.700 €
Investigador responsable/Research head:	Alfonso Caballero Martínez y Gerardo Colón Ibáñez
Componentes/Research group:	Rosa María Pereñiguez Rodríguez, Francisco Jesús Platero Moreno, Angeles María López Martín, Juan Pedro Holgado Vázquez

RESUMEN / ABSTRACT

El desarrollo de nuevos materiales con propiedades singulares en distintos campos de aplicación se ha convertido en las últimas décadas en una prioridad en multitud de áreas de la ciencia y la tecnología. Entre ellas, además de materiales micro y mesoporosos de composición variable, pueden destacarse los sólidos basados en estructura perovskita ABO_3. La versatilidad que presentan estos últimos mediante la sustitución parcial en las posiciones A y B por distintos metales alcalinos, alcalino-terreos y de transición los convierte en una alternativa interesante, y de hecho tienen aplicaciones en campos relacionados con sus propiedades electricas, ópticas, térmicas, catalíticas y como adsorbentes. En el presente proyecto coordinado se plantea la preparación de un conjunto de materiales, entre ellos algunos con estructura perovskita (Fe, Co, Mn, Cu y Bi en posiciones B; Ca, Mg, Ce y La en posiciones A), y estudiar su aplicación en distintos procesos de catálisis heterogénea y de adsorción de contaminantes. 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. De esta forma, y combinando los metales en posiciones A y B para que actuen tanto como agentes promotores como precursores de aleaciones metálicas en los sistemas reducidos, se obtendrán sistemas con propiedades muy variadas y versatiles. Asi, en el subproyecto 1 se estudiarán sus propiedades catalíticas en procesos de enorme interés para la valorización de metano, principal componente del gas natural y una de las fuentes de energía más abundantes en la actualidad. En concreto, y junto con sistemas soportados en materiales mesoporosos y otros, se estudiará en primer lugar la actividad de perovskitas de niquel para la reacción de reformado seco de metano con el fin de obtener gas de síntesis. El objetivo será obtener sistemas activos y sobre estables frente a los fenómenos de desactivación habituales por deposición de coque. En segundo lugar, se estudiarán sistemas basados principalmente en Fe y Co para la reacción de Fisher-Tropsch a olefinas C2-C4, productos de gran interés económico por ser precursores de una gran cantidad de otros productos de alto valor añadido. Por otro lado, los trabajos propuestos en el subproyecto 2 están relacionados con la aplicación de estos sólidos de estructura perovskita para el desarrollo de procesos de eliminación de contaminantes emergentes, un nuevo tipo de desechos que suelen ser resistentes a los procesos de degradación biológico convencionales, constituyendo por tanto un problema medioambiental de primer orden. En concreto, el proyecto pretende desarrollar tratamientos integrados de depuración (adsorción-POA), utilizando perovskitas para la eliminación de contaminantes emergentes (Ibuprofeno, Salicílico, Ciprofloxacina, Cafeína, Gemfibrozil y Benzafibrato), optimizando parámetros como el rendimiento, la reciclabilidad de los catalizadores, la aplicabilidad y la sostenibilidad. De especial relevancia en este tipo de procesos es la utilización de procesos de fotocatálisis heterogénea, por lo que el desarrollo de nuevos óxidos semiconductores como las perovskitas, con características fisicoquímicas y estructurales superiores a las del TiO_2, es un objetivo de primer orden del presente subproyecto.

In the present project the preparation of a set of materials, including some with perovskite structure (Fe, Co, Mn, Cu and Bi in positions B; Ca, Mg, Ce and La in positions A), and the study of its application in different processes of heterogeneous catalysis and adsorption of pollutants has been proposed. For this purpose, a number of recently described preparation techniques will be used to obtain high surface specific and controlled nanostructure systems. In this way, and combining the metals in positions A and B to act both as promoters and precursors of metal alloys in the reduced systems, systems with very varied and versatile properties will be obtained.

Thus, we will study its catalytic properties in processes of great interest for the valorization of methane, the main component of natural gas and one of the most abundant energy sources today. In particular, and together with systems supported on mesoporous materials and others, the activity of nickel perovskites for the dry methane reforming reaction will be studied first in order to obtain synthesis gas. The aim will be to obtain active and above all stable systems in the face of the usual deactivation phenomena by deposition of coke. Secondly, systems based mainly on Fe and Co for the Fisher-Tropsch reaction to C2-C4 olefins will be studied, products of great economic interest as precursors to a large number of other high added value products.

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 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Miguel Angel 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 generatre high added value products by bio-refinery processes, as alternative to currently obtained chemicals from fossil sources. A set of carbonaceous solids with tunned 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 the proposed proceses. 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.

Diseño racional de fotocatalizadores altamente eficientes mediante control nivel atómico Rational design of highly effective photocatalysts with atomic-level control

Código/Code:	RATOCAT PCIN-2017-056
Periodo/Period:	02-10-2017 / 01-10-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad. Unión Europea
Importe total/Total amount:	97.000 €
Investigador responsable/Research head:	Gerardo Colón Ibañez
Componentes/Research group:	Alfonso Caballero Martínez, Angeles Martín

RESUMEN / ABSTRACT

El uso de la energía solar para la generación de hidrógeno a partir de agua es probablemente uno de procesos más limpios y sostenibles para la obtención de energía. Sin embargo, los catalizadores que dan mejores rendimientos son demasiado caros para ser económicamente viables. El proyecto RATOCAT tiene como objetivo el desarrollo de materiales fotocatalíticos optimizados. De esta forma las prestaciones fotocatalíticas de sistemas basados en TiO_2 y gC_3N_4 podrían optimizarse mediante el diseño de su superficie con nanoestructuras de composición, nanoarquitectura, tamaño y estado químico altamente controladas. Se empleará para ello estudios de simulación teórica para proponer la nanoestructuras óptimas que serán depositadas de forma controlada y precisa mediante atomic layer deposition (ALD). Los test de actividad fotocatalítica tanto a escala de laboratorio como en planta piloto (Plataforma Solar de Almería).

Using the sun's energy to generate hydrogen from water is probably the cleanest and most sustainable source of fuel that we can envisage. Unfortunately, catalysts that do this are currently too expensive to be commercially viable. The RATOCAT project aims to develop improved photocatalyst materials, along with the processes for their production. The catalytic performance of cheap TiO_2 and C_3N_4 powders will be improved by tailoring their surface with nanostructured oxides as co-catalysts of highly-controlled composition, nanoarchitecture, size and chemical state. First principles simulations will be used to design the optimum nanostructures, which will then be deposited onto powders with the required precision using atomic layer deposition, again supported by simulation. Lab-scale tests of photocatalytic activity will provide feedback for the optimisation of the material and process, before the most promising materials are tested in the field on both pure water and wastewater.

OTROS PROYECTOS / OTHER PROJECTS

Biorefinería: Aprovechamiento de biomasa residual para la obtención de biocombustibles y compuestos de alto valor añadido

Código/Code:	EQC2019-005458-P
Periodo/Period:	2019
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades. Ayudas para la adquisición de equipamiento científico-técnico del subprograma estatal de infraestructuras de investigación y equipamiento científico-técnico (Plan estatal I+D+I 2017-2020)
Importe total/Total amount:	196.856 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón

Procesos Multidisciplinares para CCUS. Aspectos químicos, físicos, análisis tecno-económico e ingeniería. / Multidisciplinary processes involving chemistry, physics, techno-economic analysis and engineering for CCUS

Código/Code:	PRX19/00224
Periodo/Period:	1-7-2019 a 30-9-2019
Organismo Financiador/Financial source:	Estancias de Profesores e Investigadores Senior en Centros Extranjeros, incluido El Programa «SALVADOR DE MADARIAGA»
Investigador responsable/Research head:	José Antonio Odriozola Gordón

UNIDAD ASOCIADA / ASSOCIATED UNIT

Grupo de Fotocatálisis y Electroquímica Aplicada al MedioAmbiente
Laboratory of Photocatalysis and Electrochemistry Applied to the Environment

La Junta de Gobierno del CSIC, en su reunión de fecha 22 de diciembre de 2004, aprobó la propuesta de reconocimiento de la Unidad Asociada denominada "Grupo de Fotocatálisis y Electroquímica aplicada al Medio Ambiente" de la Universidad de Palmas de Gran Canaria (ULPGC) a través del Instituto de Ciencia de Materiales de Sevilla, con el Grupo de investigación del Profesor Dr. José Antonio Navío Santos. Actualmente, el responsable de esta Unidad Asociada, por parte de la ULPGC es el Prof. Dr. Óscar Manuel González Díaz y por parte del CSIC la Dra. M. Carmen Hidalgo.

Las líneas principales de actuación son:

"Fotocatálisis en procesos ambientales"

"Espectro-electroquímica aplicada al medio ambiente"

"Materiales para tratamientos de aguas residuales y de gases contaminados"

Entre las acciones propuestas destacar los objetivos de preparación de nuevos materiales fotocatalizadores, así como la caracterización de los mismos y su estudio en aplicaciones de fotocatálisis solar para descontaminación y tratamiento de aguas residuales y de gases.

The CSIC's Board in its meeting from the 22nd December 2004, approved the proposal to recognize the Associated Unit titled "Laboratory of Photocatalysis and Elec-trochemistry Applied to the Environment", of the University of Las Palmas de Gran Canaria (ULPGC) through the Institute of Materials Science of Seville.

The actual person in charge of this Associated Unit is Prof. Dr. Óscar Manuel González Díaz, from ULPGC and Dr. M. Carmen Hidalgo from the CSIC.

The main research lines are:
"Photocatalysis for environmental processes"
"Spectroscopical-electrochemistry applied to environment"
"Materials for the treatment of waste water and polluted gases"

Among the proposed actions, it may be emphasized the objectives of preparation of new photocatalysts materials, their characterization and the study of their applications in solar photocatalysis and treatment of water.

PATENTES / PATENTS

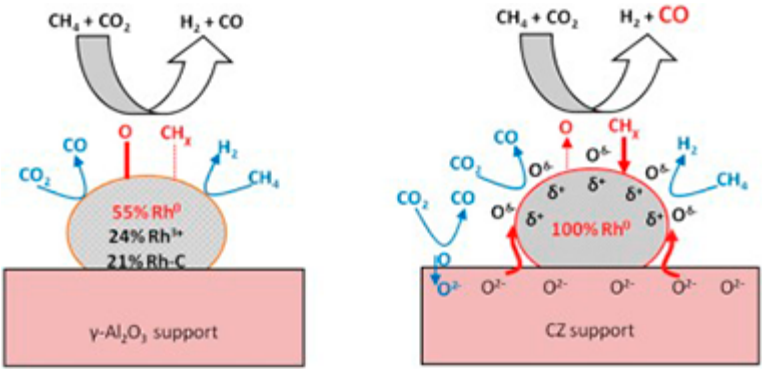
Procedimiento de obtención de monolitos integrales de carbono
Inventores: Miguel Angel Centeno Gallego, José Antonio Odriozola Gordón, Nicolás Rodríguez Riaño, José Luis Santos Muñoz
Tipo de Patente: Nacional
Número de Solicitud: 201931067
Fecha Solicitud: 2 de diciembre de 2019
Entidad Titular: Universidad de Sevilla, Universidad de Colombia

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Effect of support oxygen storage capacity on the catalytic performance of Rh nanoparticles for CO_2 reforming of methane (14,229 – D1)

Yentekakis, IV, Goula, G, Hatzisymeon, M, Betsi-Argyropoulou, I, Botzolaki, G, Kousi, K, Kondarides, DI, Taylor, MJ, Parlett, CMA, Osatiashtiani, A, Kyriakou, G, Holgado, JP, Lambert, RM

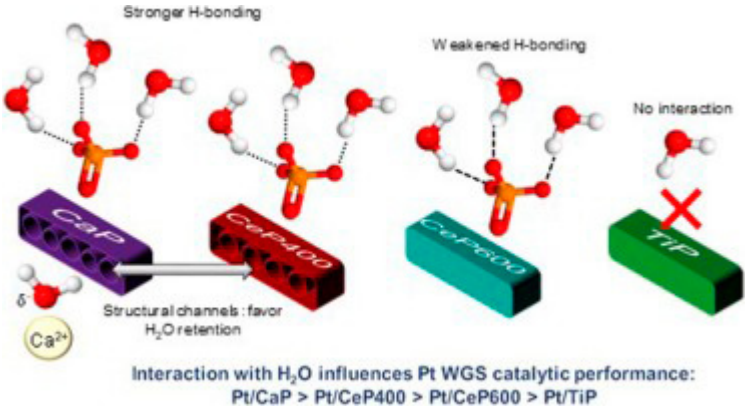
Applied Catalysis B: Enviromental, **243** (2019) 490-501
Abril, 2019 | **DOI:** 10.1016/j.apcatb.2018.10.048



The effects of the metal oxide support on the activity, selectivity, resistance to carbon deposition and high temperature oxidative aging on the Rh-catalyzed dry reforming of methane (DRM) were investigated. Three Rh catalysts supported on oxides characterized by very different oxygen storage capacities and labilities (gamma-Al_2O_3, alumina-ceria-zirconia (ACZ) and ceria-zirconia (CZ)) were studied in the temperature interval 400-750 degrees C under both integral and differential reaction conditions. ACZ and CZ promoted CO_2 conversion, yielding CO enriched synthesis gas. Detailed characterization of these materials, including state of the art XPS measurements obtained via sample transfer between reaction cell and spectrometer chamber, provided clear insight into the factors that determine catalytic performance. The principal Rh species detected by post reaction XPS was Rh, its relative content decreasing in the order Rh/CZ(100%) > Rh/ACZ(72%) > Fth/gamma Al_2O_3(55%). The catalytic activity followed the same order, demonstrating unambiguously that Rh is indeed the key active site. Moreover, the presence of CZ in the support served to maintain Rh in the metallic state and minimize carbon deposition under reaction conditions. Carbon deposition, low in all cases, increased in the order Rh/CZ < Rh/ACZ < Rh/gamma-Al_2O_3 consistent with a bi-functional reaction mechanism whereby backspillover of labile lattice O_2- contributes to carbon oxidation, stabilization of Rh and modification of its surface chemistry; the resulting O vacancies in the support providing centers for dissociative adsorption of CO2. The lower apparent activation energy observed with CZ-containing samples suggests that CZ is a promising support component for use in low temperature DRM.

Phosphate-type supports for the design of WGS catalysts

Navarro-Jaen, S, Romero-Sarria, F, Centeno, MA, Laguna, OH, Odriozola, JA
Applied Catalysis B: Environmental, **244** (2019) 853-862
Mayo, 2019 | **DOI:** 10.1016/j.apcatb.2018.12.022



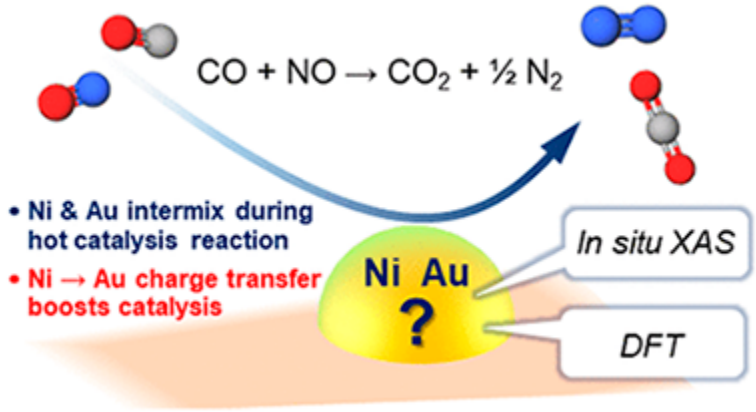
The importance of water availability during the WGS reaction has been extensively reported. Thus, the search of new supports able to interact with the water molecule is of great importance. In this work, a series of phosphate type supports containing Ce, Ca and Ti have been studied, demonstrating that water interaction with the support is closely related to the textural properties, surface composition and crystal structure of the solids. Additionally, DRIFTS results showed that different interaction mechanisms with the water molecule occur depending on the support. The system containing Ca dissociates the water molecule and interacts with it via the phosphate and Ca2+ ions. However, the Ce systems retain water in its molecular form, which interacts with the solids via hydrogen bonding with the phosphate groups. On the other hand, the Ti system experiences a loss of phosphorous, presenting a low degree of interaction with the water molecule. Additionally, the behavior of the supports with water has been successfully related to the WGS catalytic activity of the corresponding phosphate supported Pt catalysts.

Comprehensive Experimental and Theoretical Study of the CO plus NO Reaction Catalyzed by Au/Ni Nanoparticles

Kyriakou, G, Marquez, AM, Holgado, JP, Taylor, MJ, Wheatley, AEH, Mehta, JP, Sanz, JF, Beaumont, SK, Lambert, RM
ACS Catalysis, **9** (2019) 4919-4929
Junio, 2019 | **DOI:** 10.1021/acscatal.8b05154

The catalytic and structural properties of five different nanoparticle catalysts with varying Au/Ni composition were studied by six different methods, including in situ X-ray absorption spectroscopy and density functional theory (DFT) calculations. The as-prepared materials contained substantial amounts of residual capping agent arising from the commonly used synthetic procedure. Thorough removal of this material by oxidation was essential for the acquisition of valid catalytic data. All catalysts were highly selective toward N-2 formation, with 50-50 Au:Ni material being best of all. In situ X-ray absorption near edge structure spectroscopy showed that although Au acted to moderate the oxidation state of Ni, there was no clear correlation between catalytic activity and nickel oxidation state. However, in situ extended X-

ray absorption fine structure spectroscopy showed a good correlation between Au Ni coordination number (highest for Ni50Au50) and catalytic activity. Importantly, these measurements also demonstrated substantial and reversible Au/Ni intermixing as a function of temperature between 550 degrees C (reaction temperature) and 150 degrees C, underlining the importance of in situ methods to the correct interpretation of reaction data. DFT calculations on smooth, stepped, monometallic and bimetallic surfaces showed that N + N recombination rather



than NO dissociation was always rate-determining and that the activation barrier to recombination reaction decreased with increased Au content, thus accounting for the experimental observations. Across the entire composition range, the oxidation state of Ni did not correlate with activity, in disagreement with earlier work, and theory showed that NiO itself should be catalytically inert. Au-Ni interactions were of paramount importance in promoting N + N recombination, the rate-limiting step.

Does shaping catalysts modify active phase sites? A comprehensive in situ FTIR spectroscopic study on the performance of a model Ru/Al_2O_3 catalyst for the CO methanation

Bobadilla, LF, Munoz-Murillo, A, Laguna, OH, Centeno, MA, Odriozola, JA
Chemical Engineering Journal, **335** (2019) 248-257
Febrero, 2019 | **DOI:** 10.1016/j.cej.2018.09.166

Routinely, it seems assumed that the catalytic layer coated on monoliths and microchannel reactors preserve the properties of the initial powder catalyst. However, this assumption should be reasonably demonstrated since the set of chemical and physical manipulations involved in the preparation of these catalytic devices hardly does not alter the surface of the starting catalyst powders. This work aims to evaluate the transformations that takes place in a model Ru/Al_2O_3 catalyst during a typical slurry preparation procedure and their impact on the catalytic performance for the CO methanation reaction and the selective methanation of CO in CO_2-rich reformate gases. For this purpose, we have conducted an in situ comprehensive study by means of Fourier Transform Infrared Spectroscopy (FTIR) in which the nature of the species present on the surface of the catalyst during CO hydrogenation was analyzed. This study reveals that during the preparation of the slurry the starting Ru/Al_2O_3 catalyst suffers a redispersion of metallic Ru particles and more surface hydroxyls are created by the incorporation of additional alumina.

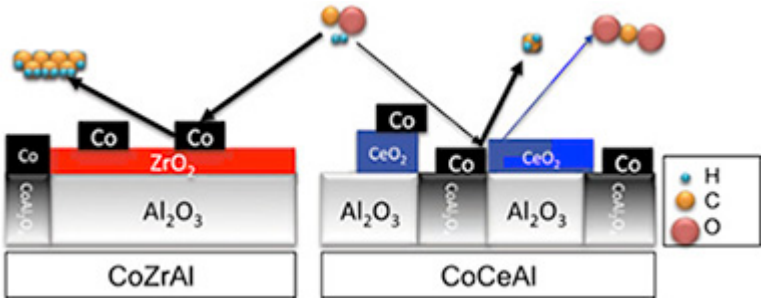
These modifications have a noticeable influence in the catalytic performance and despite their importance, these aspects have been poorly considered in other studies.

Promoting effect of CeO_2, ZrO_2 and Ce/Zr mixed oxides on Co/gamma-Al_2O_3 catalyst for Fischer-Tropsch synthesis

Garcilaso, V, Barrientos, J, Bobadilla, LF, Laguna, OH, Boutonnet, M, Centeno, MA, Odriozola, JA

Renewable Energy, **132** (2019) 1141-1150

Marzo, 2019 | **DOI:** 10.1016/j.renene.2018.08.080



A series of cobalt-based catalysts have been synthesized using as support gamma-Al_2O_3 promoted by ceria/zirconia mixed oxides with a variable Ce/Zr molar ratio. The obtained catalysts demonstrated oxide promotion results in the protection of the major textural properties, especially for Zr-rich solids. Reducibility of cobalt species was enhanced by the presence of mixed oxides. The chemical composition of the oxide promoter influenced not only physicochemical properties of final catalysts but also determined their performance during the reaction. In this sense, Zr-rich systems presented a superior catalytic performance both in total conversion and in selectivity towards long chain hydrocarbons. The observed Zr-promotion effect could be explained by two significant contributions: firstly, the partial inhibition of Co-Al spinel compound formation by the presence of Zr-rich phases which enhances the availability of Co actives site and secondly, Zr-associate acidic sites promote higher hydrocarbons selectivity.

Photodegradation of 2,4-dichlorophenoxyacetic acid over TiO_2(B)/anatase nanobelts and Au-TiO2(B)/anatase nanobelts

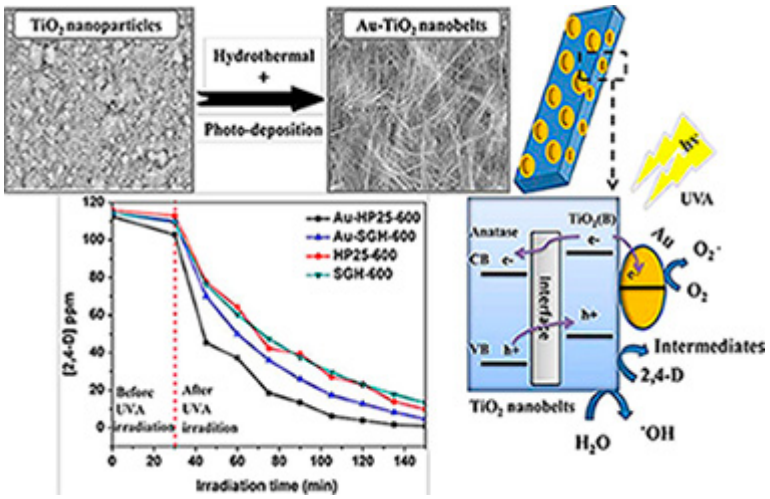
Chenchana, A., Nemamcha, A., Moumeni, H., Doña Rodríguez, J.M., Araña, J., Navío, J.A., González Díaz, O., Pulido Melián, E.

Applied Surface Science, **467-468** (2019) 1076-1087

Febrero, 2019 | **DOI:** 10.1016/j.apsusc.2018.10.175

In this work, novel TiO_2-based nanobelts with various phases were synthesized: biphasic TiO_2(B)/anatase, pure TiO_2(B) and pure anatase. These catalysts were obtained via hydrothermal reaction using two nanoparticulated TiO_2 photocatalysts as precursors: Aeroxide TiO_2 P25 (P25) and TiO_2 synthesized via a sol-gel process (SG). In addition, the surface of the photocatalysts was modified with gold using a photodeposition method. A characterization study of the different photocatalysts was performed with X-ray diffraction analysis (XRD), UV–Vis diffuse reflectance spectra (DRS), scanning electron microscopy (SEM), X-ray photoelectron spectrum analysis (XPS)

and Brunauer-Emmett-Teller measurements (BET). The photocatalytic reaction of the herbicide 2,4-dichlorophenoxyacetic acid (2,4-D) was investigated under UVA irradiation. A toxicity analysis was performed with the marine bioluminescent bacteria Vibrio fischeri. The highest 2,4-

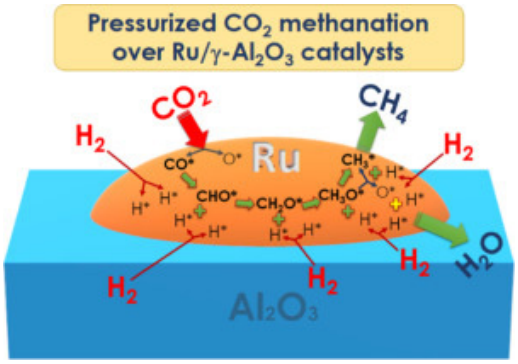


D removal efficiency of 99.2% was obtained with the biphasic Au-TiO_2(TiO_2(B)/anatase) nanobelts with anatase as predominant phase. Toxicity was mainly due to the intermediate 2,4-dichlorophenol (2,4-DCP) which was eliminated in 4 h. The TiO_2 nanobelt phase structure is shown to have a significant effect on photocatalytic activity.

Size-tailored Ru nanoparticles deposited over gamma-Al_2O_3 for the CO_2 methanation reaction

Navarro-Jaen, S, Navarro, JC, Bobadilla, LF, Centeno, MA, Laguna, OH, Odriozola, JA
Applied Surface Science, **483** (2019) 750-761
Julio, 2019 | DOI: 10.1016/j.apsusc.2019.03.248

By means of the polyol method, a series of 5 wt% Ru/Al_2O_3 catalysts was synthesized controlling the particle size of the ruthenium species. The physico-chemical characterization demonstrated the successful particle size control of the Ru species, in such a way that higher the Ru/PVP ratio,

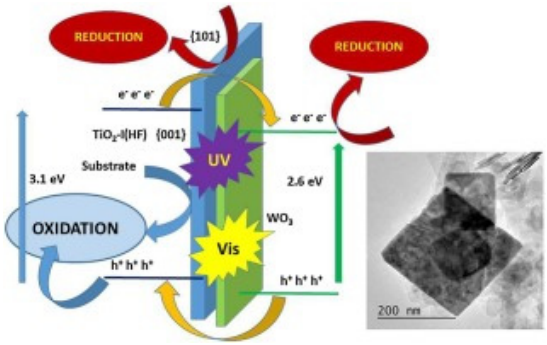


higher the Ru particle size. Moreover, there are evidences that suggest preferential growth of the RuO_2 clusters depending on the Ru/PVP ratio. Regarding the catalytic activity during the CO_2 methanation, the total conversion and the CH_4 yield increased with the particle size of Ru.

Nevertheless, a considerable enhancement of the catalytic performance of the most active system was evidenced at 4 bar, demonstrating the improvement of the thermodynamics (superior total conversion) and kinetics (superior reaction rate) of the CO_2 methanation at pressures above the atmospheric one. Finally, the in situ DRIFTS study allowed to establish that CO_2 was dissociated to CO* and O* species on the metallic Ru particles, followed by the consecutive hydrogenation of CO* towards CHO*, CH_2O*, CH_3O*, and finally CH_4 molecules, which were further desorbed from the catalyst. Thus from the mechanistic point of view, a suitable particle size of the Ru nanoparticles along with the high-pressure effects results in the enhancement of the availability of hydrogen and consequently in the formation of CHxO species that enhance the cleavage of the C-O bond, which is the rate-determining step of the overall CO_2 methanation process.

Coupling of WO_3 with anatase TiO_2 sample with high {001} facet exposition: Effect on the photocatalytic properties

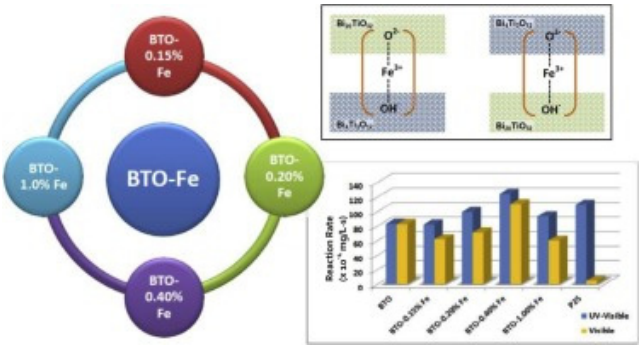
Lara, M.A., Jaramillo-Páez, C., Navío, J.A., Sánchez-Cid, P., Hidalgo, M.C.
Catalysis Today, **328** (2019) 142-148
Mayo, 2019 | **DOI:** 10.1016/j.cattod.2018.11.012



A highly faceted {001} TiO_2 catalyst was hydrothermally synthesized by using Ti(IV)-isopropoxide precursor with aqueous HF addition. WO_3 was synthesized by following a reported method. Coupled TiO_2-WO_3 samples were synthesized by adding the corresponding amount of WO3 to fluorinated TiO_2 gel followed by a hydrothermal treatment. Additionally the synthesized systems were characterized by using X-ray powder diffraction (XRD), X-ray fluorescence spectrometry (XRF), field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), UV–vis diffuse reflectance spectroscopy (DRS) and N_2-adsorption (BET) for specific surface area determination. The photocatalytic activity of the single and coupled oxides was measured by means of three model reactions: the photo-oxidation of phenol (as a colourless substrate) and methyl orange (as a dye) and the photoreduction of Cr(VI) as $K_2Cr_2O_7$. The coupling of WO_3 with a highly faceted {001} TiO_2 makes it possible to optimize the photocatalytic properties of the faceted material. In fact, {001} faceted TiO2 by itself presents a substantial improvement with respect to commercial TiO_2(P25), as it can implement its photoactivity after the incorporation of WO_3 with promising results, which can reduce the limitations of TiO_2 in terms of its photoactivity, taking advantage of a higher percentage of solar radiation.

BixTiyOz-Fe multiphase systems with excellent photocatalytic performance in the visible

Zambrano, P., Navío, J.A., Hidalgo, M.C.
Catalysis Today, **328** (2019) 136-141
Mayo, 2019 | DOI: 10.1016/j.cattod.2018.11.032



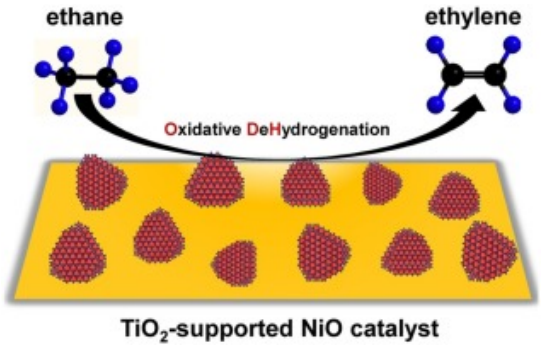
New photocatalysts based on bismuth titanates doped with iron with outstanding visible photocatalytic activity were prepared by a facile hydrothermal method followed by incipient wetness impregnation. The starting material was composed by three phases; majority of $Bi_{20}TiO_{32}$ closely interconnected to $Bi4Ti_3O_{12}$ and amorphous TiO_2. Fe doping increased the already very high visible activity of the original material. The high visible activity showed by these materials could be ascribed to a combination of several features; i.e. low band gap energy value (as low as 1.78 eV), a structure allowing a good separation path for visible photogenerated electron-holes pairs and a relatively high surface area. Fe doping could be acting as bonding paths for the bismuth titanates phases, and the amount of Fe on the surface was found to be a crucial parameter on the photocatalytic activity of the materials. Visible activity of the best photocatalyst was superior to UV-Activity of commercial TiO_2 P25 used as reference in same experimental conditions.

Support effects on NiO-based catalysts for the oxidative dehydrogenation (ODH) of ethane

Delgado, D, Sanchis, R, Cecilia, JA, Rodriguez-Castellon, E, Caballero, A, Solsona, B, Nieto, JML
Catalysis Today, **333** (2019) 10-16
Agosto, 2019 | **DOI:** 10.1016/j.cattod.2018.07.010

We report on the effect of NiO-support interactions on the chemical nature of Ni species in a series of supported NiO catalysts for the ODH of ethane. SiO_2, TiO_2-anatase, a high surface area TiO_2 and a porous clay hetero-structure (PCH) with TiO2 and SiO_2 pillars were used as supports, which led to a selectivity to ethylene in the range 30-90% over supported NiO catalysts. The catalysts were characterized by means of XRD, N-2-Adsorption, H-2-TPR, XPS and in situ (under H-2 reductive atmosphere) and ex situ XAS spectroscopy. The catalytic performance of supported materials is discussed in terms of their reducibility and specific reduction kinetics, but

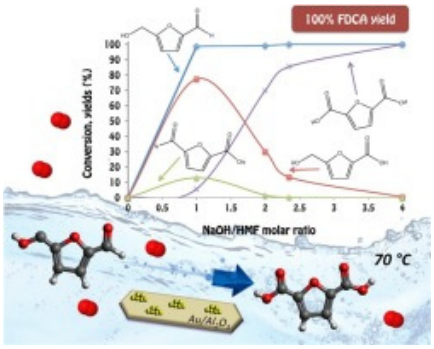
also taking into account the specific chemical nature of Ni species on each catalyst. The influence



of the particle size and the presence of Ni and O vacancies on the catalytic performance in the ODH of ethane is inferred.

Au/Al_2O_3 - Efficient catalyst for 5-hydroxymethylfurfural oxidation to 2,5-furandicarboxylic acid

Megias-Sayago, C, Lolli, A, Ivanova, S, Albonetti, S, Cavani, F, Odriozola, JA
Catalysis Today, **333** (2019) 169-175
Agosto, 2019 | **DOI:** 10.1016/j.cattod.2018.04.024

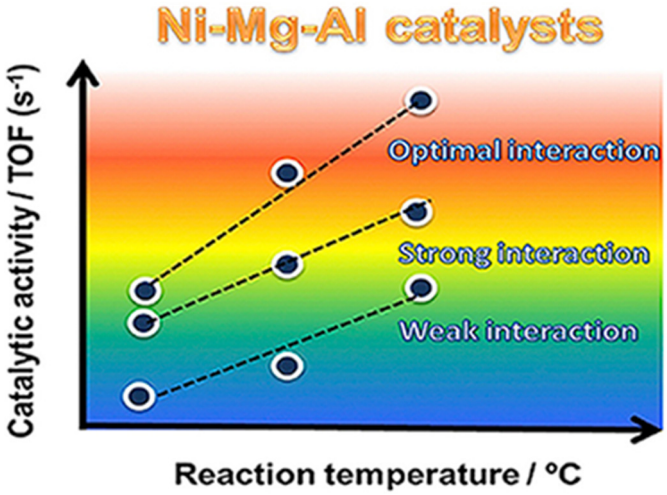


The catalytic activity of a simple Au/Al_2O_3 catalytic system prepared by the direct anionic exchange (DAE) method was evaluated in the selective 5-hydroxymethylfurfural (HMF) oxidation under mild conditions, using molecular oxygen as the oxidant. The influence of the HMF/NaOH ratio and reaction time on product yield and distribution were studied and discussed in detail. Extremely high activity and selectivity were observed in mild conditions, with 99% of 2,5-furandicarboxylic acid (FDCA) production at full HMF conversion after 4 h with the use of only 4 equivalents of NaOH at 70 degrees C. Catalyst viability and stability were verified by repeating the cycle up to five times. Changes in the nature of the support were also contemplated by introducing some ceria fraction, i.e. 20 wt%.

Colombian metallurgical coke as catalysts support of the direct coal liquefaction
Rico, D, Agamez, Y, Romero, E, Centeno, MA, Odriozola, JA, Diaz, JD
Fuel, **255** (2019) 115748
Noviembre, 2019 | **DOI:** 10.1016/j.fuel.2019.115748

A Colombian metallurgical coke was modified in its surface chemistry and was used as support of iron sulfide catalysts for direct coal liquefaction. The modification was made by treatments with diluted oxygen and HNO_3 at different conditions. Changes in surface chemistry were studied by determining the point of zero charge (PZC), the isoelectric point (IEP), thermogravimetric analysis (TGA), temperature programmed decomposition-mass spectrometry (TPD-MS), Diffuse-reflectance infrared Fourier transform spectroscopy (DRIFTS) and nitrogen adsorption at 77 K. The results show that the materials obtained have a wide range of functional groups incorporated in a different proportion and quantity. The textural parameters indicate that treatment with diluted oxygen increases the surface area and incorporates micropores while the samples treated with HNO3 maintain the textural properties of the original material. The catalysts were also characterized by Raman spectroscopy. It was found that impregnation with the iron sulfide precursor does not significantly affect the Raman characteristics of the support. Additionally, XRD analysis shows smaller pyrite crystallites in the coke enriched with oxygenated groups of phenol and lactone indicating better dispersion of the active phase. The amount of oxygen chemisorbed per gram of catalyst shows that both, oxygen and nitric acid treatments, improve the relative dispersion of the active phase. It was found that the presence of the catalysts increases the conversion and yields towards oils and gases with respect to those of the tests without catalysts. Cokes modified by dilute oxygen gaseous treatment contain surface phenol and lactone groups and present the highest yield to oils.

Influence of the preparation method in the metal-support interaction and reducibility of Ni-Mg-Al based catalysts for methane steam reforming
Azancot, L, Bobadilla, LF, Santos, JL, Cordoba, JM, Centeno, MA, Odriozola, JA
International Journal of Hydrogen Energy, **44** (2019) 19827-19840
Julio, 2019 | **DOI:** 10.1016/j.ijhydene.2019.05.167



Ni-Mg-Al based catalysts were prepared using different preparation methods (impregnation, impregnation-coprecipitation and coprecipitation) and tested in steam reforming of methane. The differences observed in catalytic activity were directly correlated to the physicochemical properties and the different degree of Ni-Mg-Al interaction. The reducibility results showed that the catalyst prepared by the impregnation-coprecipitation method presented the most optimal metal-support interaction to reduce the NiO preserving the Ni-0 particles highly dispersed on the support surface. These results demonstrate that the structure and catalytic performance of Ni-Mg-Al based catalysts can be tuned by controlling the metal-support interaction through of the preparation method.

Operando Spectroscopic Evidence of the Induced Effect of Residual Species in the Reaction Intermediates during CO_2 Hydrogenation over Ruthenium Nanoparticles

Navarro-Jaen, S, Szego, A, Bobadilla, LF, Laguna, OH, Romero-Sarria, F, Centeno, MA, Odriozola, JA

Chemcatchem, **11** (2019) 2063-2068

Abril, 2019 | **DOI:** 10.1002/cctc.201900101

In this work, we present a highly active catalyst based on Ru nanoparticles dispersed on alumina, which showed an unexpected activity for CO2 methanation. This exceptional catalytic behavior was attributed to the presence of residual species that remained on the surface after synthesis. Furthermore, through Operando DRIFTS (diffuse reflectance infrared Fourier transform spectroscopy) measurements it was demonstrated that these remaining species provoked an induced effect on the nature of the surface intermediates spectroscopically observed, and consequently on their mechanistic role during the pathway of the CO_2 hydrogenation to methane.

Noble Metal Supported on Activated Carbon for "Hydrogen Free" HDO Reactions: Exploring Economically Advantageous Routes for Biomass Valorisation

Jin, W, Santos, JL, Pastor-Perez, L, Gu, S, Centeno, MA, Reina, TR

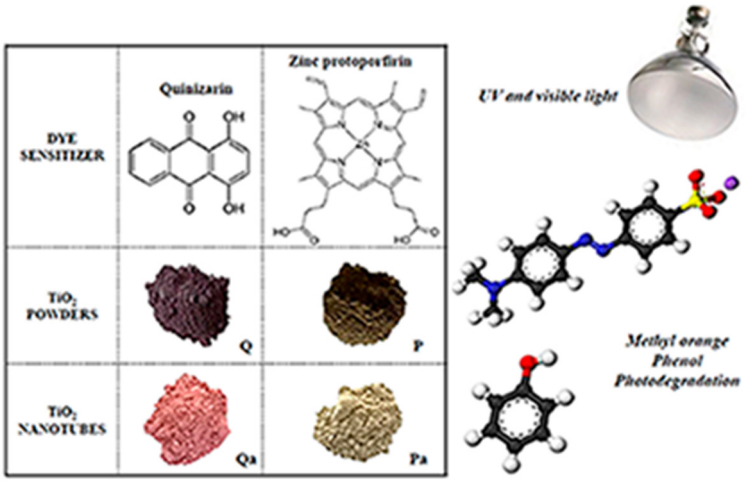
Chemcatchem, **11** (2019) 4434-4441

Agosto, 2019 | **DOI:** 10.1002/cctc.201900841

An innovative route for bio-compounds upgrading via “hydrogen-free” hydrodeoxygenation (HDO) is proposed and evaluated using guaiacol as a model compound in a high-pressure batch reactor. Experimental results showed that noble metal supported on activated carbon catalysts are able to conduct tandem multiple steps including water splitting and subsequent HDO. The activity of Ru/C catalyst is superior to other studied catalysts (i. e. Au/C, Pd/C and Rh/C) in our water-only HDO reaction system. The greater dispersion and smaller metal particle size confirmed by the TEM micrographs accounts for the better performance of Ru/C. This material also presents excellent levels of stability as demonstrated in multiple recyclability runs. Overall, the proposed novel approach confirmed the viability of oxygenated bio-compounds upgrading in a water-only reaction system suppressing the need of external H2 supply and can be rendered as a fundamental finding for the economical biomass valorisation to produce added value bio-fuels.

Powder and Nanotubes Titania Modified by Dye Sensitization as Photocatalysts for the Organic Pollutants Elimination

Murcia, JJ, Avila-Martinez, EG, Rojas, H, Cubillos, J, Ivanova, S, Penkova, A, Laguna, OH
Nanomaterials, **9** (2019) 517
Abril, 2019 | **DOI:** 10.3390/nano9040517



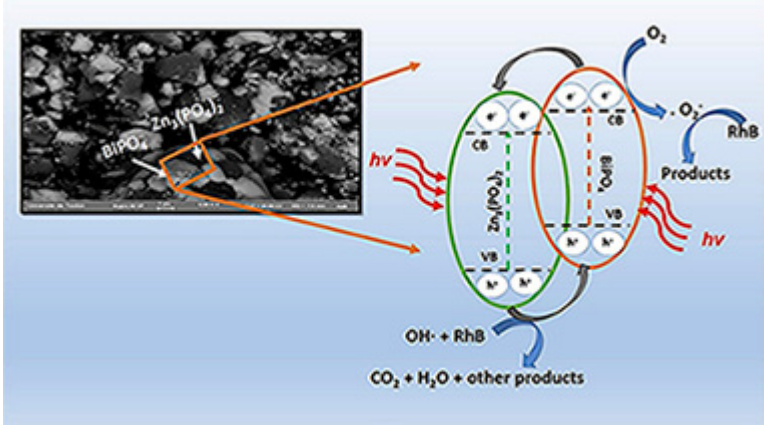
In this study, titanium dioxide powder obtained by the sol-gel method and TiO_2 nanotubes, were prepared. In order to increase the TiO_2 photoactivity, the powders and nanotubes obtained were modified by dye sensitization treatment during the oxide synthesis. The sensitizers applied were Quinizarin (Q) and Zinc protoporphyrin (P). The materials synthesized were extensively characterized and it was found that the dye sensitization treatment leads to modify the optical and surface properties of Titania. It was also found that the effectiveness of the dye-sensitized catalysts in the phenol and methyl orange (MO) photodegradation strongly depends on the dye sensitizer employed. Thus, the highest degradation rate for MO was obtained over the conventional Q-TiO_2 photocatalyst. In the case of the nanotubes series, the most effective photocatalyst in the MO degradation was based on TiO_2-nanotubes sensitized with the dye protoporfirin (ZnP). Selected catalysts were also tested in the phenol and MO photodegradation under visible light and it was observed that these samples are also active under this radiation.

Preparation, characterization and photocatalytic degradation of Rhodamine B dye over a novel $Zn_3(PO_4)_2/BiPO_4$ catalyst

Naciri, Y., Chennah, A., Jaramillo-Páez, C., Navío, J.A., Bakiz,B., Taoufyq, A., Ezahri, M., Villain,S., Guinneton,F., Benlhachemi,A.
Journal of Environmental Chemical Engineering, **7** (2019) 2103075
Junio, 2019 | **DOI:** 10.1016/j.jece.2019.103075

In this work, a facile method was used to synthesize the $Zn_3(PO_4)_2/BiPO_4$ composite photocatalysts with different Bi contents followed by heat treatment at 900 °C for 3 h. The as-prepared samples were studied by a variety of characterization techniques including X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) combined with energy dispersive X-ray diffraction (EDX), Transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and UV–vis diffuse reflectance spectroscopy (DRS). The UV–vis spectroscopy was used to

analyze the evolution of Rhodamine B discoloration in presence of the synthesized phosphate photocatalysts. The XRD, SEM-EDX, TEM, DRS and XPS analyses confirmed the formation of heterojunction structure between both materials, during the process of co-precipitation and ulterior heat treatment. The photocatalytic tests showed that photocatalytic ability of the 70%



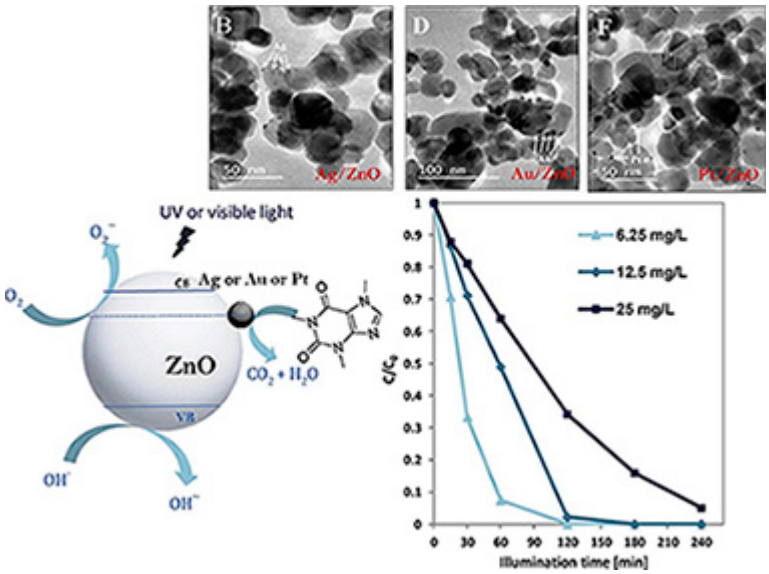
Bi-$Zn_3(PO_4)_2$ composites was higher than that of pure $Zn_3(PO_4)_2$ and $BiPO_4$ after 1 h of UV-illumination. The obviously enhanced photocatalytic activity of the 70% Bi-$Zn_3(PO_4)_2$ sample could be mainly attributed to the formation of the heterojunction, accelerating the separation of photogenerated charge carriers. A plausible mechanism of the photocatalytic degradation of RhB on $Zn_3(PO_4)_2$/$BiPO_4$ composites is proposed. The reduction in the Chemical Oxygen Demand (COD) revealed the mineralization of dye along with color removal. Thus, it can be suggested that the 70% Bi-$Zn_3(PO_4)_2$ can serve as a promising photocatalyst in the degradation of organic contaminants under UV light.

UV and visible-light driven photocatalytic removal of caffeine using ZnO modified with different noble metals (Pt, Ag and Au)

Vaiano, V., Jaramillo-Paez, C.A., Matarangolo, M., Navío, J.A., Hidalgo, M.C.
Materials Research Bulletin, **5112** (2019) 251-260
Abril, 2019 | **DOI:** 10.1016/j.materresbull.2018.12.034

In this work, ZnO photocatalyst was modified with different noble metals (Pt, Ag and Au) through photodeposition method and then characterized by different techniques (XRD, XRF, BET, UV–vis DRS, FESEM, and XPS). The addition of noble metals produces important changes in the light absorption properties with a significant absorbance in the visible region due to the existence of surface plasmon resonance (SPR) observed at about 450 nm and 550 nm for ZnO modified with Ag and Au, respectively. The morphology of the samples was studied by TEM and the size ranges of the different metals were estimated. Noble metal nanoparticles were in every case heterogeneously deposited on the larger ZnO particles. All the prepared photocatalysts were tested in the photocatalytic removal of caffeine (toxic and persistent emerging compound) under UV and visible light irradiation. It was observed an enhancement of photocatalytic caffeine removal from aqueous solutions under UV light irradiation with the increase of metal content (from 0.5 to 1 wt %) for ZnO modified with Ag and Au (Ag/ZnO and Au/ZnO). In particular, Ag/ZnO and Au/ZnO with higher Ag and Au content (1 wt %) allowed to achieve the almost complete caffeine degradation after only 30 min and a TOC removal higher than 90%

after 4 h of UV light irradiation. These two photocatalysts were investigated also under visible light irradiation and it was found that their photocatalytic performances were strongly enhanced



in presence of visible light compared to unmodified ZnO. In particular, Ag/ZnO photocatalyst was able to reach the complete caffeine degradation and a TOC removal of about 70% after 4 h of visible light irradiation.

Synthesis of sol-gel pyrophyllite/TiO_2 heterostructures: Effect of calcination temperature and methanol washing on photocatalytic activity

El Gaidoumi, A., Doña Rodríguez, J.M., Pulido Melián, E., González-Díaz, O.M., Navío Santos, J.M., El Bali, B., Kherbeche, A.

Surfaces and Interfaces, **14** (2019) 19-25
Marzo, 2019 | **DOI:** 10.1016/j.surfin.2018.10.003

We successfully synthesized an efficient photoactive pyrophyllite/TiO2 heterostructures using a sol-gel route at ambient temperature. The samples were prepared by exfoliation of a pyrophyllite layered-type clay by TiO2. The prepared samples exhibited strong photocatalytic activity for the degradation of phenol. The heterostructure PTi750 (SBET = 16.58 m2/g) calcined at 750 °C, in which the mixed phases of anatase and rutile exist (52.2% anatase/10.7% rutile), showed the highest photocatalytic activity against commercial TiO2Aeroxide P25. The methanol washed PTi750 was 5 times faster than the corresponding unwashed sample; phenol was totally degraded with a TOC reduction of 89.2%. The materials have been characterized by: X-ray diffraction (XRD), Diffuse reflectance UV–vis spectrophotometry (UV–Vis DRS), scanning electron microscopy (SEM) and BET specific surface area.

Au/CeO_2-ZnO/Al_2O_3 as Versatile Catalysts for Oxidation Reactions: Application in Gas/Liquid Environmental Processes

Megias-Sayago, C, Reina, TR, Ivanova, S, Odriozola, JA
Frontiers in Chemistry, **7** (2019) 504
Julio, 2019 | **DOI:** 10.3389/fchem.2019.00504

The present work showcases the versatility of nanogold systems supported on Zn-doped ceria when applied in two important environmental processes, the total CO oxidation, and the liquid phase oxidation of glucose to gluconic acid. In the CO oxidation the suitability of these materials is clearly demonstrated achieving full conversions even at sub-ambient conditions. Regarding the glucose oxidation our materials display high conversion values (always over 50%) and very importantly full or almost full selectivity toward gluconic acid-an added value platform chemical in the context of biomass upgrading routes. The key factors controlling the successful performance on both reactions are carefully discussed and compared to previous studies in literature. To our knowledge this is one of the very few works in catalysis by gold combining liquid and gas phase reactions and represents a step forward in the flexible behavior of nano gold catalysts.

The Success Story of Gold-Based Catalysts for Gas- and Liquid-Phase Reactions: A Brief Perspective and Beyond

Price, CAH, Pastor-Perez, L, Ivanova, S, Reina, TR, Liu, J
Frontiers in Chemistry, **7** (2019) 691
Octubre, 2019 | **DOI:** 10.3389/fchem.2019.00691

Gold has long held the fascination of mankind. For millennia it has found use in art, cosmetic metallurgy and architecture; this element is seen as the ultimate statement of prosperity and beauty. This myriad of uses is made possible by the characteristic inertness of bulk gold; allowing it to appear long lasting and above the tarnishing experienced by other metals, in part providing its status as the most noble metal.

Dry Reforming of Ethanol and Glycerol: Mini-Review

Yu, J, Odriozola, JA, Reina, TR
Catalysts, **9** (2019) 1015
Diciembre, 2019 | **DOI:** 10.3390/catal9121015

Dry reforming of ethanol and glycerol using CO_2 are promising technologies for H-2 production while mitigating CO_2 emission. Current studies mainly focused on steam reforming technology, while dry reforming has been typically less studied. Nevertheless, the urgent problem of CO_2 emissions directly linked to global warming has sparked a renewed interest on the catalysis community to pursue dry reforming routes. Indeed, dry reforming represents a straightforward route to utilize CO_2 while producing added value products such as syngas or hydrogen. In the absence of catalysts, the direct decomposition for H-2 production is less efficient. In this mini-review, ethanol and glycerol dry reforming processes have been discussed including their mechanistic aspects and strategies for catalysts successful design. The effect of support and promoters is addressed for better elucidating the catalytic mechanism of dry reforming of

ethanol and glycerol. Activity and stability of state-of-the-art catalysts are comprehensively discussed in this review along with challenges and future opportunities to further develop the dry reforming routes as viable CO_2 utilization alternatives.

Carbon Supported Gold Nanoparticles for the Catalytic Reduction of 4-Nitrophenol

Molina, HR, Munoz, JLS, Leal, MID, Reina, TR, Ivanova, S, Gallego, MNC, Odriozola, JA
Frontiers in Chemistry, **7** (2019) 548
Agosto, 2019 | **DOI:** 10.3389/fchem.2019.00548

This work is a detailed study on how to optimize gold colloids preparation and their deposition to very different in nature carbon materials. The change of the continuous phase and its dielectric constant is used to assure the good dispersion of the hydrophilic/hydrophobic carbons and the successful transfer of the preformed small size colloids to their surface. The sintering behavior of the particles during the calcination step is also studied and the optimal conditions to reduce to a minimum the particle size increase during the protecting agent removal phase are found. The as prepared catalysts have been tested in a relevant reaction in the field of environmental catalysis such as the reduction of 4-nitrophenol leading to promising results. Overall, this work proposes an important methodology to follow when a carbonaceous material are selected as catalyst supports for green chemistry reactions.

Immobilization of Stabilized Gold Nanoparticles on Various Ceria-Based Oxides: Influence of the Protecting Agent on the Glucose Oxidation Reaction

Chenouf, M; Megias-Sayago, C; Ammari, F; Ivanova, S; Centeno, MA; Odriozola, JA
Catalysts, **9** (2019) 331
Abril, 2019 | **DOI:** 10.3390/catal9020125

The influence of the protecting agent's nature on gold particle size and dispersion was studied in this work over a series of gold-based catalysts. CO and glucose oxidation were chosen as catalytic reactions to determine the catalyst's structure-activity relationship. The nature of the support appeared to be the predominant factor for the increase in activity, as the oxygen mobility was decisive for the CO oxidation in the same way that the Lewis acidity was decisive for the glucose oxidation. For the same catalyst composition, the use of montmorillonite as the stabilizing agent resulted in better catalytic performance.

Differences in the Catalytic Behavior of Au-Metalized TiO_2 Systems During Phenol Photo-Degradation and CO Oxidation

Oscar H. Laguna, Julie J. Murcia, Hugo Rojas, Cesar Jaramillo-Paez, Jose A. Navío, Maria C. Hidalgo
Catalysts, **9** (2019) 331
Abril, 2019 | **DOI:** 10.3390/catal9040331

For this present work, a series of Au-metallized TiO_2 catalysts were synthesized and characterized in order to compare their performance in two different catalytic environments:

the phenol degradation that occurs during the liquid phase and in the CO oxidation phase, which proceeds the gas phase. The obtained materials were analyzed by different techniques such as XRF, SBET, XRD, TEM, XPS, and UV-Vis DRS. Although the metallization was not totally efficient in all cases, the amount of noble metal loaded depended strongly on the deposition time. Furthermore, the differences in the amount of loaded gold were important factors influencing the physicochemical properties of the catalysts, and consequently, their performances in the studied reactors. The addition of gold represented a considerable increase in the phenol conversion when compared with that of the TiO_2, despite the small amount of noble metal loaded. However, this was not the case in the CO oxidation reaction. Beyond the differences in the phase where the reaction occurred, the loss of catalytic activity during the CO oxidation reaction was directly related to the sintering of the gold nanoparticles.

Fluorinated and Platinized Titania as Effective Materials in the Photocatalytic Treatment of Dyestuffs and Stained Wastewater Coming from Handicrafts Factories

Murcia, J.J., Cely, A.C., Rojas, H.A., Hidalgo, M.C., Navío, J.A.
Catalysts, **9** (2019) 179
Febrero, 2019 | **DOI:** 10.3390/catal9020179

In this study, commercial and lab-prepared TiO2 were modified by fluorination and platinum photodeposition; and the effect of these modifications over the physicochemical and photocatalytic properties of TiO2 was evaluated. It was found that F and Pt addition leads to the modification of the optical and textural properties of TiO2. The materials prepared were tested in the photocatalytic degradation of different organic dyestuffs such as methylene blue (MB) and methyl orange (MO); the degradation of commercial anilines employed in the staining of natural fibers was also evaluated. Photocatalysis was also studied in this work as an eco-friendly treatment of wastewater coming from handicrafts factories. In general it was observed that the effectiveness of the photocatalytic treatment strongly depends on the substrate to be degraded, thus, fluorinated and platinized commercial Titania (Pt-F-P25) showed the best photocatalytic performance in the MB and MO photodegradation and in contrast, in the case of the anilines the highest degradation was obtained over commercial TiO2 fluorinated (F-P25). These results can be explained by differences observed in the structure and in the adsorption of these dyestuffs over the photocatalysts surfaces. F-P25 photocatalyst also demonstrated to be the best material for the treatment of real wastewater coming from handicrafts factories.

Extraordinary visible photocatalytic activity of a Co0.2Zn0.8O system studied in the Remazol BB oxidation

KarimTanji, J.A.Navio, Jamal Naja, M.C.Hidalgo, Abdellah Chaqroune, C.Jaramillo-Páez, Abdelhak Kherbeche
Journal of Photochemistry and Photobiology A: Chemistry, **382** (2019) 111877
Septiembre, 2019 | **DOI:** 10.1016/j.jphotochem.2019.111877

Nanoparticles of CoxZn1-xO system with a nominal composition of x=0.2 were synthesized by the Solution Combustion Method (SCM). Structural and morphological studies as well as the chemical composition of the material were widely investigated by different techniques. Photocatalytic activity under UV and Visible illumination was studied by means of the Remazol

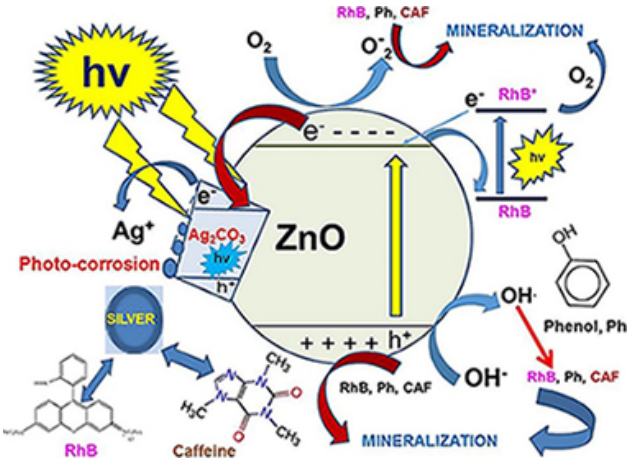
Brilliant Blue dye (RBB) oxidation reaction. The effect of different experimental parameters, such as the initial dye concentration, photocatalyst mass, pH or hydrogen peroxide concentration on the RBB discoloration under UV irradiation was studied. Optimal experimental conditions were found to be a photocatalyst mass of 1 g.L-1, dye concentration of 20 mg.L-1 and solution pH of 11. Hydrogen peroxide addition was found to have no effect in the photocatalytic behavior of the material in the range of concentration studied (0 to 6•10-4 M). The optimal parameters were chosen to investigate the degradation of RBB under UV-illumination and just visible illumination. It was observed that the UV-photocatalytic property of pristine ZnO for the RBB removal was scarcely improved after cobalt-incorporation, whereas the effect of cobalt incorporation into ZnO greatly enhanced the RBB conversion under visible illumination. Even more interesting is that, under same experimental conditions, the visible efficiency of the Co-ZnO system is the same that the one showed under UV illumination, i.e. the system does not loose efficiency when illuminated only with visible light.

Coupling of Ag_2CO_3 to an optimized ZnO photocatalyst: Advantages vs. disadvantages

P. Sánchez-Cid, C. Jaramillo-Páez, J.A. Navío, A.N. Martín-Gómez, M.C. Hidalgo
Journal of Photochemistry and Photobiology A: Chemistry, **369** (2019) 119-132
Enero, 2019 | **DOI:** 10.1016/j.jphotochem.2018.10.024

With the aim of improving the photocatalytic properties of a previously optimized zinc oxide photocatalyst, the effect of the incorporation of different amounts of Ag_2CO_3 on the aforementioned ZnO has been studied. For this purpose we report the synthesis, by means of simple precipitation procedures, of bare ZnO and Ag_2CO_3 samples as well as the coupled materials ZnO/Ag_2CO_3 (X) (where X = 1%, 2%, 4% and 5% in molar percentages). Both, single and coupled materials have been characterized by different techniques (XRD, XRF, N2-absorption, SEM, TEM, UV–vis/DRS and XPS). To assess the advantages or disadvantages that Ag_2CO_3 addition could have over the optimized ZnO, the photocatalytic properties have been established by following the photo-degradation of selected toxic molecules, both in the UV and

in the visible, as well as using complementary techniques of liquid medium analyses (TOC and Atomic Emission Spectrometry with plasma ICP). Three selected substrates were chosen: Rhodamine B (RhB) as a dye, and phenol and caffeine as colourless recalcitrant toxic molecules. Our results suggest that although the use of Ag2CO3 could be beneficial to implement the optical



absorption towards the visible region, however, other effects have to be bore in mind, such as the photo-corrosion of Ag_2CO_3 and the chemical structure of the chosen substrate, to elucidate whether the addition of Ag_2CO_3 has beneficial or detrimental effects on the photocatalytic properties of the coupled ZnO/Ag_2CO_3 materials.

Urban wastewater treatment by using Ag/ZnO and Pt/TiO_2 photocatalysts

J.J. Murcia, L.G. Arias Bolivar, H.A. Rojas Sarmiento, E.G. Ávila Martínez, C. Jaramillo Páez, M.A. Lara, J.A. Navío Santos, M.C. Hidalgo López

Environmental Science and Pollution Research, **26** (2019) 4171-4179
Febrero, 2019 | **DOI:** 10.1007/s11356-018-1592-3

In this study, the treatment of wastewater coming from a river highly polluted with domestic and industrial effluents was evaluated. For this purpose, series of photocatalysts obtained by ZnO and TiO_2 modification were evaluated. The effect of metal addition and Ti precursor (in the case of the titania series) over the physicochemical and photocatalytic properties of the materials obtained was also analyzed. The evaluation of the photocatalytic activity showed that semiconductor modification and precursor used in the materials synthesis are important factors influencing the physicochemical and therefore the photocatalytic properties of the materials obtained. The water samples analyzed in the present work were taken from a highly polluted river, and it was found that the effectiveness of the photocatalytic treatment increases when the reaction time increases and for both, wastewater samples and isolated Escherichia coli strain follow the next order Pt/TiO_2 << ZnO. It was also observed that biochemical and chemical demand oxygen and turbidity significantly decrease after treatment, thus indicating that photocatalysis is a non-selective technology, which can lead to recover wastewater containing different pollutants.

Effect of starch as binder in carbon aerogel and carbon xerogel preparation

Rodriguez, N, Agamez-Pertuz, YY, Romero, E, Diaz-Velasquez, JD, Odriozola, JA, Centeno, MA
Journal of Non-Crystalline Solids, **522** (2019) 119554
Octubre, 2019 | **DOI:** 10.1016/j.jnoncrysol.2019.119554

Carbon aerogels and carbon xerogels were synthesized through resorcinol - formaldehyde polycondensation using Na2CO3 as catalyst. The effect of soluble starch introduction in the organic gel preparation on the porous surface properties of these materials was studied. The role of the drying process of the organic gels on the changes in the surface and structural properties of these materials after the addition of soluble starch is discussed. The presence of starch in the prepared carbon xerogels results in the development of microporosity while maintaining the characteristic mesoporosity of carbon xerogels. The Brunauer - Emmett -Teller (BET) surface area increases from 309 m(2)/g in carbon xerogel without soluble starch until 685 m(2)/g when 10% of soluble starch is added. The R- value and average crystallite lattice parameters, inter-layer spacing, crystallite height, crystallite diameter and the average number of aromatic layers per carbon crystallite are discussed in function of drying step and presence of soluble starch. The surface properties were also studied by Raman and DRIFT spectroscopies.

Mesoporous pyrophyllite–titania nanocomposites: synthesis and activity in phenol photocatalytic degradation

A. El Gaidoumi, J.M. Doña-Rodríguez, E. Pulido Melián, O.M. González-Díaz, B. El Bali, J.A. Navío, A. Kherbeche
Research on Chemical Intermediates, **45** (2019) 333-353
Febrero, 2019 | **DOI:** 10.1007/s11164-018-3605-8

Pyrophyllite–TiO_2 nanocomposite PTi750 was successfully synthesized using a sol–gel method at ambient temperature based on exfoliation of the pyrophyllite layered clay by incorporation of the TiO_2 precursor titanium(IV) t-butoxide. PTi750 exhibited higher photocatalytic activity in phenol degradation compared with commercial TiO_2 Aeroxide P25. Ag-photodeposited PTi750 was more photoactive than PTi750, exhibiting detoxification, total degradation, and good mineralization of polluted solution and excellent stability after five reuses at optimal conditions in terms of the parameters pH, H_2O_2 concentration, and photocatalyst amount. The nanocomposites were investigated using several techniques, viz. diffuse-reflectance ultraviolet–visible (UV–Vis) spectrophotometry, scanning electron microscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, X-ray diffraction analysis, X-ray fluorescence spectroscopy, Fourier-transform infrared spectroscopy, and Brunauer–Emmett–Teller (BET) specific surface area measurements.

Montmorillonite-stabilized gold nanoparticles for nitrophenol reduction

Chenouf, M; Megias-Sayago, C; Ammari, F; Ivanova, S; Centeno, MA; Odriozola, JA
Comptes Rendus Chimie, **22** (2019) 621-627
Septiembre, 2019 | **DOI:** 10.1016/j.crci.2019.07.005

Two gold-based catalysts were obtained by Au chemical reduction of the HAuCl(4)precursor. The resulting nanoparticles were stabilized and immobilized on montmorillonite (Mt) and

montmorillonite-ceria (Mt/CeO2). All prepared catalysts were active in 4-nitrophenol to aminophenol reduction at room temperature. Synergy between montmorillonite and ceria is postulated in such a way that the montmorillonite phase hinders particle growth either by influencing the nucleation behavior of gold or by increasing the number of nucleation sites and raising the overall dispersion. The role of the ceria support, on the other hand, may be associated with the 4-NP adsorption at the ceria-gold interface, stabilizing the reaction intermediate and hence lowering the activation barrier for the reduction of 4-NP to 4-AP.

Synthesis and Characterization of ZnO-ZrO_2 Nanocomposites for Photocatalytic Degradation and Mineralization of Phenol

Lopez, MCU, Lemus, MAA, Hidalgo, MC, Gonzalez, RL, Owen, PQ, Oros-Ruiz, S, Lopez, SAU, Acosta, J

Journal of Nanomaterials, (2019) 1015876

Enero, 2019 | **DOI:** 10.1155/2019/1015876

ZnO-ZrO_2 nanocomposites using zinc (II) acetylacetonate and different ZnO contents (13, 25, 50, and 75% mol) were synthesized through sol-gel method. The synthesis process was strongly related to nanocomposite properties especially on their structural composition. The obtained ZnO-ZrO_2 nanomaterials presented tetragonal crystalline structure for zirconia whereas hexagonal one was formed in ZnO. Raman spectroscopy and XRD patterns confirmed the formation of tetragonal zirconia whereas inhibition of monoclinic structure was observed. Addition of ZnO affected the pore size distribution of the composite, and the measured specific surface areas were from 10 m^2/g (for pure ZnO) to 46 m^2/g (pristine ZrO_2). Eg values of ZrO_2 were modified by ZnO addition, since calculated values using Kubelka-Munk's function varied from 4.73 to 3.76 eV. The morphology and size of the nanomaterials investigated by electron microscopy showed formation of nanorods for ZnO with sizes ranging from 50 nm to 300 nm while zirconia was formed by smaller particles (less than 50 nm). The main advantage of using the nanocomposite for photocatalytic degradation of phenol was the mineralization degree, since 75ZnO-ZrO_2 nanocomposite surpassed mineralization reached by pure ZnO and also inhibited formation of undesirable intermediates.

Comparison of the effects generated by the dry-soft grinding and the photodeposition of Au and Pt processes on the visible light absorption and photoactivity of TiO_2

Galeano, L; Valencia, S; Marin, JM; Restrepo, G; Navio, JA; Hidalgo, MC

Materials Research Express, (2019) 1050d9

Octubre, 2019 | **DOI:** 10.1088/2053-1591/ab4316

The influence of dry-soft grinding and photodeposition of gold (Au) or platinum (Pt) in the improvement of the photoactivity of TiO_2 synthesized by an integrated sol-gel and solvothermal method was studied. TiO_2 was modified by a dry-soft grinding process in a planetary ball mill (TiO_2(G)). Subsequently, Au or Pt particles were photodeposited in both unmodified TiO_2 and TiO_2(G) obtaining Au-TiO_2, Pt-TiO_2, Au-TiO2(G), and Pt-TiO_2(G) materials. The photoactivity of the materials was evaluated in the phenol photodegradation under simulated solar radiation. Pt-TiO_2 showed the greatest degree of photoactivity improvement in comparison with TiO_2 and

TiO_2-P25. The dry-soft grinding process led to a high photocatalytic activity of TiO_2(G) that was similar to Pt-TiO_2 activity as consequence of a slight increase in the crystallinity in TiO_2(G) due to an additional anatase formation in comparison with TiO_2. However, further photocatalytic improvement in TiO_2(G) were not achieved with the addition of Au or Pt. Therefore, the dry-soft grinding treatment and noble metal deposition led to similar improvements in the photocatalytic activity of TiO_2 for phenol oxidation.

Catalytic Efficiency of Cu-Supported Pyrophyllite in Heterogeneous Catalytic Oxidation of Phenol

El Gaidoumi, A., Doña-Rodríguez, J.M., Pulido Melián, E., González-Díaz, O.M., Navío, J.A., El Bali, B., Kherbeche, A.
Arabian Journal for Science and Engineering, (2019) 1-13
Febrero, 2019 | **DOI:** 10.1007/s13369-019-03757-2

The copper-impregnated pyrophyllite (Cu/RC) was prepared and used as catalyst in catalytic wet peroxide oxidation (CWPO) of phenol. The catalyst was prepared by impregnation of copper (2.5 wt%) into pyrophyllite-type clay and characterized by X-ray diffraction, X-ray fluorescence, X-ray photoelectron spectroscopy, Fourier transform infrared spectroscopy, and transmission electron microscopy. The optimum operation conditions for CWPO of phenol over Cu/RC were determined by investigating the effects of pH, temperature, catalyst amount, and hydrogen peroxide concentration. Stability of the Cu/RC catalyst and toxicity of treated solution were studied, by measuring the copper concentration leached out from the catalyst and the inhibition of Vibrio fischeri bacteria bioluminescence, respectively. The probable degradation mechanism of phenol over Cu/RC was considered by HPLC analysis. The obtained results showed that Cu/RC achieved highest activity (total phenol degradation and 80% TOC reduction) and detoxification with remarkable low copper leaching concentration (0.006 mg\,L−1)mg\,L−1) at optimized conditions (pH == 3, T=50∘T=50∘C, 2 g\,L−1g\,L−1 catalyst amount, 50 mg L−1L−1phenol concentration and 7.45 mmol\,L−1mmol\,L−1 hydrogen peroxide concentration during 4 h). Meanwhile, few intermediates with low concentration were observed by the HPLC analysis for the CWPO of phenol. The Cu/RC catalyst showed a good activity after five successive runs (88% of degradation and 73% mineralization) at optimized conditions.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Editor Invitado, Special Issue "Preferential Oxidation of Carbon Monoxide", Catalysts (ISSN 2073-4344; CODEN: CATACJ), Open access Journal, MDPI, Suiza, 28/02/2019, http://www.mdpi.com/journal/catalysts/special_issues/CO-PROX
(M.A. Centeno, O.H. Laguna)

Editor Invitado, Special Issue "Catalysis by Precious Metals, Past and Future", Catalysts (ISSN 2073-4344; CODEN: CATACJ), Open access Journal, MDPI, Suiza, 31/07/2019, http://www.mdpi.com/journal/catalysts/special_issues/Precious
(M. Martínez T., S. Ivanova)

Editor Invitado, Special Issue "CO2, a Carbon Source for Chemicals and Fuels", Reactions (ISSN 2624-781X), Open access Journal, MDPI, 31/03/2019, http://www.mdpi.com/journal/reactions/special_issues/CO2_chemicals_fuels (J.A. Odriozola, O.H. Laguna)

Editor Invitado, Research Topic " Catalysis by Gold for Gas & Liquid Phase Reactions: A Golden Future for Environmental Catalysis ", Frontiers in Chemistry (ISSN: 2296-2646), Open access Journal, 2019, https://www.frontiersin.org/journals/chemistry# (S. Ivanova)

Editor Invitado, Research Topic " Women in Science: Chemistry ", Frontiers in Chemistry (ISSN: 2296-2646), Open access Journal, 2019-2020, https://www.frontiersin.org/journals/chemistry# (S. Ivanova)

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

6th International Conference on Structured Catalysts and Reactors, ICOSCAR-6
11 – 13 septiembre [Bad Herrenalb, Alemania]
José Antonio Odriozola (Miembro Comité Científico)

COMUNICACIONES / COMMUNICATIONS

IV Eurasian Conference on Civil and Environmental Engineering, IV ECOCEE
17 – 18 junio [Estambul, Turquia]

Photocatalytic degradation of methylene blue in water using platinum doped TiO_2 catalysts
Z. Fandi, N. Ameur, S. Bedrane, R. Bachir, A. Choukchou-Braham, C.A. Jaramillo-Páez, J.A. Navio
Poster

Preparation of Au/TiO_2 catalysts and study of their photocatalytic activity for the degradation of methyl orange
Z. Fandi, N. Ameur, S. Bedrane, R. Bachir, A. Choukchou-Braham, C.A. Jaramillo-Páez, J.A. Navio
Poster

6th European Conference on Environmental Applications of Advanced Oxidation Processes EAAOP-6
23 – 30 junio [Portoroz, Eslovenia]

Role of Ag-Doping on the Enhanced Degradation and Disinfection Efficiency of TiO2 and ZnO under Solar Radiation
S.D. Jojoa-Sierra, C. Jaramillo-Páez, M.C. Hidalgo, R.A. Torres-Palma, M.P. Ormad, J.A. Navío, R. Mosteo
Comunicación oral

14th European Congress on Catalysis, Europacat 2019

18 – 23 agosto [Aachen, Alemania]

"Making cheaper bio-oil" via hydrogen free HDO reactions using noble metal based catalysts supported on activated carbon
J.L. Santos, J. Wei, L. Pastor-Perez, M.A. Centeno, T. R. Reina
Conferencia invitada

Induced effect of residual species in the reaction intermediates during CO2 hydrogenation on Ru nanoparticles
S. Navarro-Jaén, A. Szego, L.F. Bobadilla, O.H. Laguna, F. Romero-Sarria, M.A. Centeno, J.A. Odriozola
Comunicación oral

Gold catalysts screening for reverse water gas shift reaction
L. Blandon, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

Glucose dehydration to valuable chemicals over functionalized carbon catalysts promoted by calcium chloride
Ch. Bounoukta, S. Ivanova, F. Ammari, M.A. Centeno, J.A. Odriozola
Poster

Catalytic reforming of model biomass-derived producer gas
L. Azancot, L.F. Bobadilla, M.A. Centeno, J.A. Odriozola
Poster

6th International Conference on Structured Catalysts and Reactors, ICOSCAR-6

11 – 13 septiembre [Bad Herrenalb, Alemania]

On the kinetics of CCVD synthesis of graphene related materials over stainless steel foams
F. Cazaña, P. Tarifa, N. Latorre, V. Sebastián, E. Romeo, M.A. Centeno, A. Monzón
Poster

Gas-phase mild amine oxidation on structured carbon-based catalysts
B. Jurca, L. Zavaleanu, J.A. Odriozola, M.A. Centeno, V.I. Parvulescu
Poster

Effect of the modification of the fluid dynamics during Sabatier reaction over metallic micromonoliths
J.C. Navarro, M.A. Centeno, O.H. Laguna, J.A. Odriozola
Poster

CO_2 hydrogenation through the Sabatier reaction using metallic micromonoliths
J.C. Navarro, L.M. Tejada, M.A. Centeno, O.H. Laguna, J.A. Odriozola
Poster

XI Simposio Colombiano de Catálisis
25 – 27 septiembre [Cauca, Colombia]

Propiedades fotocatalíticas de nanoplaquetas de TiO2 modificadas por fotodeposición de plata
César Jaramillo, José A. Navío, Pablo Sánchez, Felipe Puga, María C. Hidalgo
Comunicación oral

IX AUSE CONGRESS and IV ALBA USER'S MEETING
8 – 11 octubre [Barcelona, España]

Operando IR spectroscopy applied to high-pressure reactions for innovative catalytic research
L.F. Bobadilla
Conferencia invitada

8th International Conference on Carbons for Energy Storage and Environment Protection, CESEP´19
20 – 24 octubre [Alicante, España]

Noble metals supported on mesoporous biochar as a suitable catalyst for upgrading bio oils
J.L. Santos, S. Ivanova, Päivi Mäki-Arvela, Dmitry Yu. Murzin, M.A. Centeno
Comunicación oral

Biomass upgrading via H2-free HDO using highly effective carbon-based catalysts
J.L. Santos, J. Wei, L. Pastor-Perez, M.A. Centeno, T. R. Reina
Comunicación oral

XVI Congreso Mexicano de Catálisis cardenas
10 – 15 noviembre [Tabasco, Mexico]

Evaluación del método de síntesis en la formación de partículas de ZnO y su aplicación fotocatalítica
Y.N. Bolaina, M. Álvarez Lemus, R. López-González, P. Quintana, M.C. Hidalgo, J.A. Navío
Comunicación oral

Nanoestructuras de dióxido de titanio soportadas en membranas poliméricas para su aplicaciónen fotocatálisis
M. Uribe, M. Álvarez Lemus, M.C. Hidalgo, P. Quintana, R. López-González, S. Uribe
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

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

Reunión de la Sociedad Española de Catálisis SECAT 2019
24 – 26 junio [Córdoba, España]
José Antonio Odriozola (Miembro Comité Científico)
Miguel Angel Centeno Gallego (Miembro Comité Científico)

COMUNICACIONES / COMMUNICATIONS

12th International Symposium of the Romanian Catalysis Society, RomCat 2019
5 – 7 junio [Bucarest, Rumania]

Overruling the size effect of nanocatalysts: the role of the interface
J.A. Odriozola
Conferencia plenaria

Biocarbon for biorefinery reaction
C.E. Bounoukta, G. Delgado, S. Ivanova, A. Monzón, J.A. Odriozola, M.A. Centeno
Conferencia invitada

Structured carbocatalysts with hierarchical graphene layers active for aliphatic amines gas-phase O2 mild oxidation processes: a kinetic study
B. Jurca, L. Zavaleanu, M.A. Centeno, J.A. Odriozola, V.I. Parvulescu
Poster

Escuela de Verano SECAT 2019: Avances en Catálisis Orgánica
24 – 26 junio [Córdoba, España]

Formic acid as energetic vector
M.A. Centeno

Conferencia invitada

Reunión de la Sociedad Española de Catálisis SECAT 2019

24 – 26 junio [Córdoba, España]

Catalizadores bimetálicos PdAu/C soportados sobre carbón para la producción de hidrógeno a partir de ácido fórmico
J.L. Santos, C. León, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

Economically viable biomass upgrading: hydrogen free HDO reactions using noble metal based Catalysts supported on activated carbon
J.L. Santos, J. Wei, L. Pastor-Pérez, M.A. Centeno, T.R. Reina
Comunicación oral

Estudio espectrocópico operando DRIFTS-MS del efecto promotor del potasio sobre un catalizador Ni-K/MgAl2O4 en la reacción de reformado seco de metano
L. Azancot, L.F. Bobadilla, F. Romero-Sarria, M.A. Centeno, J.A. Odriozola
Comunicación oral

Exfoliación de grafito expandido en fase líquida mediante sonicación en presencia de surfactantes
G. Delgado, F. Cazaña, M.I. Domínguez, S. Ivanova, A. Monzón, M.A. Centeno
Comunicación oral

Hidrogenación del dióxido de carbono a partir de la reacción de Sabatier empleando catalizadores estructurados metálicos
J.C. Navarro, L.M. Tejada, M.A. Centeno, O.H. Laguna, J.A. Odriozola
Comunicación oral

La estructura cristalina de la espinela MgAl2O4 como condicionante de la actividad de catalizadores MN/MgAl2O4 (MN:Rh y Pt) en reformado de biogás
V. Garcilaso, J.M. Córdoba, M.A. Centeno, J.A. Odriozola
Poster

Reformado catalítico de los efluentes gaseosos obtenidos a partir de la gasificación de biomasa: efecto de la interacción Ni-K
L. Azancot, L.F. Bobadilla, M.A. Centeno, J.A. Odriozola
Poster

Estudio operando DRIFTS de la separación CH4/CO2 sobre materiales SAPO-34: efecto de la relación Si/Al/P
M. Romero, J.C. Navarro, L.F. Bobadilla, S. Ivanova, M.I. Domínguez, F. Romero-Sarria, M.A. Centeno, J.A. Odriozola
Poster

Conversión de glucosa en ácido levulínico sobre catalizadores funcionalizados de carbón en presencia de cloruro cálcico
Charf Eddine Bounoukta, F. Ammari, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

Catalizadores de oro soportado para la reacción de desplazamiento del gas de agua inversa (RWGS)
L. Blandón, S. Ivanova, A. Penkova, M.A. Centeno, J.A. Odriozola
Poster

36 Jornadas Nacionales de Ingeniería Química
4 – 6 septiembre [Zaragoza, España]

Eliminación de la Actividad Antibiótica de una Fluoroquinolona de Extenso Uso Farmacológico mediante Fotocatálisis con ZnO Modificado con Ag
S.D. Jojoa-Sierra, C. Jaramillo-Páez, M.C. Hidalgo, J.A. Navío, M.P. Ormad, R.A. Torres-Palma, R. Mosteo
Comunicación oral

XVI Congreso Mexicano de Catálisis cardenas
10 – 15 noviembre [Tabasco, Mexico]

Evaluación del método de síntesis en la formación de partículas de ZnO y su aplicación fotocatalítica
Y.N. Bolaina, M. Álvarez Lemus, R. López-González, P. Quintana, M.C. Hidalgo, J.A. Navío
Comunicación oral

Nanoestructuras de dióxido de titanio soportadas en membranas poliméricas para su aplicaciónen fotocatálisis
M. Uribe, M. Álvarez Lemus, M.C. Hidalgo, P. Quintana, R. López-González, S. Uribe
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Phosphate-based catalysts for the WGS reaction: synthesis, reactivity and mechanistic considerations**
Autor:	Sara Navarro Jaén
Directores:	Oscar H. Laguna Espitia y José Antonio Odriozola Gordón
Calificación:	Sobresaliente “Cum Laude”

Centro: Universidad de Sevilla
Fecha: 21 de enero de 2019

Título: **Valorización de biomasa residual: Biocarbones como soportes catalíticos**
Autor: José Luis Santos Muñoz
Directores: Miguel Angel Centeno y José Antonio Odriozola Gordón
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla
Fecha: 27 de septiembre de 2019

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Recubrimientos anti-rayadura para lentes oftálmicas**
Autor: Diego Bermúdez Domínguez
Directores: Victor Lopez Flores y Leidy Marcela Martínez Tejada
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (4 julio 2019)

Título: **Nanopartículas magnéticas para hipertermia en tratamientos contra el cáncer**
Autor: Cristina Suárez Rufo
Directores: Victor Lopez Flores y Leidy Marcela Martínez Tejada
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (4 julio 2019)

Título: **Aprovechamiento de CO2: Catalizadores para la reacción inversa de desplazamiento del gas de agua (RWGS)**
Autor: Laura Blandón Évora
Directores: Svetlana Lyuomirova Ivanova
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (9 julio 2019)

Título: **Estructuras fotónicas multicapa para la detección selectiva de compuestos orgánicos volátiles**
Autor: Hermine Berthon
Directores: Anna Dimitrova Penkova y Agustín R. González-Elipe
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (9 julio 2019)

Título:	**Synthesis and characterization of three-dimensional ITO nanoelectrodes**
Autor:	Javier Castillo Seoane
Directores:	Ana Isabel Borras Martos, Leidy Marcela Martínez Tejada y Jorge Gil Rostra
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (9 julio 2019)

Título:	**Láminas delgadas nanoestructuradas mediante aplicación de ondas acústicas superficiales**
Autor:	José Andrés Espino Román
Directores:	Anna Dimitrova Penkova y Agustín R. González-Elipe
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (9 julio 2019)

Título:	**Valorización Catalítica de la biomasa**
Autor:	Vicente Sánchez Cabrera
Directores:	José Antonio Odriozola Gordón, Oscar H. Laguna Espitia y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (9 julio 2019)

Título:	**Catalizadores estructurados en base a Pt para la reacción de Water Gas Shift**
Autor:	Adrián Megías Sánchez
Directores:	Svetlana Lyuomirova Ivanova y María Isabel Domínguez Leal
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (9 julio 2019)

Título:	**Producción de syngas mediante reformado catalítico de biogás**
Autor:	Maria Ángeles Corrales Bernabé
Directores:	Leidy Marcela Martínez Tejada y José Antonio Odriozola Gordón
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (12 julio 2019)

Título:	**Nanopartículas de metales nobles soportadas**
Autor:	Claudia León Fernández
Directores:	Svetlana Lyuomirova Ivanova
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (12 julio 2019)

Título:	**Catalizadores basados en carbón: Estudio de la estructuración del soporte y el depósito de la fase activa**
Autor:	Beatriz María López Rodríguez
Directores:	Leidy Marcela Martínez Tejada y María Isabel Domínguez Leal
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (19 septiembre 2019)

Título:	**CO2 valorization using structured catalysts**
Autor:	Xiwen Zhang
Directores:	Luis F. Bobadilla Baladrón y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (20 septiembre 2019)

Título:	**Catalizadores Pd/C para la producción de hidrógeno a partir de la descomposición de ácido fórmico**
Autor:	Salvador Moreno García
Directores:	Miguel Ángel Centeno Gallego y María Isabel Domínguez Leal
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (20 septiembre 2019)

Título:	**Propiedades fotocatalíticas de un ZnO optimizado y modificado mediante la deposición de malaquita. Influencia del Cu(II) y procesos Foto-Fenton**
Autor:	Pablo Miguel Sánchez-Cid Bueno
Directores:	José Antonio Navio Santos y Mª Carmen Hidalgo López
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (20 septiembre 2019)

Título:	**Síntesis y caracterización de zeolitas para su uso como catalizadores heterogéneos**
Autor:	Ligia Amelia Luque Alvarez
Directores:	Luis F. Bobadilla y Francisca Romero Sarria
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (23 septiembre 2019)

Título:	**Materiales para la Biorefinería: obtención de Moléculas Plataforma de Alto Valor Añadido**
Autor:	Aitana María Sollo Doña
Directores:	Svetlana Lyuomirova Ivanova
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (24 septiembre 2019)

Título:	**Preparación y caracterización de carbones mesoporosos obtenidos por técnicas de nanocasting**
Autor:	María Manuela Ortega Franqueza
Directores:	María Isabel Domínguez Leal y Miguel Ángel Centeno Gallego
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (24 septiembre 2019)

Título:	**Síntesis y caracterización morfológica y estructural de partículas coloidales**
Autor:	Maria Escamilla Rebollo
Directores:	José Antonio Navio Santos y Nuria Ofelia Núñez Álvarez
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (24 septiembre 2019)

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

University of Surrey		
Reino Unido	**José Antonio Odriozola Gordon**	1/7/2019 al 30/9/2019
Huazhong University of Science and Technology		
China	**José Antonio Odriozola Gordon**	4/10/2019 al 3/11/2019

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS
PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Universidad Cooperativa de Colombia		
Medellin, Colombia	**Oscar Felipe Arbelaez Pérez**	1/07/2019 al 12/07/2019
Universitá Degli Studi di Salerno		
Salerno, Italia	**Luigi Panzone**	24/09/2018 al 10/02/2019
	Daniela Imperiale	01/10/2019 al 10/03/2020

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 Angel Castro Arroyo
Dr. Manuel Jiménez Melendo
Dra. Pilar Malet Maenner
Dr. Julián Martínez Fernández

Científicos Titulares

Dr. José Jesús Benítez Jiménez
Dra. María Dolores Alba Carranza

Profesores Titulares

Dr. Alfonso Bravo León
Dra. Caroline M. Clauss Klamp (hasta septiembre)
Dr. Joaquin Ramírez Rico

Personal Contratado

Dra. Esperanza Pavón González

Becarios Predoctorales

Ldo. Francisco Javier Osuna Barroso (hasta marzo)

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Nuevo concepto de caldera de biomasa basada en materiales biocerámicos y combustión porosa para operación eficiente con residuos

Bioceramic Materials for New Biomass Domestic Bolier Concept based on Porous Combustion for a Wide Biomass/Residues Feedstock

Código/Code:	MAT2016-76526-R
Periodo/Period:	30-12-2016 / 29-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	60.500 €
Investigador responsable/Research head:	Joaquin Ramírez Rico /Ricado Chacartegui
Componentes/Research group:	Manuel Jiménez Melendo, Julián Martínez Fernández

RESUMEN / ABSTRACT

En la Unión Europea se generan anualmente más de cinco toneladas de residuos por personas, siendo aproximadamente el 60% de esta cantidad materia orgánica. La tecnología de las calderas de biomasa actuales no permiten el uso de estos residuos funcionando con altos valores de eficiencia, bajas emisiones y alta fiabilidadad de operación, siendo especialmente relevante en calderas de pequeño tamaño.

El principal objetivo de este proyecto es desarrollar un nuevo concepto de tecnología para calderas de biomasa doméstica capaz de operar con gran variedad de mezclas de biomasas y residuos agrícolas. Para ello se aprovecharán las sinergias de la integración de investigadores del: i) Grupo Maquinas y Motores Térmicos, GMTS, especialistas en combustión, calderas y máquinas térmicas ii) Grupo Materiales Biomiméticos y Multifuncionales, MBM, especialistas en la obtención de cerámicos porosos bioderivados, así como en caracterización físico-química y microestructural. El proyecto se completa con la colaboración de empresas en la evaluación de la tecnología y su aplicabilidad industrial En la Unión Europea se generan anualmente más de cinco toneladas de residuos por personas, siendo aproximadamente el 60% de esta cantidad materia orgánica. La tecnología de las calderas de biomasa actuales no permiten el uso de estos residuos funcionando con altos valores de eficiencia, bajas emisiones y alta fiabilidadad de operación, siendo especialmente relevante en calderas de pequeño tamaño.

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El proyecto se basa en la innovadora integración de material biocerámico microporoso en las cámaras de combustión de calderas de biomasa de modo que actúen con diferentes funciones: combustor microporoso, filtro de partículas y recuperador de calor. Estas funcionalidades pueden ser simultáneas, en función de la región del flujo en que se encuentren y el rango de temperaturas de esa región. Este material biocerámico es desarrollado a partir de precursores vegetales para obtener elementos de Carburo de Silicio (SiC). Para ello se usan materiales locales sin tratar, produciendo elementos hechos a medida con propiedades microestructurales adecuadas para trabajar con altas temperaturas. Así, productos con geometrias complejas pueden ser obtenidos con relativamente bajo coste comparados con otros materiales con características macánicas y químicas similares. La integración de componentes basados en estos materiales posibilita nuevos diseños de calderas de biomasa con un alto control de la combustión, las temperaturas y la emisión de partículas. El nuevo diseño evita la sinterización y fusión de las cenizas, actuando en la formación y evolución de contaminantes, inhibiendo los mecanismos de producción de dioxinas y activando la completa oxidación del monóxido de carbono (CO) y soots. El nuevo concepto permitirá la operación con una importante varieda de mezclas biomasa/ residuos agrícolas con bajas emisiones aun cuando el combustible pesente un alto contenido de cenizas, resolviendo el principal reto para el desarrollo del uso residuos agrícolas en calderas de biomasa (especialmente las de menor tamaño). El desarrollo de esta tecnología permitirá ampliar los recursos de la Unión Europea para calefacción de uso doméstico. En la actualidad este uso supone un 30% del consumo energético total en la misma. La propuesta incluye el estudio de los procesos básicos de combustión, flujos, fabricación a medida de las matrices de materiales biocerámicos, así como estudio y desarrollo de prototipos de componentes y del sistema final. Estos serán estudiados a nivel de ensayos de laboratorio con residuos agrícolas, forestales y de la industria olivarera.

EU generates more than five tons of waste per person every year and about 60 % is organic waste. Current biomass domestic boiler technology does not allow the use of these residues with high efficiency, ultra-low emissions and high reliability operation. The main objective of this proposal is the development of a new concept of biomass domestic boiler technology able to combine these characteristics for operation with multiple biomass/residues blends. It is based on the integration of novel bioceramic porous materials matrices in combustion chamber and gases pathflow with functions as microporous combustors, particles filters and heat accumulators. These functions are simultaneous depending on the region of the boiler. Matrices of bioceramic materials are developed from wood precursors to obtain SiC elements through a process patented by the University of Seville. It uses local raw material, and produces parts with tailor made microstructure/properties, adequate for high temperature and reactive operation. Products with complex geometries can be obtained at relatively low cost compared with other materials of similar chemical and mechanical properties. The integration of components based on these materials allows new designs of biomass boilers with high control of combustion, temperature and particle emission. It avoids ash sintering and melting, acting on the formation and evolution mechanisms of ash and dioxins and activating the complete oxidation of CO and soots. The new concept allows the operation to a wider biomass/residues feedstock with low emissions and low maintenance even with fuels with high ash content, produced from many residues, solving main challenges for their extended use and increasing the European fuel resources for domestic heating. Domestic heating in Europe consumes 30% of the total energy. The proposal includes prototypes development, fuel supply characteristics and preparation (geometry, compactness, composition, etc.) and combustion products management.

Biomass/residues blends from agriculture, forestry, olive oil industry among others will be tested both in laboratory.

Tratamiento sostenible de residuos industriales: Materiales adsorbentes de diseño y bionanocomposites en la inmovilization de metales pesados y productos de fision **Susteinable industrial waste treatment: designed adsorbent materials and bionanocomposites for inmobilizing heavy metals and fision products**

Código/Code:	MAT2015-63929-R
Periodo/Period:	01-01-2016 / 31-06-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	60.500 €
Investigador responsable/Research head:	Maria Dolores Alba Carranza
Componentes/Research group:	Miguel Angel Castro Arroyo, Ana Carmen Perdigón Aller, María del Mar Orta Cuevas

RESUMEN / ABSTRACT

El Proyecto de investigación que se presenta aborda la exigencia tecnológica y medioambiental de desarrollar metodologías avanzadas para la eliminación de agentes contaminantes. El interés y los esfuerzos encaminados al desarrollo de nuevas tecnologías orientadas a tratamientos más eficientes en la inmovilización y revalorización de los residuos peligrosos es creciente en los planes de I+D+i. El objetivo central del proyecto se basa en el diseño de una estrategia de funcionalización de silicatos laminares de alta carga expansibles y en la síntesis de bionanocomposites a partir de ellos para conseguir una actividad eficaz respecto de la inmovilización de residuos tóxicos y peligrosos, catiónicos y aniónicos. Este objetivo es un cambio cualitativo en el trabajo que se viene desarrollando en el diseño de sistemas modelos adsorbentes con aplicaciones medioambientales de clara repercusión en la mejora de la calidad de vida de la población y conservación del medioambiente, ya que la funcionalización de diseño de los silicatos sintéticos permitirá la adsorción de un amplio abanico de adsorbentes en estado de oxidación aniónico-catiónico. La finalidad y objetivos del proyecto se centran en la Focus Area WASTE de H2020 y esta Focus Area se desarrolla dentro del reto 2 y 5 de H2020 y dentro del Reto 5 y 3 de los Planes Estatales de Investigación.

El proyecto ha despertado el interés de diversas empresas observadoras, EPOs, (ENRESA y la Agencia de la Energía y para la Sostenibilidad del Ayuntamiento de Sevilla) impulsando la colaboración público-privada. Por tanto, la investigación desarrollada auna los principios básicos de la estrategia estatal de Ciencia y Tecnología: Poner la I+D+I al servicio de la ciudadanía, del bienestar social y de un desarrollo sostenible, hacer de la I+D+I un factor de mejora de la competitividad empresarial (transferencia de los resultados al sector privado, ver interes de los

EPOs) y reconocer y promover la I+D como un elemento esencial para la generación de nuevos conocimientos de excelencia.

La viabilidad de la propuesta se garantiza porque el equipo de investigación, EI, por un lado, ha desarrollado con éxito la síntesis de silicatos laminares hidratables de alta carga, a través de un método novedoso que permite ajustar la carga deseada para el material, y, por otro, ha conseguido exitosamente su organofuncionalización (patente ES2362597B1). Además, ha desarrollado la metodología necesaria para el correcto progreso de este proyecto, en estrecha colaboración con otros Grupos de investigación internacionales de reconocido prestigio (e.g. CNRS-Universidad de Lille, Universidad de Cambridge...). Además el EI ha demostrado que potencia la agrupación de las capacidades y competencias científico-técnicas esenciales para abordar esta propuesta de marcado caracter transversal.

The focus of the project addresses the requirement of advanced environmental technology methodologies for removing pollutants. Recently, the interest and efforts to develop new technologies for more efficient treatments for the immobilization and the revaluation of hazardous waste are increasing in R & D plans. The overall object of the project is based on the design of a strategy of functionalization of highly charged swelling phyllosilicates and their later transformation on bionanocomposite for the effective retention and immobilization of hazardous waste, both cationic and anionic. This object represents a qualitative change in the work that is being nowdays developed in the field of model adsorbents systems with environmental applications that will improve the quality life of the population and the environmental conservation, because the designed functionalization of the synthetic silicates will allow the adsorption of a wide range of adsorbents in different oxidation states, cationic or anionic. The objectives are conformed to the Focus Area WASTE of the H2020 program and it is developed on the 2nd and 5th challenge of the H2020 program and on the 5th and 3rd challenge of the national research program.

The project has attracted interest from various observers companies, EPOs, (ENRESA and the Water and Local Energy Agency and Sustainability of the City of Seville), the public-private collaboration being promoted. Therefore, the research combines the basic principles of the National Strategy of Science and Technology: Putting the R&D&I at the service of citizens, social welfare and sustainable development, making the R&D&I a factor of improving business competitiveness (transfer of results to the private sector, see interest of EPOs) and recognize and promote R&D&I as an essential element for the generation of new excellence knowledge.

The viability of the proposal is ensured, first, because the research team, RT, has accomplished the synthesis of hydratable high charged phyllosilicates, with a novel and original method that allows setting the material desired charge, and, later, has successfully achieved their organofunctionalization (patent ES 2 362 597 B1). Second, the RT has developed the required methodology for the development of this project in closed scientific collaboration with other well recognized international groups (i.e. CNRS-University of Lille, University of Cambridge...). The RT enhances the clustering of their capabilities and scientific-technical skills which are essential to address this proposal with a remarkable transverse character.

Estudio de la inmovilización de metales pesados por micas de alta carga sintéticas organofuncionalizadas: pruebas a escala de laboratorio / Inmobilization of heavy metals by synthetic high-charged organomicas: Test at laboratory scale

Código/Code:	P12-FQM-567
Periodo/Period:	16-05-2014 / 16-02-2019
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	174.455 €
Investigador responsable/Research head:	María Dolores Alba Carranza

RESUMEN / ABSTRACT

El tema central del proyecto aborda la exigencia tecnológica mediambiental de desarrollar metodologías avanzadas para la eliminación de agentes contaminantes. El interés y los esfuerzos encaminados al desarrollo de nuevas tecnologías orientadas a tratamientos más eficientes en la inmovilización y revalorización de los residuos peligrosos es crecientes en los planes de I + D + i de los últimos años. Es en este escenario donde debe encuadrarse el presente proyecto y en concreto en el marco de la gestión de cationes de metales pesados, tema de elevado interés social en la presente década.

Desde la segunda mitad del siglo XX la Humanidad se ha enfrentado a un enorme desarrollo científico y tecnológico que es el responsable de un incremento de la contaminación mediambiental. Como ejemplo podemos mencionar dos problemas que en la actualidad son motivos de preocupación y actuación de la Junta de Andalucía: contaminación de los litorales andaluces y las aguas residuales urbanas. Por tanto, estamos ante un problema complejo en el que los agentes contaminantes son variados, las fuentes de procedencia son diversas y las vías o rutas seguidas por los distintos contaminantes, frecuentemente, escapan al control necesario para evitar efectos indeseados sobre el entorno natural y urbano. Es por ello, que se demanda una investigación a nivel básico y aplicado de los mecanismos necesarios para la inmovilización de dichos cationes nocivos.

Los objetivos y alcance de este proyecto se basan en los avances llevados a cabos por otros grupos de investigación de la gestión de estos tipos de contaminantes y en los últimos resultados de la investigación llevada a cabo por el equipo de investigación que han permitido el diseño de silicatos laminares expansibles de alta carga con especiales propiedades como precursores para la retención de residuos nocivos. Por tanto, se propone en este proyecto la organofuncionalización de dichas micas sintéticas con grupos tioles o con cationes de alquilamonio de longitud de cadena variable y la evaluación de su capacidad de adsorción y retención irreversible de metales pesados.

The focus of the project addresses the environmental technological requirement to develop advanced methods for removing pollutants. The interest and efforts to develop new technologies aimed at more efficient treatment in detention and revaluation of hazardous waste is increasing in R & D plans. It is in this scenario where this project should be framed and in

particular in the framework of the management of heavy metal cations, issue of high public interest in this decade.

Since the second half of the twentieth century, humanity has faced a huge scientific and technological development that is responsible for increased environmental pollution. As an example, we can mention two problems that are currently of concern and action of the Andalusian: Andalusian coastal pollution and urban wastewater. Therefore, this is a complex problem that pollutants sources are varied of origin and routes followed by various pollutants are diverse and, frequently, it is beyond the control necessary to avoid urban undesirable effects on the natural environment and. Therefore, a basic level research is demaned to implement the necessary mechanisms for the immobilization of such harmful cations.

The objectives and scope of this project are based on advances made by other research groups in the management of these types of contaminants and the latest research conducted by the research team that allowed design expandable high-charged layered silicates with special properties as precursors for the retention of harmful residues. Therefore, it is proposed in this project the organofunzionalization of such synthetic micas with thiol groups or alkylammonium cations of varying chain length and evaluation of its adsorption capacity and irreversible retention of heavy metals.

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)
Componentes/Research group:	José Jesús Benítez, Luz Divina Gómez Pulido (IHSM), Manuel León Camacho (IG)

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

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

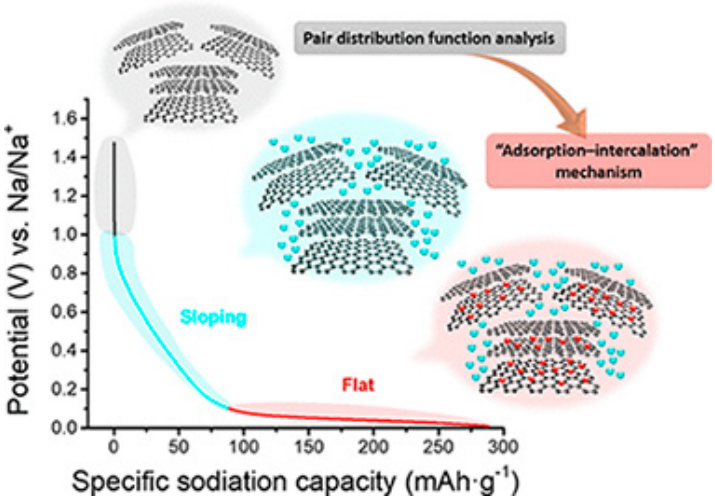
Desarrollo de Materiales compuestos zeolitas-enzimas fúngicas para la remoción de contaminantes emergentes de residuales farmacéuticos.

Código/Code:	COOPA20190
Periodo/Period:	01-01-2018 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad (Programa CSIC de Cooperación Científica para el desarrollo I-COOP+)
Importe total/Total amount:	20.000 €
Investigador responsable/Research head:	María Dolores Alba Carranza
Componentes/Research group:	Tania Farrias Piñeras, Rosa Meneau Hernández

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Correlation of Structure and Performance of Hard Carbons as Anodes for Sodium Ion Batteries

Gomez-Martin, A, Martinez-Fernandez, J, Ruttert, M, Winter, M, Placke, T, Ramirez-Rico, J
Chemistry of Materials, **31** (2019) 7288-7299
Septiembre, 2019 | **DOI:** 10.1021/acs.chemmater.9b01768

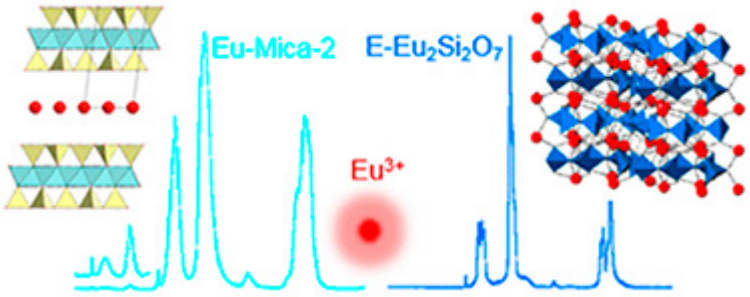


Hard carbons are the material of choice as negative electrode in sodium ion batteries. Despite being extensively studied, there is still debate regarding the mechanisms responsible for storage in low- and high-potential regions. This work presents a comprehensive approach to elucidate the involved storage mechanisms when Na ions insert into such disordered structures. Synchrotron X-ray total scattering experiments were performed to access quantitative

information on atomic ordering in these materials at the nanoscale. Results prove that hard carbons undergo an atomic rearrangement as the graphene layers cross-link at intermediate temperatures (1200-1600 degrees C), resulting in an increase of the average interplanar distance up to 1400 degrees C, followed by a progressive decrease. This increase correlates with the positive trend in the reversible capacity of biomass-derived carbons when processed up to 1200-1600 degrees C due to an increased capacity at low potential (<= 0.1 V vs Na/Na+). A decrease in achievable sloping capacity with increasing heat-treatment temperature arises from larger crystalline domains and a lower concentration of defects. The observed correlation between structural parameters and electrochemical properties clearly supports that the main storage of Na ions into a hard-carbon structure is based on an adsorption-intercalation mechanism.

Eu^{3+} Luminescence in High Charge Mica: An In Situ Probe for the Encapsulation of Radioactive Waste in Geological Repositories

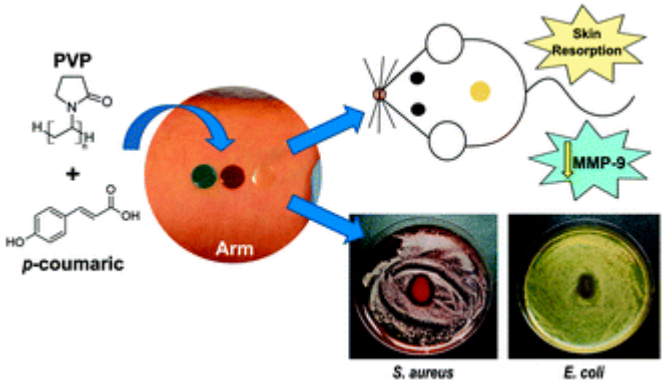
Martin-Rodriguez, R, Aguado, F, Alba, MD, Valiente, R, Perdigon, AC
ACS Applied Materials & Interfaces, **11** (2019) 7559-7565
Febrero, 2019 | **DOI:** 10.1021/acsami.8b20030



Isolation of high-level radioactive waste (HLW) in deep geological repositories (DGR) through a multibarrier concept is the most accepted approach to ensure long-term safety. Clay minerals are one of the most promising materials to be used as engineered barriers. In particular, high charge micas, as components of the engineered barrier, show superselectivity for some radioactive isotopes and a large adsorption capacity, which is almost twice that of the other low charge aluminosilicates. In addition, high charge micas are optimum candidates for decontamination of nuclear waste through two different mechanisms; namely an ion exchange reaction and a nonreversible mechanism involving the formation of new stable crystalline phases under hydrothermal conditions. In this work, we report a new in situ optical sensor based on the incorporation of Eu^{3+} in these high charge micas for tracking the long-term physical-chemical behavior of HLW contaminants in DRG under mild hydrothermal conditions. The incorporation of Eu3+ into the interlayer space of the mica originates a well resolved green and red luminescence, from both the 5D1 and 5D0 excited states, respectively. The formation of new crystalline phases under hydrothermal conditions involves important changes in the Eu3+ emission spectra and lifetime. The most interesting features of Eu^{3+} luminescence to be used as an optical sensor are (1) the presence or absence of the Eu3+ green emission from the 5D1 excited state, (2) the energy shift of the 5D0 → 7F0 transition, (3) the crystal-field splitting of the 7F1 Eu^{3+} level, and (4) the observed luminescence lifetimes, which are directly related to the interaction mechanisms between the lanthanide ions and the silicate network.

Combining dietary phenolic antioxidants with polyvinylpyrrolidone: transparent biopolymer films based on p-coumaric acid for controlled release

Contardi, M, Heredia-Guerrero, JA, Guzman-Puyol, S, Summa, M, Benitez, JJ, Goldoni, L, Caputo, G, Cusimano, G, Picone, P, Di Carlo, M, Bertorelli, R, Athanassioua, A, Bayer, IS
Journal of Materials Chemistry B, **7** (2019) 1384-1396
Marzo, 2019 | **DOI:** 10.1039/c8tb03017k

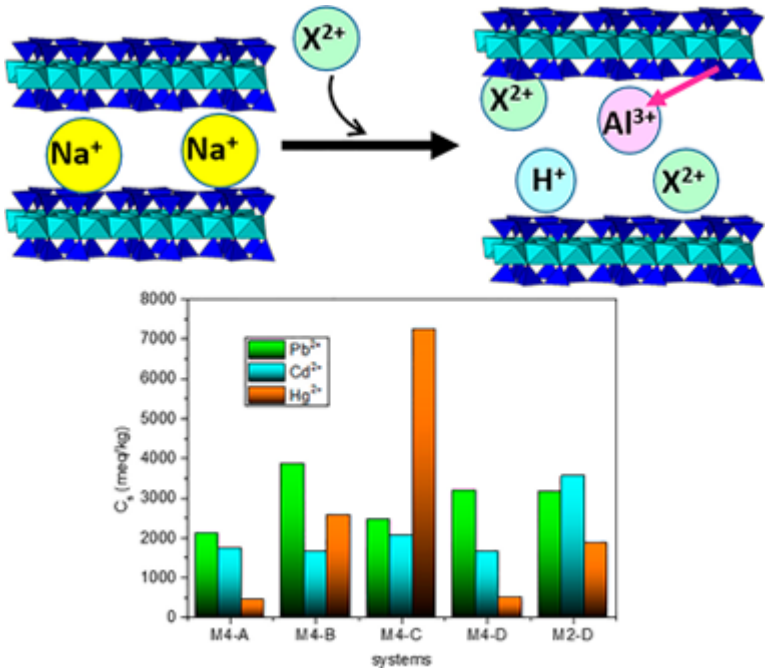


Polyvinylpyrrolidone (PVP) has probably been one of the most utilized pharmaceutical polymers with applications ranging from a blood plasma substitute to nanoparticle drug delivery, since its synthesis in 1939. It is a highly biocompatible, non-toxic and transparent film forming polymer. Although high solubility of PVP in aqueous environment is advantageous, it still poses several problems for some applications in which sustained targeting and release are needed or hydrophobic drug inclusion and delivery systems are to be designed. In this study, we demonstrate that a common dietary phenolic antioxidant, p-coumaric acid (PCA), can be combined with PVP covering a wide range of molar ratios by solution blending in ethanol, forming new transparent biomaterial films with antiseptic and antioxidant properties. PCA not only acts as an effective natural plasticizer but also establishes H-bonds with PVP increasing its resistance to water dissolution. PCA could be released in a sustained manner up to a period of 3 days depending on the PVP/ PCA molar ratio. Sustained drug delivery potential of the films was studied using methylene blue and carminic acid as model drugs, indicating that the release can be controlled. Antioxidant and remodeling properties of the films were evaluated in vitro by free radical cation scavenging assay and in vivo on a murine model, respectively. Furthermore, the material resorption of films was slower as PCA concentration increased, as observed from the in vivo full-thickness excision model. Finally, the antibacterial activity of the films against common pathogens such as Escherichia coli and Staphylococcus aureus and the effective reduction of inflammatory agents such as matrix metallopeptidases were demonstrated. All these properties suggest that these new transparent PVP/ PCA films can find a plethora of applications in pharmaceutical sciences including skin and wound care.

Design swelling micas: Insights on heavy metals cation exchange reaction

Osuna, FJ, Pavon, E, Alba, MD
Applied Clay Science, **182** (2019) 105298
Diciembre, 2019 | **DOI:** 10.1016/j.clay.2019.105298

Heavy metal pollution has become one of the most serious environmental problems, demanding specialized remediation mechanisms. Among the studied treatments, ion-exchange processes have been widely used due to their high remediation capacity, efficiency and fast kinetic. Here, the potential use of a new family of design micas as cation exchanger has been analysed. Micas with a layer charge in the range of brittle micas have been synthesized and their heavy metals



cation exchange capacity analysed as a function of the nature of the heavy metal cations (Pb2+, Cd2+ or Hg2+), the nature of the counterions (Cl− or NO3−), concentration of the solutions and the micas layer charge. A cation exchange ratio between 35% and 154% of their cation exchange capacity (CEC) was achieved, being more efficient when mica layer charge diminished. In general, the maximum adsorption capacity followed the trend: Hg2+ > Pb2+ > Cd2+. The efficiency of the cation exchange and adsorption mechanism of the synthetic micas depended on the experimental conditions and they were more efficient than raw and modified natural clay minerals.

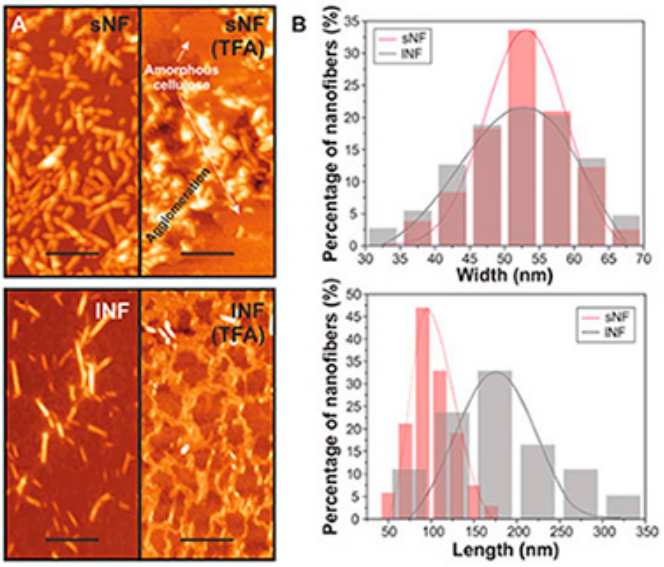
Transparent and Robust All-Cellulose Nanocomposite Packaging Materials Prepared in a Mixture of Trifluoroacetic Acid and Trifluoroacetic Anhydride

Guzman-Puyol, S, Ceseracciu, L, Tedeschi, G, Marras, S, Scarpellini, A, Benitez, JJ, Athanassiou, A, Heredia-Guerrero, JA

Nanomaterials, **9** (2019) 368
Marzo, 2019 | **DOI:** 10.3390/nano9030368

All-cellulose composites with a potential application as food packaging films were prepared by dissolving microcrystalline cellulose in a mixture of trifluoroacetic acid and trifluoroacetic anhydride, adding cellulose nanofibers, and evaporating the solvents. First, the effect of the solvents on the morphology, structure, and thermal properties of the nanofibers was evaluated by atomic force microscopy (AFM), X-ray diffraction (XRD), and thermogravimetric analysis

(TGA), respectively. An important reduction in the crystallinity was observed. Then, the optical, morphological, mechanical, and water barrier properties of the nanocomposites were determined. In general, the final properties of the composites depended on the nanocellulose content. Thus, although the transparency decreased with the amount of cellulose nanofibers due to increased light scattering, normalized transmittance values were higher than 80% in all



the cases. On the other hand, the best mechanical properties were achieved for concentrations of nanofibers between 5 and 9 wt.%. At higher concentrations, the cellulose nanofibers aggregated and/or folded, decreasing the mechanical parameters as confirmed analytically by modeling of the composite Young's modulus. Finally, regarding the water barrier properties, water uptake was not affected by the presence of cellulose nanofibers while water permeability was reduced because of the higher tortuosity induced by the nanocelluloses. In view of such properties, these materials are suggested as food packaging films.

Bionanocomposites based on chitosan intercalation in designed swelling high-charged micas

Alba, MD, Cota, A, Osuna, FJ, Pavon, E, Perdigon, AC, Raffin, F
Scientific Reports, **9** (2019) 10265
Julio, 2019 | **DOI:** 10.1038/s41598-019-46495-z

Bionanocomposites based on layered inorganic components, as clays, and polymers of biological origin, as chitosan, have a major impact in medical and environmental fields, being economical and environmentally friendly materials. Na-Mn micas (n = 2 and 4) with controlled surface charge, high cation exchange capacity and swelling behaviour, are attractive inorganic composite components that exhibit improved adsorption properties compared to other inorganic solids which makes them potentially useful for bionanocomposites. The goal of this research was to explore the potential use of those synthetic brittle micas to form eco-friendly bionanocomposites with chitosan biopolymer. Hence, chitosan-mica bionanocomposites were prepared by ion-exchange reaction between chitosan solution and synthetic high charge mica. X-ray diffraction, Fourier transform infrared spectroscopy, thermal analysis, MAS-NMR

spectroscopy and zeta-potential have been employed for bionanocomposites characterization. The results showed that the adsorption of chitosan is effective, although a chitosan portion remains in the outer surface being hydrogen-bonded to the tetrahedral sheet of the silicate.

Insoluble and Thermostable Polyhydroxyesters From a Renewable Natural Occurring Polyhydroxylated Fatty Acid

Benitez, JJ, Guzman-Puyol, S, Cruz-Carrillo, MA, Ceseracciu, L, Moreno, AG, Heredia, A, Heredia-Guerrero, JA
Frontiers in Chemistry, **7** (2019) 643
Septiembre, 2019 | **DOI:** 10.3389/fchem.2019.00643

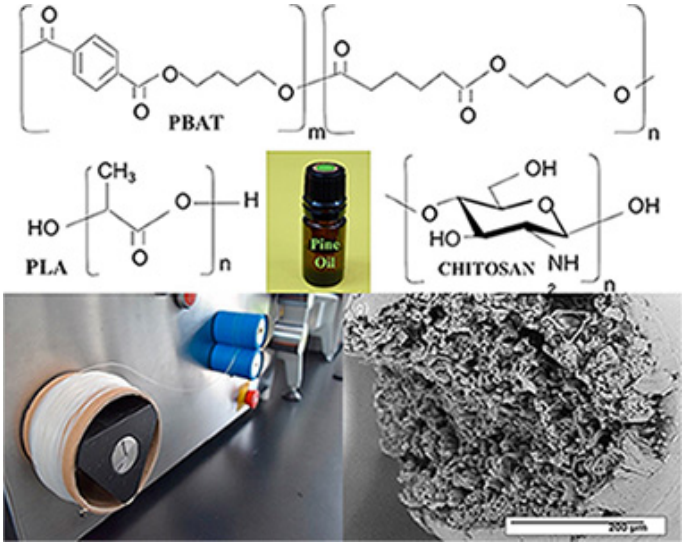
To explore the potential of long chain polyhydroxyalkanoates as non-toxic food packaging materials, the characterization of polyesters prepared from a natural occurring polyhydroxylated C16 carboxylic acid (9,10,16-trihydroxyhexadecanoic or aleuritic acid) has been addressed. Such monomer has been selected to elucidate the reactivity of primary and secondary hydroxyl groups and their contribution to the structure and properties of the polyester. Resulting polyaleuritate films have been produced using an open mold in one-step, solvent-free self-polycondensation in melt state and directly in air to evaluate the effect of oxygen in their final physical and chemical properties. These polymers are amorphous, insoluble, and thermostable, being therefore suitable for solvent, and heat resistant barrier materials. Structurally, most of primary hydroxyls are involved in ester bonds, but there is some branching arising from the partial participation of secondary O-H groups. The oxidative cleavage of the vicinal diol moiety and a subsequent secondary esterification had a noticeable effect on the amorphization and stiffening of the polyester by branching and densification of the ester bond network. A derivation of such structural modification was the surface compaction and the reduction of permeability to water molecules. The addition of Ti(OiPr)(4) as a catalyst had a moderate effect, likely because of a poor diffusion within the melt, but noticeably accelerated both the secondary esterification and the oxidative processes. Primary esterification was a high conversion bulk reaction while oxidation and secondary esterification was restricted to nearby regions of the air exposed side of cast films. The reason was a progressive hindering of oxygen diffusion as the reaction progresses and a self-regulation of the altered layer growth. Despite such a reduced extent, the oxidized layer noticeably increased the UV-vis light blockage capacity. In general, characterized physical properties suggest a high potential of these polyaleuritate polyesters as food preserving materials.

Bio-based composite fibers from pine essential oil and PLA/PBAT polymer blend. Morphological, physicochemical, thermal and mechanical characterization

Hernandez-Lopez, M, Correa-Pacheco, ZN, Bautista-Banos, S, Zavaleta-Avejar, L, Benitez-Jimenez, JJ, Sabino-Gutierrez, MA, Ortega-Gudino, P
Materials Chemistry and Physics, **234** (2019) 345-353
Agosto, 2019 | **DOI:** 10.1016/j.matchemphys.2019.01.034

Biodegradable aliphatic polyesters are an alternative to reduce the use of synthetic plastic materials that cause severe damage to the environment. Formulations based on poly (lactic acid) (PLA) and poly (butylene adipate-co-terephthalate) (PBAT), were mixed in a 60:40 ratio, adding

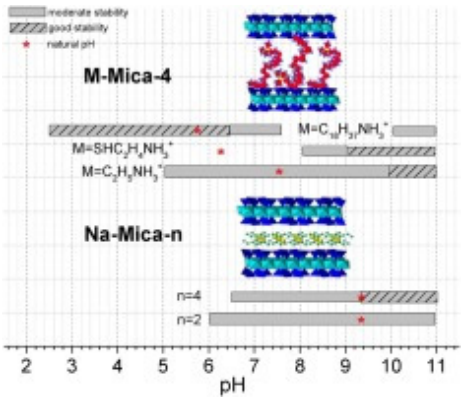
different concentrations of pine essential oil through the use of extrusion technology to obtain biodegradable polymer fibers. Some formulations were coated with chitosan. All the elaborated fibers were characterized by Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy-Attenuated Total Reflection (FTIR-ATR), Differential Scanning Calorimetry (DSC), X-ray Diffraction (XRD) and mechanical properties. The SEM studies showed that the PBAT



improves the tenacity and provides greater elasticity promoting the interaction between the blends phases with fibril formation. In the FTIR-ATR analysis, compatibility between the blends was observed due to a possible interaction of the carbonyl group of PBAT with PLA. The DSC and the mechanical properties showed partial miscibility of the blends, indicating, that the plasticizing action of the essential oil gave greater mobility, flexibility, less rigidity and crystallization in the blends. A lower Young's modulus and greater elongation at break was also observed.

Influence of framework and interlayer on the colloidal stability of design swelling high-charged micas

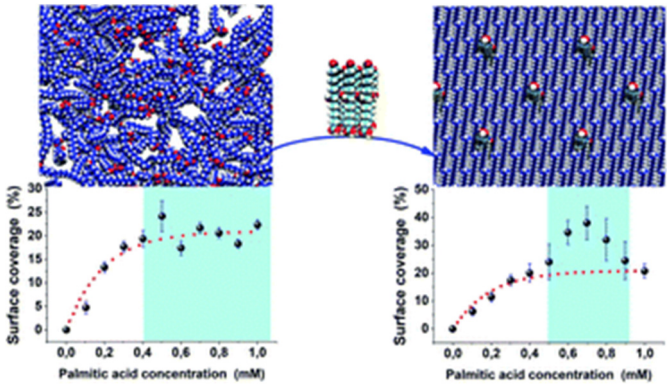
Osuna, FJ, Cota, A, Fernandez, MA, Pavon, E, Sanchez, RMT, Alba, MD
Colloids and Surfaces A: Physicochemical and Engineering Aspects, **561** (2019) 32-38
Enero, 2019 | **DOI:** 10.1016/j.colsurfa.2018.09.086



Stability of colloidal clay minerals dispersion of fundamental in many industrial processes. Therefore, surface charge of the synthetic swelling high-charged mica family, Na-Mica-n (n = 2 or 4, n is the total layer charge) were investigated to study its colloidal dispersion stability as a function of the framework and interlayer space composition. Na-Mica-n (n = 2 or 4) micas were synthesized and functionalized with ethylammonium, mercaptoethylammonium or octadecylammonium. Their zeta-potential evolutions as a function of solution pH were correlated with their structural, compositional and morphological characteristics. The results have shown that the total charge of swelling high charged micas, Mica-n, didn´t affect significantly their colloidal dispersion stability, the interlayer composition and interlayer cation arrangement were the main factors of colloidal behaviour. The synthesis and functionalization of those synthetic micas can be tuned for their optimal use.

Understanding segregation processes in SAMs formed by mixtures of hydroxylated and non-hydroxylated fatty acids

Bueno, OVM, Benitez, JJ, San-Miguel, MA
RSC Advances, **9** (2019) 39252-39263
Diciembre, 2019 | **DOI:** 10.1039/c9ra06799j

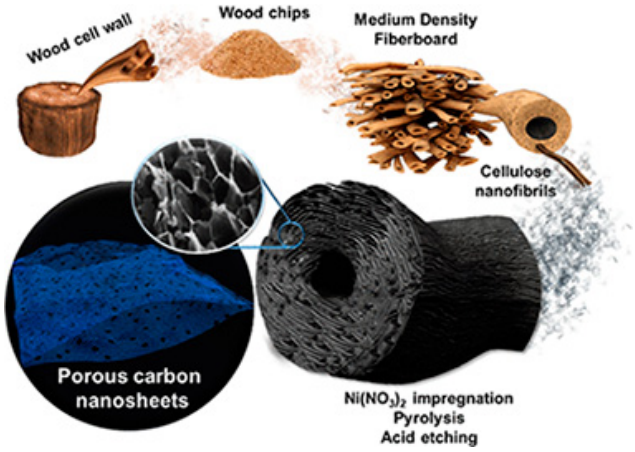


In this paper, we focus on the segregation processes emerging when preparing mixtures with different compositions of aleuritic (9,10,16 trihydroxyhexadecanoic) (ALE) and palmitic (hexadecanoic) (PAL) acids. The combination of atomic force microscopy (AFM) and molecular dynamics (MD) simulations enabled us to prove the role of the functional groups in the formation of self-assembled monolayers (SAMs) on muscovite mica surfaces. MD simulations indicate that segregation processes are favored in high ALE composition mixtures in agreement with the experimental evidence, whereas low ALE compositions promote the co-existence between segregated and dispersed systems. The secondary hydroxyl groups play a central role in the self-assembling mechanism because they control the formation of hydrogen bonding networks guarantying system stability.

Porous Graphene-like Carbon from Fast Catalytic Decomposition of Biomass for Energy Storage Applications

Gomez-Martin, A, Martinez-Fernandez, J, Ruttert, M, Winter, M, Placke, T, Ramirez-Rico, J
ACS Omega, **4** (2019) 21446-21458
Diciembre, 2019 | **DOI:** 10.1021/acsomega.9b03142

A novel carbon material made of porous graphene-like nanosheets was synthesized from biomass resources by a simple catalytic graphitization process using nickel as a catalyst for applications in electrodes for energy storage devices. A recycled fiberboard precursor was impregnated with saturated nickel nitrate followed by high-temperature pyrolysis. The highly exothermic combustion of in situ formed nitrocellulose produces the expansion of the cellulose



fibers and the reorganization of the carbon structure into a three-dimensional (3D) porous assembly of thin carbon nanosheets. After acid washing, nickel particles are fully removed, leaving nanosized holes in the wrinkled graphene-like sheets. These nanoholes confer the resulting carbon material with approximate to 75% capacitance retention, when applied as a supercapacitor electrode in aqueous media at a specific current of 100 A.g(-1) compared to the capacitance reached at 20 mA.g(-1), and approximate to 35% capacity retention, when applied as a negative electrode for lithium-ion battery cells at a specific current of 3720 mA.g(-1) compared to the specific capacity at 37.2 mA.g(-1). These findings suggest a novel way for synthesizing 3D nanocarbon networks from a cellulosic precursor requiring low temperatures and being amenable to large-scale production while using a sustainable starting precursor such as recycled fiberwood.

Low molecular weight epsilon-caprolactone-p-coumaric acid copolymers as potential biomaterials for skin regeneration applications

Contardi, M, Alfaro-Pulido, A, Picone, P, Guzman-Puyol, S, Goldoni, L, Benitez, J, Heredia, A, Barthel, MJ, Ceseracciu, L, Cusimano, G, Brancato, OR, Di Carlo, M, Athanassiou, A, Heredia-Guerrero, JA

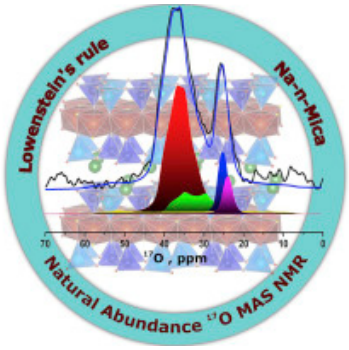
PLoS One, **14** (2019) e0214956
Abril, 2019 | **DOI:** 10.1371/journal.pone.0214956

epsilon-caprolactone-p-coumaric acid copolymers at different mole ratios (epsilon-caprolactone: p-coumaric acid 1:0, 10:1, 8:1, 6:1, 4:1, and 2:1) were synthesized by melt-polycondensation and using 4-dodecylbenzene sulfonic acid as catalyst. Chemical analysis by NMR and GPC showed that copolyesters were formed with decreasing molecular weight as p-coumaric acid content was increased. Physical characteristics, such as thermal and mechanical properties, as well as water uptake and water permeability, depended on the mole fraction of p-coumaric acid. The p-coumarate repetitive units increased the antioxidant capacity of the copolymers, showing antibacterial activity against the common pathogen Escherichia coli. In

addition, all the synthesized copolyesters, except the one with the highest concentration of the phenolic acid, were cytocompatible and hemocompatible, thus becoming potentially useful for skin regeneration applications.

Natural abundance O-17 MAS NMR and DFT simulations: New insights into the atomic structure of designed micas

Pavon, E, Osuna, FJ, Alba, MD, Delevoye, L
Solid State Nuclear Magnetic Resonance, **100** (2019) 45-51
Agosto, 2019 | **DOI:** 10.1016/j.ssnmr.2019.03.006

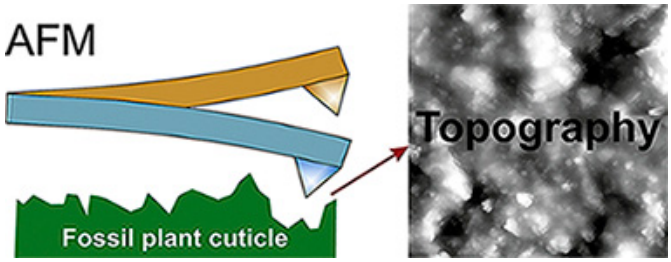


Combining O-17 Magic-Angle Spinning (MAS) NMR at natural abundance with DFT calculations is a promising methodology to shed light on the structure and disorder in tetrahedral sheets of designed micas with enhanced properties. Among brittle micas, synthetic mica is an important alternative to natural ones with a swelling sheet-like structure that results in many applications, by exploiting unique characteristics. Lowenstein's rule is one of the main chemical factor that determines the atomic structure of aluminosilicates and furthermore their properties. In the present article, O-17 MAS NMR spectroscopy is used to validate (or not) the agreement of the Lowenstein's rule with the distribution of Si and Al sites in the tetrahedral sheets of synthetic micas. O-17 MAS spectra of synthetic high-charged micas exhibit two regions of signals that revealed two distinguishable oxygen environments, namely Si-O-X (with X = Si, Al-tet , Mg) and Al-tet -O-Y (Y=Mg or Al-tet). DFT calculations were also conducted to obtain the O-17 chemical shift and other NMR features like the quadrupolar coupling constant, C-Q, for all of the oxygen environments encountered in the two model structures, one respecting the Lowenstein's rule and the other involving Al-tet -O-Al-tet and Si-O-Si environments. Our DFT calculations support the O-17 assignment, by confirming that Al-tet -O-3Mg and Al tet -O-Al tet oxygen environments show chemical shifts under 30 ppm and more important, with quadrupolar coupling constants of about 1 MHz, in line with the spectral observation. By quantifying the O-17 MAS NMR spectra at natural abundance, we demonstrate that one of the synthetic mica compositions does not meet the Lowenstein's rule.

Applications and potentialities of Atomic Force Microscopy in fossil and extant plant cuticle characterization

Benitez, JJ, Guzman-Puyol, S, Dominguez, E, Heredia, A, Heredia-Guerrero, JA
Review of Palaeobotany and Palynology, **268** (2019) 125-132
Septiembre, 2019 | **DOI:** 10.1016/j.revpalbo.2019.06.015

Atomic Force Microscopy (AFM) is a versatile technique of surface characterization, providing accurate information about the topography and other wide variety of magnitudes at submicron scale. It is extensively utilized in materials science, but its use in other disciplines such as paleobotany is infrequent. In this review, we introduce the main concepts of AFM to



paleobotanists, comparing the characteristics of this technique to common electronic and optical microscopies. Then, main works with extant plants, in particular plant cuticles, are described. Finally, realistic applications with fossils are reviewed and their potential use in the characterization of plant fossils discussed. AFM is proposed as a complementary technique to common microscopies to characterize plant cuticle fine details at nanoscale.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

Sustainable polycondensation of multifunctional fatty acids from tomato pomace agro-waste catalysed by tin (II) 2-ethylhexanoate
J.A. Heredia-Guerrero, G. Caputo, S. Guzmán-Puyol, G. Tedeschi, A. Heredia, L. Ceseracciu, J.J. Benítez, A. Athanassiou
Materials Today Sustainability, **3-4** (2019) 100004
Marzo, 2019 | **DOI:** 10.1016/j.mtsust.2018.12.001

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Structural studies of radionuclides immobilized matrix by solid state NMR
Maria D alba, Juan I Corredor, Esperanza Pavon
"Applications of NMR Spectroscopy in the Solid State", (2019) Editorial Consejo Superior de Investigaciones Científicas (CSIC), Eds. L. Sanchez-Muñoz, L. Garrido, F. Muñoz y J. Sanz, págs. 171-204
ISBN: 978-84-00-10514-3

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

International Conference on Nanoscience and materials World
18 – 19 noviembre [Barcelona, España]

Adsorption of organophosphates on porous heterostructure mica and organosilicates
D. Alejandra Fonseca, M. Carolia Pazos, Esperanza Pavón, Maria D. Alba
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Funcionalización de micas de alta carga expansibles para la adsorción de metales pesados**
Autor:	Francisco Javier Osuna Barroso
Directores:	Esperanza Pavón González y M. Dolores Alba Carranza
Calificación:	Sobresaliente "Cum Laude"
Centro:	Universidad de Sevilla
Fecha:	25 de noviembre de 2019

DOCENCIA / TEACHING

Programa Maestría en Química (VII y VII cohorte)
Caracterización Estructural del orde a largo y corto alcance desde los sólidos policristalinos hasta los amorfos, asignatura Química Inorgánica
María Dolores Alba Carranza
Lugar: Universidad Pedagógica y Tecnológica de Colombia (UPTC, Tunja)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Horno tubular hasta 1200º C
- Horno tubular en atmósfera controlada hasta 1700º C
- Horno de Cámara hasta 1650º C
- Horno de Cámara hasta 1200 ºC
- Estufa hasta 400º C
- Estufa hasta 200º 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 de Sólidos
Mechanical Properties of Solids [642016]

Reactividad de Sólidos
Reactivity of Solids [642008]

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Dr. Pedro José Sánchez Soto

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Dra. María Jesús Sayagués de Vega

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Becarios Predoctorales

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Personal Contratado

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PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

Código/Code:	CTQ2017-83602-C2-1-R
Periodo/Period:	01-01-2018 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
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 proyecto 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. En 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 demostrase en la planta piloto que se construye en el marco el 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 1 will coordinate the new project. Moreover, subproject 1 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 1, 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. Finaly, the results of the project will be directly applied to the pilot plant constructed within the H2020 SOCRATCES project.

Almacenamiento TErmoQuímico Híbrido de energía SOLAR concentrada SOLARTEQH Hybrid thermochemical storage of concentrated solar energy SOLARTEQH

Código/Code:	CTQ2014-52763-C2-1-R
Periodo/Period:	01-01-2015 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	82.280 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda
Componentes/Research group:	María Jesús Diánez Millán, José Manuel Criado Luque

RESUMEN / ABSTRACT

Actualmente existen proyectos dentro de los programas Sunshot (USA) y FP7 (UE) en los que se analiza la viabilidad de lechos fluidizados de sólidos granulados para el almacenamiento químico de energía solar concentrada. Uno de los materiales considerados es la caliza natural ($CaCO_3$), abundante y barata. Usando una mezcla CO_2/aire en porcentajes relativos adecuados a las temperaturas de trabajo (600ºC-900ºC) se descarbonataría el $CaCO_3$ mediante reacción endotérmica en períodos de elevada irradiación o se carbonataría el CaO liberando calor cuando la temperatura descendiese por debajo de un cierto valor. Mediante la variación del $\%CO_2$ en el gas de fluidización se provocarían las reacciones de descarbonatación-carbonatación según se desee reducir o aumentar la temperatura del lecho en función de la intensidad de radiación solar y de la demanda. Este control ayudaría a paliar el efecto de la variabilidad de la intensidad de radiación solar sobre la transferencia de calor al ciclo de vapor para la producción de corriente eléctrica. Además de tratarse de un almacenamiento de energía sin pérdidas, la densidad energética del $CaCO_3$ (~1 MWhr/m^3) es mayor que la de las sales fundidas actualmente empleadas en plantas comerciales (0.25-0.40 MWhr/m^3), siendo además la caliza un material no corrosivo, no degradable y que permitiría operar a mayores temperaturas y aumentar así la eficiencia de conversión termoeléctrica. No obstante, la fluidización de la caliza es altamente heterogénea, formándose canales de gas y agregados no fluidizables en el lecho que reducirían en gran medida la transferencia térmica, la eficacia de contacto sólido/gas y por tanto el grado de conversión. Por otra parte, se han puesto en marcha plantas piloto basadas en almacenamiento térmico en lechos fluidizados de sólidos granulados inertes de alta capacidad calorífica como la arena o el carburo de silicio que presentan un estado de fluidización uniforme con alta transferencia térmica. Estos sistemas poseen inevitables pérdidas térmicas y son necesarios grandes volúmenes para garantizar el suministro de calor al ciclo de vapor en períodos de baja radiación. Nuestro proyecto se basa en complementar de manera sinérgica las ventajas del almacenamiento térmico en lechos fluidizados de sólidos inertes con el químico mediante lechos fluidizados de mezclas de sólidos inertes fluidizables con otros basados en CaO (arena y caliza naturales por ejemplo). En nuestro trabajo evaluaremos la transferencia y almacenamiento de energía solar concentrada de estos sistemas híbridos. El plan de trabajo contemplará acotar las condiciones óptimas de concentración de CO_2 en el gas de fluidización y proporción de arena/caliza en función de la temperatura para las que la eficacia de almacenamiento se viera optimizada. Estudiaremos las propiedades físicas y químicas de

mezclas de arena/caliza y los parámetros físicos que favorezcan la transferencia y almacenamiento de calor en función de la intensidad de la radiación solar. Así mismo se explorarán métodos de estabilización térmica del CaO con el objeto de incrementar la reversibilidad de carbonatación/calcinación en condiciones prácticas. De manera paralela se desarrollará un modelo termodinámico que incluya aquellos procesos que afectan a la eficiencia energética del mismo y sirva para establecer parámetros óptimos de operación con el objetivo final de transferencia al sector tecnológico para lo que se contará con el apoyo de Abengoa Solar.

There are current projects within the Sunshot (USA) initiative and UE FP7 program in which the feasibility of fluidized beds for permanent chemical storage of concentrated solar energy is analyzed. One of the materials considered is the cheap and abundantly available natural limestone ($CaCO_3$). Using a CO_2/air mixture in suitable relative proportions according to the operating temperatures (600-900ºC), $CaCO_3$ would be decarbonated by endothermic reaction in periods of high irradiation or the CaO would be carbonated releasing heat when the temperature falls below a certain value. By varying the $\%CO_2$ in the fluidization gas, either decarbonation or carbonation would be provoked as desired to reduce or increase the bed temperature based on the intensity of solar radiation and electricity demand. This control would help to alleviate the effect of the variability of sunlight intensity. Besides of the permanent storage of energy, the energy density of $CaCO_3$ (about 1 MWhr/m^3) is greater than that of molten salts currently used in commercial plants (0.25-0.40 MWhr /m^3). Furthermore, natural limestone is non-corrosive material, not degradable and would allow operation at higher temperatures thus increasing the thermoelectric conversion efficiency. However, the fluidization of limestone is typically very heterogeneous, being characterized by the formation of gas channels and large unfluidizable aggregates in the bed which greatly reduce the effectiveness of solid/gas contact and thus the heat transfer efficiency of the reaction. On the other hand, other projects have leaded to the development of successful small-scale pilot plants based on the thermal storage in fluidized beds of inert solids with high heat capacity such as fine silica sand or silicon carbide with good fluidization properties and thus characterized by a high heat transfer. However, these systems present unavoidable heat losses and large volumes are needed to ensure a supply of heat to the power cycle in periods of low solar irradiation. Our project is based on synergistically combine the heat storage in fluidized beds of fluidizable inert solids (such as sand) with the permanent chemical storage of CaO precursors (such as natural limestone) by the use of fluidized beds of mixtures of both granular materials. Experimental measurements will allow characterizing the behavior of the sand/natural limestone mixtures for the transfer and storage of concentrated solar energy. The working plan shall limit the optimum concentration of CO_2 in the fluidizing gas and proportion of sand/limestone as a function of temperature for optimizing the energy storage efficiency. The physic-chemical properties of mixtures of sand/limestone that favor heat transfer and storage according to the intensity of solar radiation will be delimited. Also thermal and chemical stabilization methods will be explored in order to increase the reversibility of the carbonation/calcination reaction under practical conditions. In parallel, a thermodynamic modeling work will be carried out that includes processes that affect the energy efficiency and serve as a starting point to establish optimum operating parameters with the ultimate goal of transferring the knowledge to the technology sector. For this final purpose the project has the support of Abengoa Solar.

Cerámicas nanoestructuradas a base de carburo de boro y nitruro de titanio para aplicaciones estructurales Boron carbide and titanium nitride-based nanostructured ceramics for structural applications

Código/Code:	MAT2015-71411-R
Periodo/Period:	01-01-2016 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	157.300 €
Investigador responsable/Research head:	Diego Gómez García / Arturo Domínguez Rodríguez
Componentes/Research group:	Francisco L. Cumbreras Hernández, Felipe Gutíerrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

El proyecto tiene como misión la fabricación de forma controlada científicamente de nanocerámicos de carburo de boro y de nitruro de titanio mediante la técnica de chispa de plasma. Se estudiarán las propiedades mecánicas de ambos cerámicos a temperatura ambiente (dureza y tenacidad), así como su plasticidad a alta temperatura (resistencia a la fluencia, deformación a velocidad constante).

Se pretende estudiar la influencia de la microestructura en la respuesta mecánica, así como dilucidar los mecanismos que controlan la plasticidad (particularmente la interacción de dislocaciones con maclas). Los resultados se modelarán analíticamente o mediante simulación a escala mesoscópica (vía modelos de campos de fases).

Boron carbide and titanium nitride are among the most promising ceramic materials nowadays. In the first case, this is due to the outstandng mechanical properties (it is the third hardest material in nature) and its high resistance to chemical attack. In the case of Titanium nitride, its remarkable optical properties and electrical conductivity makes this a potential material for electronic devices. In both cases, sintering is a challenging issue due to the low diffusitivity. In this project, sintering of these materials by spark plasma sintering will be studied and the conditions for nanostructuration will be determined. Preliminary results show that average grain sizes as low as 100 nm can be achieved. In a second stage, plasticity will be studied. A previous model developed by the authors show that twinning is a key ingredient as a driving force of plasticity of boron carbide. The case of titanium nitride is mostly exciting because the stacking fault energy is the lowest ever known and it can make twinnin very favoured. The comparison between these two systems can be a clue about the basic mechanism for hardening in these ceramic materials.

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales Processing and characterization of ceramic composites with two-dimensional laminar nanomaterials

Código/Code:	PGC2018-101377-B-I00
Periodo/Period:	01-01-2019 / 31-12-2022
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
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

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 analizar 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.

OTROS PROYECTOS / OTHER PROJECTS

Sínterización 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

Solar calcium-looping integration for thermo-chemical energy storage

Código/Code:	727348
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	Union Europea (SOCRATES)
Importe total/Total amount:	250.000 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda

Desarrollo y caracterización de nuevos composites geopoliméricos basados en residuos de la industria del olivar. Hacia una construcción sostenible

Código/Code:	MAT2017-88097-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	MINECO, proyecto I+D+I RETOS
Importe total/Total amount:	108.900 €
Investigador responsable/Research head:	D. Eliche-Quesada (UJaén)
Participante del ICMS como investigador:	Pedro José Sánchez Soto

PATENTES / PATENTS

Utilización de polvo de filtro residual de la industria del aluminio para la obtención de espumas rígidas geopoliméricas, material obtenido y usos del mismo

Inventores: Pedro José Sánchez Soto, Luis Pérez Villarejo, Sara Ruiz Molina, Cristina Gallardo López, Dolores Eliche Quesada
Número de Solicitud: 201931008
Fecha Solicitud: 19 de noviembre de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas, Universidad de Jaén

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

The Calcium-Looping ($CaCO_3$/CaO) process for thermochemical energy storage in Concentrating Solar Power plants

Ortiz, C, Valverde, JM, Chacartegui, R, Perez-Maqueda, LA, Gimenez, P
Renewable & Sustainable Energy Reviews, **113** (2019) 109252
Octubre, 2019 | **DOI:** 10.1016/j.rser.2019.109252

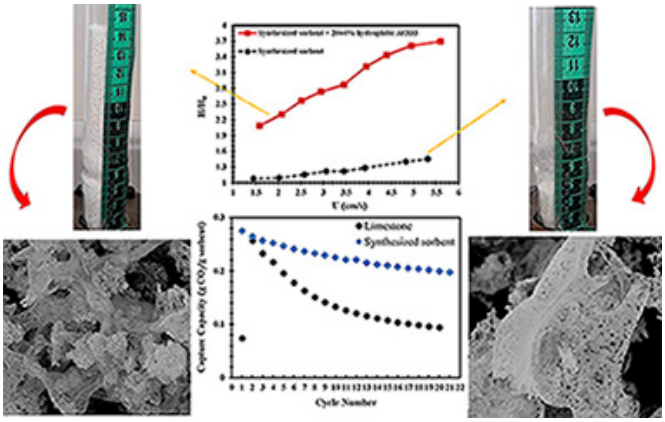
Energy storage based on thermochemical systems is gaining momentum as a potential alternative to molten salts in Concentrating Solar Power (CSP) plants. This work is a detailed review about the promising integration of a $CaCO_3$/CaO based system, the so-called Calcium-Looping (CaL) process, in CSP plants with tower technology. The CaL process relies on low cost, widely available and non-toxic natural materials (such as limestone or dolomite), which are necessary conditions for the commercial expansion of any energy storage technology at large scale. A comprehensive analysis of the advantages and challenges to be faced for the process to reach a commercial scale is carried out. The review includes a deep overview of reaction mechanisms and process integration schemes proposed in the recent literature. Enhancing the multicycle CaO conversion is a major challenge of the CaL process. Many lab-scale analyses carried out show that residual effective CaO conversion is highly dependent on the process conditions and the CaO precursors used, reaching values in a wide range (0.07–0.82). The selection of the optimal operating conditions must be based on materials performance, process integration, technology and economics aspects. Global plant efficiencies over 45% (without considering solar-side losses) show the interest of the technology. Furthermore, the technological maturity and potential of the process is assessed. The direction towards which future works should be headed is discussed.

Multicycle CO_2 capture activity and fluidizability of Al-based synthesized CaO sorbents

Azimi, B, Tahmasebpoor, M, Sanchez-Jimenez, PE, Perejon, A, Valverde, JM
Chemical Engineering Journal, **358** (2019) 679-690 (8,355 – D1)
Febrero, 2019 | **DOI:** 10.1016/j.cej.2018.10.061

CaO-based materials have been identified as promising sorbents for highly efficient pre-combustion and post-combustion CO_2 capture in fluidized beds operated at high temperatures by means of the Calcium Looping (CaL) process. However, Ca-based sorbents suffer from a decline of the capture capacity over multiple sorption/desorption cycles, mainly due to sintering, and from a markedly heterogeneous fluidization behavior due to the strength of interparticle attractive forces as compared to particle weight. The present study is focused on the development of novel synthetic CaO/Al_2O_3 sorbents for CO_2 capture with enhanced CaL performance and fluidizability by dry mixing with flow conditioner nanopowders. The influence of initial precursors on the sorbents multicycle activity at realistic CaL conditions has been investigated. The formation of a stable Ca9Al6O18 mixed-phase during the preparation of the sorbents promotes the multicycle capture capacity. The type of Ca and Al precursors, either soluble or insoluble, can significantly affect the dispersion of this stabilizer (Ca9Al6O18) in the sorbent matrix and, consequently, may affect the carbonation activity of the materials. The

sorbent prepared from soluble aluminum nitrate and calcium nitrate precursors by sol-gel method exhibits a very stable multicycle capture capacity with a capture capacity around 0.2 g

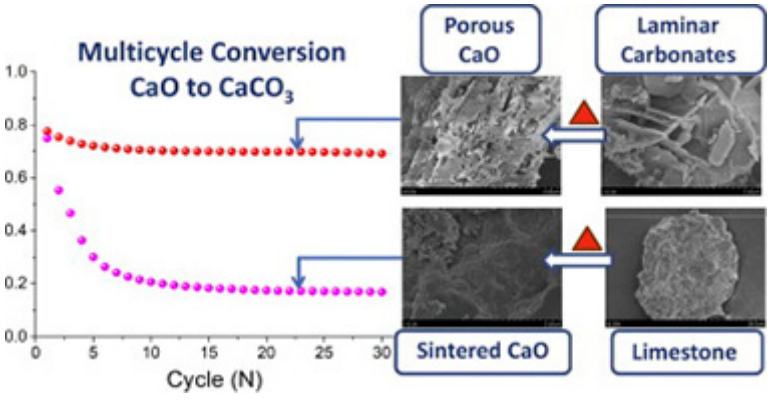


of CO_2/g of sorbent after 21 cycles keeping a 72% of its initial capture capacity. The fluidizability of this promising sorbent was also investigated as affected by the addition of three different flow conditioners. Fluidization experiments confirmed the positive effect of using hydrophilic alumina and hydrophobic silica nanoparticles on improving the fluidizability of the synthesized sorbents.

High-performance and low-cost macroporous calcium oxide based materials for thermochemical energy storage in concentrated solar power plants

Jimenez, PES, Perejon, A, Guerrero, MB, Valverde, JM, Ortiz, C, Maqueda, LAP
Applied Energy, **235** (2019) 543-552 (8,426 – D1)
Febrero, 2019 | **DOI:** 10.1016/j.apenergy.2018.10.131

High energy density, cycling stability, low cost and scalability are the main features required for thermochemical energy storage systems to achieve a feasible integration in Concentrating Solar Power plants (CSP). While no system has been found to fully satisfy all these requirements, the reversible $CaO/CaCO_3$ carbonation reaction (CaL) is one of the most promising since CaO natural



precursors are affordable and earth-abundant. However, CaO particles progressively deactivate due to sintering-induced morphological changes during repeated carbonation and calcinations cycles. In this work, we have prepared acicular calcium and magnesium acetate precursors using

a simple, cost-effective and easily scalable technique that requires just the natural minerals and acetic acid, thereby avoiding expensive reactants and environmentally unfriendly solvents. Upon thermal decomposition, these precursors yield a stable porous structure comprised of well dispersed MgO nanoparticles coating the $CaO/CaCO_3$ grains that is resistant to pore-plugging and sintering while at the same time exhibits high long term effective conversion. Process simulations show that the employment of these materials could significantly improve the overall CSP-CaL efficiency at the industrial level.

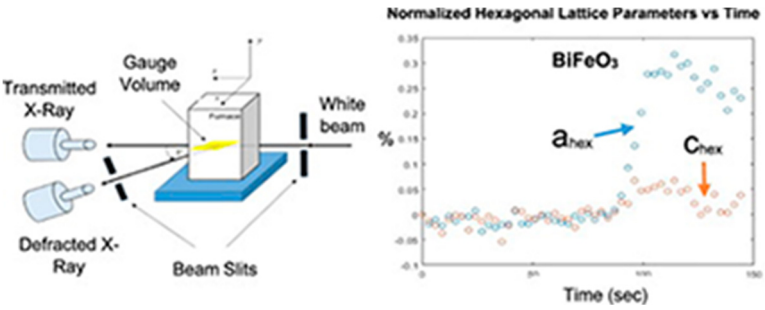
Manufacturing optimisation of an original nanostructured (beta plus gamma)-TiNbTa material

Garcia-Garrido, C, Gutierrez-Gonzalez, C, Torrecillas, R, Perez-Pozo, L, Salvo, C, Chicardi, E
Journal of Materials Research and Technology-JMR&T, **8** (2019) 2573-2585 (3,327 – Q1)
Mayo, 2019 | **DOI:** 10.1016/j.jmrt.2019.03.004

An original (beta + gamma)-TiNbTa material was manufactured by an optimised powder metallurgy treatment, based on a mechanical alloying (MA) synthesis, carried out at low energy, and a subsequently field assisted consolidation technique, the pulsed electric current sintering (PECS). The successful development of this (beta + gamma)-TiNbTa material was possible by the optimisation of the milling time (60 h) for the MA synthesis and the load and sintering temperature for the PECS (30 MPa and 1500 degrees C), as key parameters. Furthermore, the selected heating and cooling rates were 500 degrees C min(-1) and free cooling, respectively, to help maintain the lowest particle size and to avoid the formation of a detrimental high stiffness, hexagonal (alpha)-Ti alloy. All these optimised experimental conditions enabled the production of a full densified (beta + gamma)-TiNbTa material, with partially nanostructured areas and two TiNbTa alloys, with a body centred cubic (beta) and a novel face-centred cubic (gamma) structures. The interesting microstructural characteristics gives the material high hardness and mechanical strength that, together with the known low elastic modulus for the beta-Ti alloys, makes them suitable for their use as potential biomaterials for bone replacement implants.

Anisotropic lattice expansion determined during flash sintering of $BiFeO_3$ by in-situ energy-dispersive X-ray diffraction

Wassel, MAB, Perez-Maqueda, LA, Gil-Gonzalez, E, Charalambous, H, Perejon, A, Jha, SK, Okasinski, J, Tsakalakos, T
Scripta Materialia, **162** (209) 286-291 (4,539 - D1)
Marzo, 2019 | **DOI:** 10.1016/j.scriptamat.2018.11.028

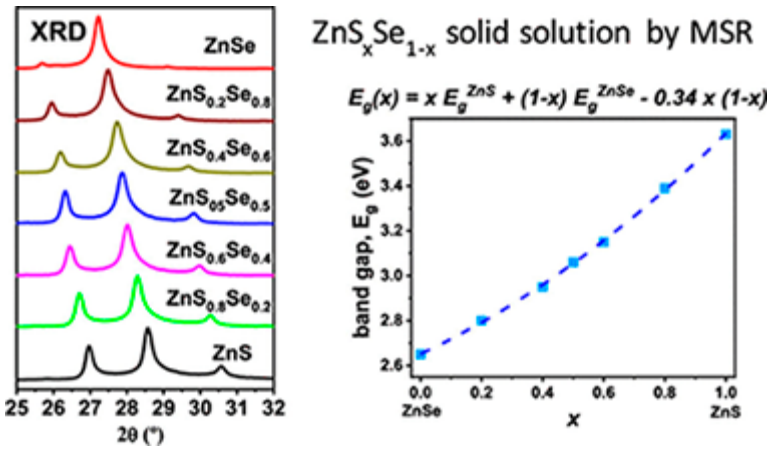


$BiFeO_3$ has a Curie temperature (T-c) of 825 degrees C, making it difficult to sinter using conventional methods while maintaining the purity of the material, as unavoidably secondary phases appear at temperatures above T-c Flash sintering is a relatively new technique that saves time and energy compared to other sintering methods. $BiFeO_3$ was flash sintered at 500 degrees C to achieve 90% densification. In-situ energy dispersive X-ray diffraction (EDXRD) revealed that the material did not undergo any phase transformation, having been sintered well below the Tc. Interestingly, anisotropic lattice expansion in the material was observed when the sample was exposed to the electric field.

Tailoring the Band Gap in the ZnS/ZnSe System: Solid Solutions by a Mechanically Induced Self-Sustaining Reaction

Aviles, MA, Cordoba, JM, Sayagues, MJ, Gotor, FJ
Inorganic Chemistry, **58** (2019) 2565-2575 (4,85 – D1)
Febrero, 2019 | **DOI:** 10.1021/acs.inorgchem.8b03183

The complete ZnSxSe1-x solid solution was successfully obtained by the mechanochemical process denoted as a mechanically induced self-sustaining reaction. Excellent control of the chemical stoichiometry of the solid solution was possible by adjusting the atomic ratio of the starting Zn/S/Se elemental mixture subjected to milling. A mixture of both wurtzite-2H (hexagonal) and zinc blende (cubic) structures was always obtained, although for a similar



milling time the proportion of the zinc blende structure increased with the Se content in the solid solution. However, wurtzite was the major phase for S-rich compositions when milling was stopped just after ignition. It was demonstrated that milling induces the wurtzite-to-zinc blende phase transition. The 8H hexagonal polytype was also observed in samples subjected to long milling times. Variation of the lattice parameters for both structures with the x value in the solid solution presented an excellent linearity, confirming the validity of Vegard's law. However, variation of the band-gap energy (E-g) with x was not perfectly linear, and a small bowing parameter of 0.34 was obtained. It was possible to tune the E-g value between those of the end members of the solid solution in a continuous manner by adjusting the stoichiometry of the solid solution. The morphology and crystalline domain size can also be controlled by adjusting, in this case, the postignition milling time of the mechanochemical process.

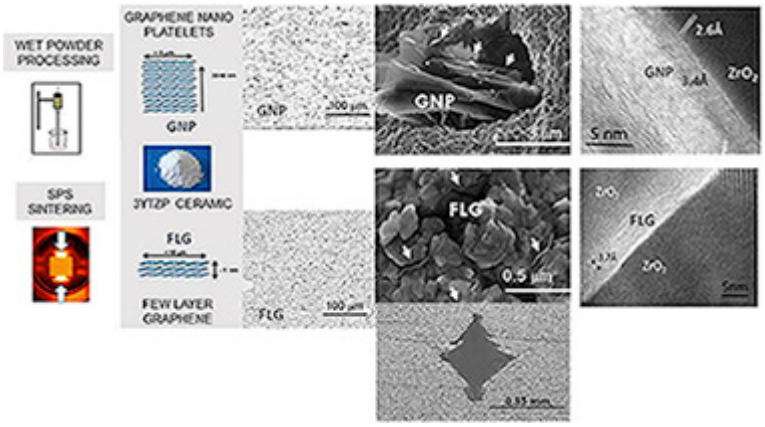
Microstructure, interfaces and properties of 3YTZP ceramic composites with 10 and 20 vol% different graphene-based nanostructures as fillers

Munoz-Ferreiro, C, Morales-Rodriguez, A, Rojas, TC, Jimenez-Pique, E, Lopez-Pernia, C, Poyato, R, Gallardo-Lopez, A

Journal of Alloys and Compounds, **777** (2019) 213-224 (4,175 – D1)

Marzo, 2019 | **DOI:** 10.1016/j.jallcom.2018.10.336

The graphene family comprises not only single layer graphene but also graphene-based nanomaterials (GBN), with remarkably different number of layers, lateral dimension and price. In this work, two of these GBN, namely graphene nanoplatelets (GNP) with n similar to 15-30 layers and few-layer graphene (FLG) with n < 3 layers have been evaluated as fillers in 3 mol% yttria stabilized tetragonal zirconia (3YTZP) ceramic composites. Composites with 10 and 20 vol% GNP or FLG have been fabricated by wet powder processing and spark plasma sintering (SPS)



and the influence of the content and number of layers of the graphene-based filler has been assessed. For both graphene-based fillers, an intermediate zirconia oxycarbide has been detected in the grain boundaries. The lower stacking degree and much more homogeneous distribution of the FLG, revealed by transmission electron microscopy (TEM), can improve load transfer between the GBNs and the ceramic matrix. However, high FLG contents lower densification of the composites, due partly to the larger FLG interplanar spacing also estimated by TEM. The hardness (both Vickers and nanoindentation) and the elastic modulus decrease with increased GBN content and with improved graphene dispersion. The FLG greatly inhibit the crack propagation that occur perpendicular to their preferential orientation plane. The composites with thinner FLG have higher electrical conductivity than those with GNP. The highest electrical conductivity is achieved by composites with 20 vol% FLG in the direction perpendicular to the compression axis during sintering, sigma(perpendicular to) = 3400 +/- 500 Sm-1.

Sintering kinetics, defect chemistry and room-temperature mechanical properties of titanium nitride prepared by spark plasma sintering

Chavez, JMM, Moshtaghioun, BM, Hernandez, FLC, Garcia, DG

Journal of Alloys and Compounds, **807** (2019) 151666 (4,175 – D1)

Octubre, 2019 | **DOI:** 10.1016/j.jallcom.2019.151666

Fully dense titanium nitride polycrystals have been prepared by spark plasma sintering. The kinetics of the sintering process and the optimized conditions for SPS processing have been put forward. Microstructural analyses of the resulting samples have unambiguously shown the coexistence of titanium as Ti2+, Ti3+ and Ti4+, thus driving the presence of cation vacancies. This fact is a new ingredient which is shown to influence the mechanical properties of this strategic ceramic.

Does grain size have an influence on intrinsic mechanical properties and conduction mechanism of near fully-dense boron carbide ceramics?

Moshtaghioun, BM, Laguna-Bercero, MA, Gomez-Garcia, D, Pena, JI
Journal of Alloys and Compounds, **795** (2019) 408-415
Julio, 2019 | **DOI:** 10.1016/j.jallcom.2019.05.03

This work is concentrated on getting a reply to the following question: how does the grain size of boron carbide specimens influence on their mechanical and electrical response? It is a common issue that both essential properties are usually affected by the grain boundaries. To this purpose, a set of near fully-dense boron carbide specimens were prepared by spark plasma sintering. In order to reduce residual porosity and grain-size effects, nanoindentation tests at room temperature were conducted. DC conductivity was measured through four-point test technique from room temperature up to 800 °C. The results show that hardness can reach values as high as ∼60 GPa and plasticity onset takes place at around 23 GPa by dislocation nucleation. Regarding the conductivity, it is found that grain boundaries can block the mobility of bipolarons in an effective way. A simple additive law is provided to account for the resistivity of boron carbide polycrystals.

Microbiological induced carbonate ($CaCO_3$) precipitation using clay phyllites to replace chemical stabilizers (cement or lime)

Morales, L, Garzon, E, Romero, E, Sanchez-Soto, PJ
Applied Clay Science, **174** (2019) 15-28 (3,89 – Q1)
Junio, 2019 | **DOI:** 10.1016/j.clay.2019.03.018

The objective of the present study is to develop a biotechnological tool for a new application of clay phyllites as stabilized materials in linear works replacing chemical stabilizer (e.g. cement or lime) by natural cement, formed by precipitated calcium carbonate generated by microorganisms of the Bacillaceae family (Bacilluspasteurii). Part of the development process conducting a chemical and mineralogical characterization and an examination of physical and hydromechanical properties. The results of this study show that the effect of bacteria on clay phyllites increases the calcium carbonate content, specific surface area and plasticity values. These increased values are caused by the addition of a non-plastic component to clay phyllites resulting in a more aggregated structure through the precipitation of calcium carbonate from the bacteria, ultimately filling the pores of this material. Microbiological treatments on clay phyllites tends to aggregate the original particles, creating aggregates that are partially associated with the formation of calcium carbonate. Said process is influenced by the curing and compaction procedures conducted on samples, which also cause breakage of carbonated structures formed during treatment. As a result of this breaking process of aggregates, some

compaction energy is lost and the treated samples do not reach the maximum dry density of the natural state for the same level of compaction energy applied. Treated samples display a slightly larger friction angle with no cohesion, consistent with filling properties and denser condition. Compressibility is consistently lower than that of the natural state. Comparison of collapse data showsthat the occurrence and amount of collapse are controlled by the as-compacted dry density. It is also determined that higher compaction effort is even more effective than increasing the amount of bacteria introduced to stabilize the sample for the filling of pores (size ranges 3–50 μm) with calcium carbonate. However, the post-ageing compaction destroys the initial binding/cementation effect.

Electrical properties of bismuth ferrites: $Bi_2Fe_4O_9$ and $Bi_{25}FeO_{39}$

Perejon, A, Gil-Gonzalez, E, Sanchez-Jimenez, PE, West, AR, Perez-Maqueda, LA
Journal of the European Ceramic Society, **39** (2019) 330-339 (4,029 – Q1)
Febrero, 2019 | **DOI:** 10.1016/j.jeurceramsoc.2018.09.008

$Bi_2Fe_4O_9$ was prepared by solid-state reaction and the electrical properties measured by impedance spectroscopy. After annealing in O-2 at 900 degrees C, $Bi_2Fe_4O_9$ is an electrically-homogeneous insulator. Its high frequency permittivity is constant (similar to 14.1) over the temperature range 300-400 degrees C and shows no evidence of incipient ferroelectricity at lower temperatures. On annealing in N-2 at 900 degrees C, the pellets gradually decompose.
$Bi_{25}FeO_{39}$ was prepared by both solid-state reaction and mechanosynthesis. It showed a modest amount of mixed conduction of both oxide ions and holes. Impedance analysis showed a complex response that best fitted an equivalent circuit consisting of a parallel combination of long-range conduction and short range dielectric relaxation elements.
The electrical conductivity of both Bi-2 Fe_4O_9 and $Bi_{25}FeO_{39}$ is less than that of $BiFeO_3$ prepared by solid-state reaction, which indicates that any leakage conductivity of $BiFeO_3$ is not due to the possible presence of small amounts of these secondary phases.

Tribological behavior of graphene nanoplatelet reinforced 3YTZP composites

Gutierrez-Mora, F, Morales-Rodriguez, A, Gallardo-Lopez, A, Poyato, R
Journal of the European Ceramic Society, **39** (2019) 1381-1388 (4,029 – D1)
Abril, 2019 | **DOI:** 10.1016/j.jeurceramsoc.2018.11.005

The tribological behavior of graphene nanoplatelet (GNP) reinforced 3 mol% yttria tetragonal zirconia polycrystals (3YTZP) composites with different GNP content (2.5, 5 and 10 vol%) was analyzed and discussed. Their dry sliding behavior was studied using a ball-on-disk geometry with zirconia balls as counterparts, using loads between 2 and 20 N at ambient conditions and compared to the behavior of a monolithic 3YTZP ceramic used as a reference material. The composites showed lower friction coefficients and higher wear resistance than the monolithic 3YTZP. An outstanding performance was achieved at 10 N, where the friction coefficient decreased from 0.6 to 0.3 and the wear rates decreased 3 orders of magnitude in comparison with the monolithic ceramic. A layer adhered to the worn surface was found for all the composites, but it did not acted as a lubricating film. The composites with the lowest GNP content showed an overall improved tribological behavior.

Graphene nanoplatelets for electrically conductive 3YTZP composites densified by pressureless sintering

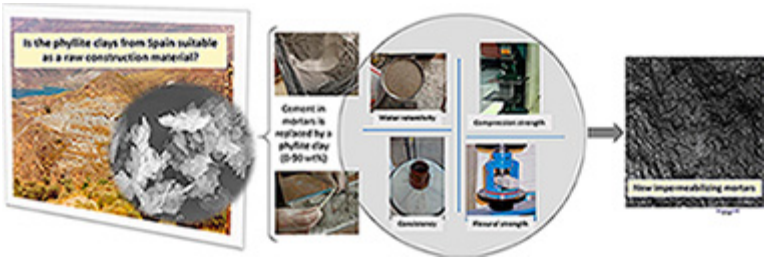
Lopez-Pernia, C, Gallardo-Lopez, A, Morales-Rodriguez, A, Poyato, R
Journal of the European Ceramic Society, **39** (2019) 4435-4439 (4,029 – D1)
Noviembre, 2019 | **DOI:** 10.1016/j.jeurceramsoc.2019.05.067

3 mol% yttria tetragonal zirconia polycrystalline (3YTZP) ceramic composites with 2.5, 5 and 10 vol% graphene nanoplatelets (GNP) were pressureless sintered in argon atmosphere between 1350 and 1450 degrees C. The effects of the GNP content and the sintering temperature on the densification, microstructure and electrical properties of the composites were investigated. An isotropic distribution of GNP surrounding ceramic regions was exhibited regardless the GNP content and sintering temperature used. Electrical conductivity values comparable to the ones of fully dense composites prepared by more complex techniques were obtained, even though full densification was not achieved. While the composite with 5 vol% GNP exhibited electrical anisotropy with a semiconductor-type behaviour, the composite with 10 vol% GNP showed an electrically isotropic metallic-type behaviour.

Phyllite clays as raw materials replacing cement in mortars: Properties of new impermeabilizing mortars

Arce, Carolina, Garzon, Eduardo, Sanchez-Soto, Pedro J.
Construction and Building Materials, **224** (2019) 348-358 (4,046 – D1)
Noviembre, 2019 | **DOI:** 10.1016/j.conbuildmat.2019.07.081

The aim of this investigation was to determine the suitability of phyllite clays as a raw construction material. For that purpose, the cement in mortars was replaced by a phyllite clay (0–90 wt%) making this study the first of its kind to be performed. These materials were prepared with different water proportions according to the water content and water/cement



and water/binder (cement plus phyllite clay) relationships. A comparative study of the most important properties of the resulting experimental mortars was carried out, such as apparent density, water retentivity, consistency and mechanical strength (flexural and compressive strength), along with an evaluation of the pozzolanic activity and permeability. The results showed that the increase of phyllite decreases the apparent density, the consistency and mechanical properties of the mortar, while water retentivity fluctuates. Good correlations ($R2 > 0.84$) were obtained between flexural and compressive strength for the mortars after 28 days of curing. Pozzolanic activity was observed at cement replacement of 80 wt% of phyllite. Moreover, new impermeabilizing mortars constituted by phyllite clay and cement have been obtained according to the low coefficients of permeability. Taking into account the findings of this research, phyllite clays can be applied as raw construction materials with savings derived

from replacing cement in mortars and the low energy consumption involved in their production. However, the present study concluded that the use of phyllite clays did not improve the mechanical strength of these new mortars but, in contrast, they can be applied for impermeabilization purposes in Construction and Civil Engineering.

Elusive super-hard B6C accessible through the laser-floating zone method

Moshtaghioun, BM; Cumbrera, FL; Gomez-Garcia, D; Pena, JI
Scientific Reports, **9** (2019) art. 13340
Septiembre, 2019 | **DOI:** 10.1038/s41598-019-49985-2

Boron carbide is among the most promising ceramic materials nowadays: their mechanical properties are outstanding, and they open potential critical applications in near future. Since sinterability is the most critical drawback to this goal, innovative and competitive sintering procedures are attractive research topics in the science and technology of this carbide. This work reports the pioneer use of the laser-floating zone technique with this carbide. Crystallographic, microstructural and mechanical characterization of the so-prepared samples is carefully analysed. One unexpected output is the fabrication of a B6C composite when critical conditions of growth rate are adopted. Since this is one of the hardest materials in Nature and it is achievable only under extremely high pressures and temperatures in hot-pressing, the use of this technique offers a promising alternative for the fabrication. Hardness and elastic modulus of this material reached to 52 GPa and 600 GPa respectively, which is close to theoretical predictions reported in literature.

Design of highly stabilized nanocomposite inks based on biodegradable polymer-matrix and gold nanoparticles for Inkjet Printing

Begines, Belen; Alcudia, Ana; Aguilera-Velazquez, Raul; Martinez, Guillermo; He, Yinfeng; Wildman, Ricky; Sayagues, Maria-Jesus; Jimenez-Ruiz, Aila; Prado-Gotor, Rafael
Scientific Reports, **9** (2019) 16097
Noviembre, 2019 | **DOI:** 10.1038/s41598-019-52314-2

Nowadays there is a worldwide growing interest in the Inkjet Printing technology owing to its potentially high levels of geometrical complexity, personalization and resolution. There is also social concern about usage, disposal and accumulation of plastic materials. In this work, it is shown that sugar-based biodegradable polyurethane polymers exhibit outstanding properties as polymer-matrix for gold nanoparticles composites. These materials could reach exceptional stabilization levels, and demonstrated potential as novel robust inks for Inkjet based Printing. Furthermore, a physical comparison among different polymers is discussed based on stability and printability experiments to search for the best ink candidate. The University of Seville logo was printed by employing those inks, and the presence of gold was confirmed by ToF-SIMS. This approach has the potential to open new routes and applications for fabrication of enhanced biomedical nanometallic-sensors using stabilized AuNP.

Insight into the $BiFeO_3$ flash sintering process by in-situ energy dispersive X-ray diffraction (ED-XRD)

Perez-Maqueda, LA, Gil-Gonzalez, E, Wassel, MA, Jha, SK, Perejon, A, Charalambous, H, Okasinski, J, Sanchez-Jimenez, PE, Tsakalakos, T
Ceramics International, **45** (2019) 2828-2834 (3,45 – D1)
Febrero, 2019 | **DOI:** 10.1016/j.ceramint.2018.07.293

The sintering mechanism of BiFeO3 has been investigated in-situ by energy dispersive X-ray diffraction (ED-XRD) using a high-energy white collimated X-ray beam from the Advanced Photon Source (Argonne National Laboratories). Such radiation is very penetrating thereby allowing measurements of the sample even when placed inside the flash sintering set up. Additionally, the fast ED-XRD measurements permit monitoring the flash sintering process by providing information about phase composition and sample temperature in real time. Moreover, profile scans, obtained by moving the stage vertically while recording the ED-XRD spectra, permit investigating the homogeneity of the flash for the entire length of the sample. All experiments have been complemented by ex-situ studies. It has been concluded that flash sintering of BiFeO3 is a homogeneous process without any directionality effects. Furthermore, flash sintering takes place at quite low temperatures (below the Tc ≈ 830 °C), which may be related to the high quality of the samples, as pure, highly insulating ceramics without evidence of secondary phases with a homogenous nanostructured grain size distribution are obtained by this technique. Moreover, it is also evidenced that the rapid heating of the sample does not seem to justify, at least by itself, the densification process. Therefore, it appears that the electric current should play a role in the enhanced mobility during the sintering process.

The influence of mechanical activation process on the microstructure and mechanical properties of bulk Ti_2AlN MAX phase obtained by reactive hot pressing

Salvo, C, Chicardi, E, Garcia-Garrido, C, Jimenez, JA, Aguilar, C, Usuba, J, Mangalaraja, RV
Ceramics International, **45** (2019) 17793-17799
Octubre, 2019 | **DOI:** 10.1016/j.ceramint.2019.05.350

The effect of mechanical activation process on the microstructure and mechanical properties of bulk nanostructured Ti2AlN compound has been investigated in this work. The mixture of Ti and AlN powders was prepared in a 2:1 molar ratio, and a part of this powder was subjected to a high-energy milling process under argon atmosphere for 10 h using agate as grinding media. Finally, the densification and formation of the ternary Ti_2AlN MAX phase through solid state reaction of both unmilled and milled powders were carried out by hot pressing under 15 or 30 MPa at 1200 degrees C for 2 h. The microstructure of precursor powder mixtures and the consolidated samples was characterized by using X-ray diffraction (XRD) and a scanning electron microscope equipped with an energy dispersive X-ray spectroscopy (SEM/EDS). The X-ray diffraction patterns were fitted using the Rietveld refinement for phase quantification and to determine their most important microstructural parameters. Microstructure and mechanical properties of the consolidated samples were correlated with the load used for the hot pressing process. The substantial increase of hardness, the higher densification and the lower grain sizes observed in the samples prepared from the activated powders were attributed to the formation of second phases like Ti_5Si_3 and Al_2O_3.

Synthesis of a cubic Ti(BCN) advanced ceramic by a solid-gas mechanochemical reaction

Chicardi, E, Garcia-Garrido, C, Beltran, AM, Sayagues, MJ, Gotor, FJ
Ceramics International, **45** (2019) 3878-3885
Febrero, 2019 | **DOI:** 10.1016/j.ceramint.2018.11.060

In this work, a titanium boron carbonitride advanced ceramic was successfully synthesised by a solid-gas mechanochemical reaction in a planetary ball mill from a mixture of elemental Ti, B, and C under nitrogen atmosphere. This material, with a general formula of Ti(BCN), exhibits a face-centred cubic structure (NaCl type) that is analogous to Ti(CN). This phase was gradually formed with sufficient milling time as a result of diffusional processes, which were permitted by the reduction of the energy in the system caused by the decrease in the spinning rate of the planetary ball mill. In contrast, under more energetic milling conditions, a mechanically induced self-sustaining reaction (MSR) took place, leading to the formation of a TiB2-Ti(CN) ceramic composite. The microstructural characterisation revealed that Ti(BCN) was composed of ceramic particles constituted of misoriented nanocrystalline domains. B, C and N were optimally distributed in the Ti(BCN) phase. The TiB2-Ti (CN) ceramic composite was composed of micrometric and nanometric particles homogeneously distributed. Additionally, the nitrogen content obtained for Ti(BCN) was higher than for the Ti(CN) phase in the composite material.

Low temperature synthesis of an equiatomic (TiZrHfVNb)C5 high entropy carbide by a mechanically-induced carbon diffusion route

Chicardi, E, Garcia-Garrido, C, Gotor, FJ
Ceramics International, **45** (2019) 21858-21863 (3,45 – D1)
Diciembre, 2019 | **DOI:** 10.1016/j.ceramint.2019.07.195

A novel, homogeneous, nanostructured and equiatomic (TiZrHfVNb)C-5 High Entropy Carbide (HEC) was successfully synthesised in a powder form by a mechanosynthesis process from the elemental mixture. This synthesis method for HECs, not previously reported, is simple, reproducible and carried out at room temperature. During milling, the transition metals (Ti, Zr, Hf, V and Nb) alloying and the diffusion of carbon (introduced as graphite) into the alloy structure are simultaneously induced, obtaining the expected (TiZrHfVNb)C-5 HEC. The room temperature method employed contrasts with those reported in the bibliography from binary carbides that are carried out at a very high temperature (1800-2200 degrees C), with the consequent energy savings.

Mechanically induced combustion synthesis and thermoelectric properties of nanostructured strontium hexaboride (SrB6)

Jalaly, M, Khosroshahi, BK, Gotor, FJ, Sayagues, MJ, Yamini, SA, Failamani, F, Mori, T
Ceramics International, **45** (2019) 14426-14431 (3,45 – D1)
Agosto, 2019 | **DOI:** 10.1016/j.ceramint.2019.04.163

The nanoparticles of strontium hexaboride (SrB6) were synthesized by a mechanically induced magnesiothermic combustion in the Mg/B2O3/SrO system. Ignition time in this system was recorded to be 23 min of milling. X-ray diffraction(XRD), X-ray photoelectron spectroscopy (XPS),

and high-resolution transmission electron microscopy (HRTEM) techniques were used to characterize the combustion product. Thermal analysis was employed to assess the formation mechanism. It was revealed that Mg initially reduced B2O3 in a combustive manner to generate elemental boron and a large amount of heat, resulting in the reduction of SrO by Mg at high temperature. The in-situ formed elemental Sr and B react immediately to generate SrB6. Thermoelectric properties of consolidated SrB6, including thermal conductivity, Seebeck coefficient, electrical conductivity, and figure-of-merit were evaluated at the temperature range of 300–873 K.

Sample-Controlled analysis under high pressure for accelerated process studies

Perejon, A, Sanchez-Jimenez, PE, Soria-Hoyo, C, Valverde, JM, Criado, JM, Perez-Maqueda, LA
Journal of the American Ceramic Society, **102** (2019) 1338-1346 (3,094 – D1)
Marzo, 2019 | **DOI:** 110.1111/jace.15960

The potential of controlled rate thermal analysis (CRTA) for studying high-pressure gas-solid processes has been evaluated. CRTA is a type of smart temperature program based on a feedback system that uses any experimental signal related to the process evolution for commanding the temperature evolution. In this work, an instrument that uses the gravimetric signal for CRTA control has been designed and used for the study of two high-pressure gas-solid reactions: the highly exothermic thermal oxidation of TiC under high pressure of oxygen and the reduction in Fe_2O_3 under high pressure of hydrogen. Advantages of CRTA for discriminating overlapping processes and appraising kinetic reaction mechanisms are shown.

Mechanochemical combustion synthesis of vanadium carbide (VC), niobium carbide (NbC) and tantalum carbide (TaC) nanoparticles

Jalaly, M, Gotor, FJ, Sayagues, MJ
International Journal of Refractory Metals & Hard Materials, **79** (2019) 177-184 (2,794 – Q1)
Febrero, 2019 | **DOI:** 10.1016/j.ijrmhm.2018.12.011

The nanoparticles of vanadium, niobium, and tantalum carbides were synthesized by a mechanically induced magnesiothermic combustion in the separate Mg/V2O5/C, Mg/Nb2O5/C, and Mg/Ta2O5/C systems. Initial materials in these systems ignited after short milling times of 10, 10, and 23 min, respectively. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), energy-dispersive X-ray spectroscopy (EDS), high-resolution transmission electron microscopy (HRTEM) and elemental mapping techniques were employed to characterize the combustion products. In this process, magnesium reduces initial oxides to generate elemental V/Nb/Ta to react with carbon, forming the carbide phases.

Technological evolution of ceramic glazes in the renaissance: In situ analysis of tiles in the Alcazar (Seville, Spain)

de Viguerie, Laurence; Robador, Maria D.; Castaing, Jacques; Perez-Rodriguez, Jose L.; Walter, Philippe; Bouquillon, Anne
Journal of the American Ceramic Society, **102** (2019) 1402-1413
Marzo, 2019 | **DOI:** 10.1111/jace.15955

The Alcazar Palace (Seville, Spain) is famous for its ceramic decorations; 16th century wall tiles of different typologies have been analyzed in order to relate the manufacturing process of their colored glazes to the evolving technologies of the Renaissance. Chemical and mineralogical compositions have been determined in situ by nondestructive X-ray fluorescence and X-ray diffraction on arista ceramics in the Cenador de Carlos Quinto, and majolica ceramics in the Palacio Gotico and the Royal oratory. The arista style belongs to the local Hispano-Moresque ceramic tradition. Majolica tiles have the complex microstructures of glazes from Italy. The two types are clearly differentiated by their typology, morphology (curved vs flat surface), and also microstructure (single vs multi-layers), glaze chemistry, and use of different coloring agents. Moreover, we found different glaze chemistries in the investigated majolicas, which correspond to different artists and/or practices.

Production of Ag-ZnO powders by hot mechanochemical processing

Guzman, D, Aguilar, C, Rojas, P, Criado, JM, Dianez, MJ, Espinoza, R, Guzman, A, Martinez, C
Transactions of nonferrous metals society of China, **298** (2019) 365-373
Febrero, 2019 | **DOI:** 10.1016/S1003-6326(19)64946-0

Ag-CdO composites are still one of the most commonly used electrical contact materials in low-voltage applications owing to their excellent electrical and mechanical properties. Nevertheless, considering the restriction on using Cd due to its toxicity, it is necessary to find alternative materials that can replace these composites. In this study, the synthesis of Ag-ZnO alloys from Ag-Zn solid solutions was investigated by hot mechanochemical processing. The hot mechanochemical processing was conducted in a modified attritor mill at 138 degrees C under flowing O-2 at 1200 cm(3)/min for 3.0 h. The microstructure and phase evolution were investigated using X-ray diffractometry, field emission gun scanning electron microscopy and transmission electron microscopy. The results suggest that it is possible to complete the oxidation of Ag-Zn solid solution by hot mechanochemical processing at a low temperature and short time. This novel synthesis route can produce Ag-ZnO composites with a homogeneous distribution of nanoscale ZnO precipitates, which is impossible to achieve using the conventional material processing methods. Considering the fact that the fundamental approach to improving electric contact material performance resides in obtaining uniform dispersion of the second-phase in the Ag matrix, this new processing route could open the possibility for Ag-ZnO composites to replace non-environmentally friendly Ag-CdO.

Degradation processes of historic metal threads used in some Spanish and Portuguese ornamentation pieces

Duran, A; Perez-Maqueda, R; Perez-Rodriguez, JL
Journal of Cultural Heritage, **36** (2019) 135-142
Marzo, 2019 | **DOI:** 10.1016/j.culher.2018.09.006

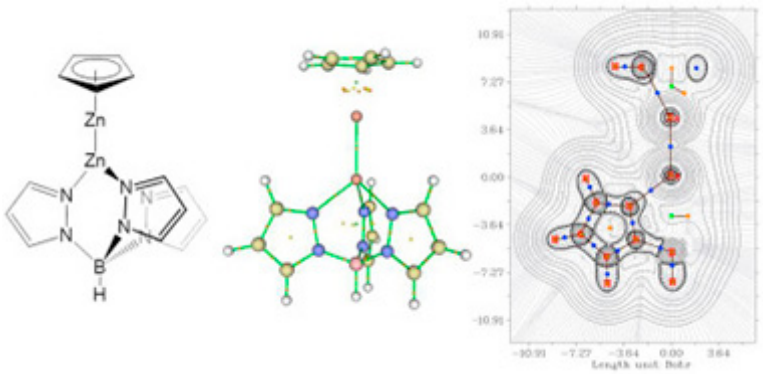
The degradation processes that occurred on metal threads applied in the embroidery used for clothing and in the ornamentation of sculptures, the Sevillian Holy Week processions, and Portuguese and Spanish palace and museum are thoroughly analyzed. Some threads from the 14th and 18–19th centuries were considered. In the metal threads, sulphur- and chlorine-based

compounds were detected either individually or together, depending on the degradation process. Basic silver carbonate, sodium bicarbonate and copper-based compounds were also observed. The different degradation processes were attributed to different factors, such as environmental contamination, degradation of the fibrous cores, and inadequate cleaning and/or mechanical treatments.

A QTAIM and DFT study of the dizinc bond in non-symmetric [CpZn2Ln] complexes

Ayala, R, Galindo, A
Journal of Organometallic Chemistry, **898** (2019) UNSP 120878 (2,066 – Q2)
Octubre, 2019 | **DOI:** 10.1016/j.jorganchem.2019.120878

Several [Zn2L2] and [CpZn2Ln] dizinc compounds have been studied by density functional theory (DFT) and quantum theory of atoms in molecules (QTAIM) in order to compare the nature and



topology of the Zn-Zn bond in symmetrical and non-symmetrical complexes. The stability of these complexes have been evaluated on the basis of the formation energies. The disproportionation reaction has also been analysed indicating that symmetric complexes are less stable than non-symmetric ones. To certain extent, the properties of the [CpZn2Ln] complexes are between those of the [Zn2L2] and [Zn2Cp2] compounds. The asymmetry of the [CpZn2Ln] compounds is illustrated in terms of the topological properties, especially in the Source Function (SF) and Natural Bond Orbital (NBO) analysis.

Laboratory multi-technique study of Spanish decorated leather from the 12th to 14th centuries

Franquelo, ML; Duran, A; Perez-Rodriguez, JL
Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, **218** (2019) 331-341
Julio, 2019 | **DOI:** 10.1016/j.saa.2019.04.012

This work comprises an exhaustive study of Spanish decorative leathers dating from the 12th to 14th centuries. These paintings are considered a key example of a crucible of artistic styles: Gothic, Islamic and Florentine Trecento. The goal of this work was to use the scientific information provided by a number of experimental techniques – namely EDX, micro-FTIR, micro-Raman and micro-XRD – to assess the dating of the wooden vault, leather preparation and filling

fibres. Another goal was to assess the artistic technique based on the characterization of pigments and the differentiation between original materials and those added throughout its history. Gypsum was the original preparation layer extended over the leather. A new preparation stratum was added in further interventions with the artwork. The original pictorial materials and those used during refurbishments have been identified. Original pigments were: red lead, Mars red, red lake, cinnabar, lapis lazuli, red ochres, raw sienna, white lead and charcoal black. Gilding was also found. Pigments added during restoration were: barite, emerald green, rutile, anatase, Mars red, cadmium red, lithopone, cadmium yellow, charcoal black and orpiment.

Effects of Boron Addition on the Microstructure and Mechanical Properties of (Ti,Ta)(C,N)-Co Based Cermets

Chicardi, E, Martinez, FJG
Metals, **9** (2019) 787 (2,259 – Q1)
Julio, 2019 | **DOI:** 10.3390/met9070787

In this work, a titanium-tantalum carbonitride based cermet, with cobalt as the binder phase and boron as a sintering additive, was developed by a mechanically induced self-sustaining reaction process using two different methodologies. The boron additive was added to prevent the formation of brittle intermetallic compounds generally formed during the liquid phase sintering step due to the excessive ceramic dissolution into the molten binder phase. A systematic study was carried out to understand the effects of boron addition on the nature of the phases, microstructure, and mechanical properties of cermets. With the boron addition, the formation of two different boride solid solutions, i. e., (Ti, Ta)B-2 and (Ti, Ta)(3)B-4, was observed. Moreover, the nature of the binder was also modified, from the (Ti, Ta)Co-2 brittle intermetallic compound (for cermets without boron addition) to ductile and tough (Ti, Ta)Co-3 and alpha-Co phases (for cermets with boron addition). These modifications caused, as a general trend, the increase of hardness and toughness in cermets.

La0.59Li0.24TiO3 ceramics obtained by spark plasma sintering: Electric behavior analysis

Pereira, JS, Guerrero, F, Romaguera-Barcelay, Y, Anglada-Rivera, J, Sales, JCC, Silva, RS, Zulueta, Y, Poyato, R, Gallardo, A, Almeida, A, Moreira, JA, Leyet, Y
Materials Research Express, **6** (2019) 015504 (1,449 – Q3)
Enero, 2019 | **DOI:** 10.1088/2053-1591/aae496

This work describes the electric study of Lithium lanthanum titanate (La0.59Li0.24TiO3) ceramics performed by Complex Impedance Spectroscopy. The nanoparticle powders have been synthesized through high energy ball milling and sintered via Spark Plasma Sintering technique. The experimental impedance data have been analyzed using the equivalent circuit model, the Extended Jonscher universal law and the derivative method. From these models, we have determined the dielectric response as well as the grain and grain boundary conductivity. The samples show ionic conductivity values between 10(-5) to 10(-4) S cm(-1) in the studied temperature range, and activation energy values 0.24 eV and 0.48 eV for grain and grain

boundary, respectively. These results confirm the Li+ ions mobility through the crystalline structure of the material.

A theoretical study of the bonding capabilities of the zinc-zinc double bond

Ayala, R, Galindo, A
International Journal of Quantum Chemistry, **119** (2019) e25823 (2,263 – Q1)
Marzo, 2019 | **DOI:** 10.1002/qua.25823

The theoretical knowledge about the zinc-zinc bond has been recently expanded after the proposal of a zinc-zinc double bond in several [Zn-2(L)(4)] compounds (Angew. Chem. Int. Ed.2017, 56, 10151-10155). Prompted by these results, we have selected the [Zn-2(CO)(4)] species, isolobally related to ethylene, and theoretically investigated the possible (2)-Zn-2-coordination to several first-row transition metal fragments. The [Zn-2(CO)(4)] coordination to the metal fragment produces an elongation of the dizinc bond and a concomitant pyramidalization of the [Zn(CO)(2)] unit. These structural parameters are indicative of -backdonation from the metal to the coordinated dizinc moiety, as occurred with ethylene ligand. A quantum theory of atoms in molecules study of the ZnZn bond shows a decrease of (BCP), delta(2)(BCP) (ZnZn) and delocalization indexes (Zn,Zn), relative to corresponding values in the parent [Zn-2(CO)(4)] molecule. The ZnZn and MZn bonds in these [((2)-Zn-2(CO)(4))M(L)(n)] complexes can be described as shared interactions with an important covalent component where the ZnZn bond is preserved, albeit weakened, upon coordination.

Influence of pre-deformation on the precipitation hardening in Cu-Ni-Si alloy

Donoso, E, Dianez, MJ, Criado, JM
Revista de Metalurgia, **55** (2019) e157 (0,54)
Octubre, 2019 | **DOI:** 10.3989/revmetalm.157

The effects of pre-deformation on the precipitation processes in a Cu-2.8 Ni-1.4 Si (at.%) alloy were studied using differential scanning calorimetric (DSC), transmission electron microscopy (TEM) and microhardness measurements. The calorimetric curves shows the presence of one exothermic reaction attributed to the formation of delta-Ni2Si precipitates in the copper matrix that was confirmed by TEM. In addition it can be observed that the temperature of the maximum of the DSC peak decreases with the increase of the pre-deformation to the aging treatments. The activation energies calculated for the precipitation of by the Kissinger method, were similar to those calculated by an Arrhenius function, from the maximum hardening of the matrix due to aging treatments (saturation of the hardness during isothermal aging). The analysis of the microhardness measurements together with the calorimetric curves and the TEM micrographs confirm, on the one hand, that the formation of the delta-Ni2Si phase, during the aging treatments, are responsible for the hardening of the copper matrix, and on the other hand that the deformation prior to the aging treatment partially inhibits the formation of the precipitates.

Effect of heat treatment on apatite coatings deposited on pre-calcified titanium substrates

Beltran, AM, Martin-Santana, Y, Gonzalez, JE, Montealegre-Melendez, I, Gonzalez, E, Peon-Aves, E, Gotor, FJ, Torres, Y
International Journal of Materials Research, **110** (2019) 351-358 (0,851 – Q3)
Abril, 2019 | **DOI:** 10.3139/146.111746

Titanium and its alloys are considered interesting materials for endosseous implants. However, they still present drawbacks related to their in-vivo behavior that can be overcome by coatings, such as apatite. This work focuses on the deposition of apatite coatings on commercially pure titanium (grade II) substrates previously pre-calcified. The influence of the temperature used in the thermal treatment on the microstructure and tribo-mechanical surface properties was analyzed. The coatings were structurally and chemically characterized and their tribo-mechanical behavior was evaluated. The nano-apatite coatings were only formed on surfaces with successive treatments in NaOH and $CaCl_2$ solutions. In addition, scratch tests showed that after the heat treatment the nanoapatite coatings had high bond strength to the substrate.

Effects of the speed ratio on the efficiency of planetary mills

Real, C, Gotor FJ
Heliyon, **5** (2019) e01227 (0)
Febrero, 2019 | **DOI:** 10.1016/j.heliyon.2019.e01227

The ignition time (tig) of the mechanically induced self-sustaining reaction (MSR) process involving the formation of TiB2 from Ti/2B elemental mixtures was used to study the influence of the ratio (k = -ωv/ωd) between the rotational speed of the supporting disc (ωd) and vials (ωv) on the milling efficiency of a Pulverisette 4 planetary mill. The variation of the inverse of the ignition time (1/tig), which is directly related to the milling power provided by the planetary mill, with the process conditions has shown that it is not possible to find a single k value as optimal independently of the experimental conditions used (ωd and the ball-to-powder ratio, BPR). Moreover, it was observed that the lowest milling efficiency (longer tig values) was found for k = 1, which is the usual value employed in routine laboratory works. The best efficiencies were found for the larger k values (2.5 or 3). At lower ωd, the shortest tig was obtained for k = 2.5 and at higher ωd for k = 3, independently of BPR.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

"#CienciaenelParlamento". Una nueva iniciativa de gran interés para acercar la Ciencia y la Política

P.J. Sánchez-Soto
Revista QUÍMICOS DEL SUR, **111** (2019) 34-35

La Real Maestranza y la Academia Sevillana de Ciencias premian a los investigadores jóvenes en su convocatoria de 2018
P.J. Sánchez-Soto
Revista QUÍMICOS DEL SUR, **111** (2019) 42-43

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Ceramic Materials. Synthesis, Characterization, Applications and Recycling
D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto (Editores)
IntechOpen, London (UK), 94 páginas
ISBN (Print): 978-1-78985-773-3; ISBN (Online): 978-1-78985-774-0

Introductory Chapter: Ceramic Materials-Synthesis, Characterization, Applications and Recycling
D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto
Ceramic Materials. Synthesis, Characterization, Applications and Recycling
D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto (Editores)
IntechOpen, London (UK), págs. 1-5

Relationship between the scratch and wear behavior of 3Y-TZP reinforced with graphene nanoplatelets
F. Gutiérrez-Mora, A. Gallardo-López, C. Muñoz-Ferreiro, A. Muñoz, J.C. Sánchez-López, R. Poyato
"IBERTRIVA 2019 Conference Proceedings", (2019) Editorial IBERTRIVA2019, J.A. Martín Gago y J.C. Sánchez López, págs. 118-119
ISBN: 978-84-09-12421-3

Nanostructured bidimensional composites from vermiculite as precursors in the synthesis of advanced ceramic materials
M.A. Avilés, P.J. Sánchez-Soto, S. Martínez-Martínez, E. Bonet-Martínez, D. Eliche-Quesada, L. Pérez-Villarejo
"Nanostructured Materials: Synthesis, Properties and Applications", Editorial Nova Science Publishers, New York. Junhui He (Editor), págs. 253-272
ISBN: 978-1-53615-013-1

Educación artística para la formación de futuros profesionales: actividades y colaboraciones de la Escuela "Della Robbia" de Artes y Artesanías de Andalucía de Gelves (Sevilla)
J.J. Lupión Alvarez, G. Durán-Domínguez, P.J. Sánchez-Soto
Arte, Educación y Patrimonio del Siglo XXI
Z. Calzado, G. Durán, R. Espada (Editores)
Facultad de Educación, Universidad de Extremadura, Fundación CB; IMDEX Impresores, Badajoz (España), págs. 900-904
ISBN: 978-84-09-16330-4

Educación en Arte y Patrimonio Cultural. La preparación y examen científico de microestratigrafías como herramienta formativa en educación Artística y formación de especialistas del Patrimonio Cultural
A.Kriznar, G. Durán-Domínguez, P.J. Sánchez-Soto
Arte, Educación y Patrimonio del Siglo XXI
Z. Calzado, G. Durán, R. Espada (Editores)
Facultad de Educación, Universidad de Extremadura, Fundación CB; IMDEX Impresores, Badajoz (España), págs. 905-911
ISBN: 978-84-09-16330-4

Alkali-activated geopolymers derived from industrial and agro-industrial by-products
L. Pérez-Villarejo, E. Bonet-Martínez, D. Eliche-Quesada, D. Vera-Candeas, P.J. Sánchez-Soto
Vitrification and Geopolymerization of wastes for immobilization or recycling M.M. Jordán, O. Pinet, J.Ma. Rincón (Editores), International Comission of Glass ICG (TC05) y Universidad Miguel Hernández (UMH), Editorial de la UMH, Elche (Alicante, España), págs. 259-273
ISBN: 978-84-16024-78-0

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

CEEC-TAC5 & MEDICTA 2019 5th Central and Eastern European Conference on Thermal Analysis and Calorimetry & 14th Mediterranean Conference on Calorimetry and Thermal Analysis
27 – 30 agosto [Roma, Italia]
Pedro José Sánchez Soto (Miembro Comité Científico)
Antonio Perejón (Miembro Comité Científico)
Pedro José Sánchez Soto (Miembro Comité Científico)
Pedro Enrique Sánchez-Jiménez (Miembro Comité Científico)
Luis Allan Pérez Maqueda (Chair)

COMUNICACIONES / COMMUNICATIONS

International Workshop on Woman in Ceramic Science
7 – 9 abril [Budapest, Hungria]

GBN/zirconia composites: a study of the effect of powder processing method and sintering technique on the microstructural, mechanical and electrical properties

C. López-Pernía, C. Muñoz-Ferreiro, C. González-Orellana, A. Morales-Rodríguez, A. Gallardo-López, R. Poyato
Poster

3YTZP ceramic composites with different 2D nanomaterials
C. Muñoz-Ferreiro, A. Morales-Rodríguez, A. Gallardo-López, R. Poyato
Poster

ICG2019 CONSTRUCTION International Conference on Green Construction
8 – 9 abril [Córdoba, España]

Ecocement clinker design valorizing wastes from different industries
S. Martínez-Martínez, L. Pérez-Villarejo, P.J. Sánchez-Soto, D. Eliche-Quesada
Poster

16th Conference and Exhibition of the European Ceramic Society |16-ECERS
16 – 20 junio [Turin, Italia]

Hardness and fracture toughness of alumina and zirconia composites with different amounts of reduced graphene-oxide sintered by Spark Plasma Sintering
R. Cano-Crespo, P. Rivero-Antúnez, R. Moreno, D. Gómez-García, A.Domínguez-Rodríguez
Comunicación oral

Microstructure and toughening mechanisms in zirconia composites with different graphene-based nanostructures and various powder processing routines
R. Poyato, J. Castillo-Seoane, A. Morales-Rodríguez, Á. Gallardo-López
Comunicación oral

X Iberian Conference on Tribology / XI Iberian Vacuum Conference | IBERTRIVA 2019
26 – 28 junio [Sevilla, España]

Tribo-Mechanical Behaviour of Layered Materials for High Speed Machining Applications
Luis M. González, Francisco J. Gotor, Raúl Bermejo, Ernesto Chicardi, Luis M. Llanes, Elena Fernández, Eduardo Rodríguez, Daniel Ordóñez, Yadir Torres
Comunicación oral

EUROCLAY 2019 CONFERENCE International Conference on Clay Science and Technology
1 – 5 julio [Paris, Francia]

Design of eco-cement clinker by valorizing wastes from the ceramic, the marble and the aluminum industries

S. Martínez-Martínez, D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto, A. Christogerou, D. Kanellopoulou, G.N. Angelopoulos
Poster

CEEC-TAC5 & MEDICTA 2019 5th Central and Eastern European Conference on Thermal Analysis and Calorimetry & 14th Mediterranean Conference on Calorimetry and Thermal Analysis
27 – 30 agosto [Roma, Italia]

Thermal behaviour of the different parts of almonds shells as waste biomass
E. Garzón, C. Arce, A.J. Callejón-Ferré, P.J. Sánchez-Soto
Poster

Characterization, thermal and ceramic properties of phyllite clays from SE Spain
E. Garzón, L. Pérez-Villarejo, P.J. Sánchez-Soto
Poster

30th International Conference on Diamond and Carbon Materials
8 – 12 septiembre [Sevilla, España]

Synthesizing graphene based nanomaterials for zirconia matrix composites
C. Muñoz-Ferreiro, R. Verdugo-Manzanares, A. Gallardo-López, A. Morales-Rodríguez, R. Poyato
Poster

Electrical conductivity of in-situ reduced graphene oxide-zirconia composites
C. López-Pernía, P. Luna, C. Muñoz-Ferreiro, A. Morales-Rodríguez, Á. Gallardo-López, R. Poyato
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

I Jornada de jóvenes investigadores de cerámica y vidrio en Sevilla
28 marzo [Sevilla, España]

Diseño de Clinker de eco-cemento a partir de residuos de las industrias cerámica, del mármol y del aluminio secundario
S. Martínez-Martínez, D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Sistemas de almacenamiento termoquímico para energía solar concentrada**
Autor: Beatriz Sarrión Aceytuno
Directores: Luis Allan Pérez Maqueda y José Manuel Valverde Millán (US)
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla
Fecha: 9 de noviembre de 2019

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Estudio del efecto del tratamiento mecánico de la biomasa en la naturaleza de los residuos obtenidos de su pirólisis**
Autor: Pablo Cerero Sánchez
Directores: María Dolores Alcalá González y Concepción Real Pérez
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (17 septiembre 2019)

Título: **Actividad catalítica de carbones procedentes de biomasa**
Autor: Magdalena Ferrero Guerrero
Directores: María Dolores Alcalá González y Concepción Real Pérez
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (18 septiembre 2019)

Título: **Obtención de SiC por reaccion magnesiotérmica a partir de cascarilla de arroz**
Autor: Alejandro Martínez Jiménez
Directores: María Dolores Alcalá González y Concepción Real Pérez
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (20 septiembre 2019)

Título: **Obtención de nanoláminas de grafeno a partir de grafito para su uso en compuestos cerámicos**
Autor: Reyes Verdugo Manzanares
Directores: Rosalía Poyato Galán
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (25 septiembre 2019)

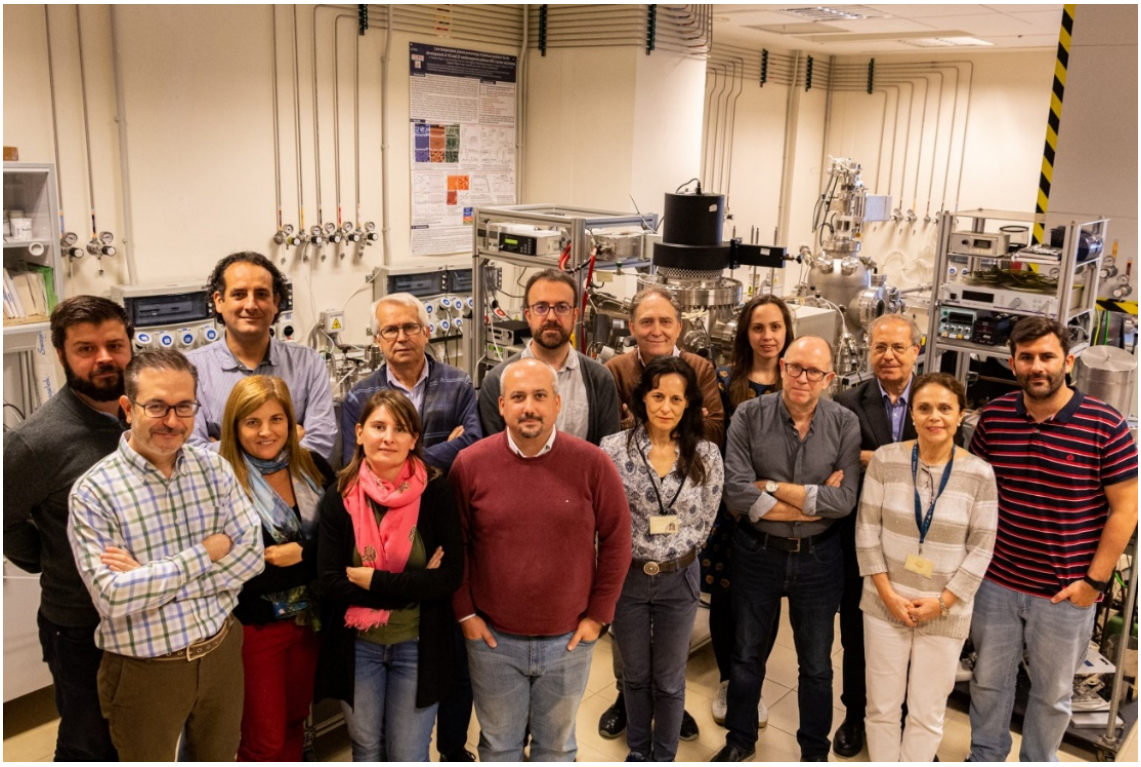
ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

Master en Biotecnología Sanitaria
Nanotecnología
María Jesús Sayagués de Vega
Lugar: Universidad Pablo de Olavide

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 1800ºC AGNI
- Horno horizontal alta temperatura 1600ºC Carbolite
- Horno horizontal 1200ºC 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 1150ºC 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

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Dra. Asunción Fernández Camacho
Dr. Agustín Rodríguez González-Elipe

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Dra. María Aránzazu Díaz Cuenca
Dr. Alberto Palmero Acebedo
Dra. T. Cristina Rojas Ruiz

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Dr. Rafael Álvarez Molina
Dra. Gisela M. Arzac Di Tomaso (hasta agosto)
Dra. Vanda C. Fortio Godinho
Dr. Jorge Gil Rostra
Dra. Ana María Gómez Ramírez
Dra. M. Carmen López Santos
Dr. Juan Ramón Sánchez Valencia

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Ldo. José Manuel Obrero Pérez

Personal Contratado

Ldo. Dirk Hufschmidt
Ing. Tec. M. Rocío García Gil (hasta agosto)

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Superficies super-hielofóbicas para prevenir la formación de hielo en aeroplanos Super-IcePhobic surfaces to prevent ice formation on aircraft

Código/Code:	H2020-TRANSPORT/0149
Periodo/Period:	01-02-2016 / 31-01-2019
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	86.579,04 €
Investigador responsable/Research head:	Agustín R. González-Elipe

RESUMEN / ABSTRACT

La acumulación de hielo representa un grave problema para los aviones, ya que la presencia incluso de una capa apenas visible puede limitar seriamente la funcionalidad de las alas, las hélices, los parabrisas, las antenas, las rejillas de ventilación, las tomas de aire y las cubiertas. El Proyecto PHOBIC2ICE tiene como objetivo desarrollar tecnologías y herramientas de simulación predictiva para evitar o mitigar este fenómeno.

El proyecto PHOBIC2ICE, mediante la aplicación de un enfoque innovador de simulación y modelado, permitirá el diseño y fabricación de superficies hielofóbicas con funcionalidades mejoradas. Se desarrollarán varios tipos de recubrimientos poliméricos, metálicos e híbridos usando diferentes métodos de deposición. Se prepararán superficies tratadas con láser y anodizadas. En consecuencia, el proyecto se centra en la recopilación de conocimientos fundamentales sobre los fenómenos asociados con los problemas de repulsión de hielo. Este conocimiento dará una mejor comprensión del proceso de acreción de hielo en diferentes superficies modificadas y recubiertas. La infraestructura de investigación certificada (túnel de viento de hielo) y las pruebas de vuelo previstas ayudarán a desarrollar soluciones integrales para abordar la cuestión de la formación de hielo y elevarán el nivel de innovación del Proyecto.

La solución propuesta será respetuosa con el medio ambiente, contribuirá a la reducción del consumo de energía y ayudará a eliminar la necesidad de procedimientos frecuentes de deshielo sobre suelo. Esto contribuirá a la reducción del coste, la contaminación y el retraso de vuelo.

The accretion of ice represents a severe problem for aircraft, as the presence of even a scarcely visible layer can severely limit the function of wings, propellers, windshields, antennas, vents, intakes and cowlings. The PHOBIC2ICE Project aims at developing technologies and predictive simulation tools for avoiding or mitigating this phenomenon.

The PHOBIC2ICE project, by applying an innovative approach to simulation and modelling, will enable the design and fabrication of icephobic surfaces with improved functionalities. Several types of polymeric, metallic and hybrid coatings using different deposition methods will be developed. Laser treated and anodized surfaces will be prepared. Consequently, the Project focuses on collecting fundamental knowledge of phenomena associated with icephobicity issues. This knowledge will give better understanding of the ice accretion process on different coatings and modified surfaces. Certified research infrastructure

(ice wind tunnel) and flight tests planned will aid in developing comprehensive solutions to address ice formation issue and will raise the Project's innovation level.

The proposed solution will be environment-friendly, will contribute to the reduction of energy consumption, and will help eliminate the need for frequent on-ground de-icing procedures. This in turn will contribute to the reduction of cost, pollution and flight delay.

Arquitecturas de multicapas nanostructuradas para el desarrollo de dispositivos optofluídicos sensores y procesos de funcionalización superficial avanzada (NANOFLOW)

Nanostructured multilayered architectures for the development of optofluidic responsive devices, smart labers, and advanced Surface functionalization (NANOFLOW)

Código/Code:	MAT2016-79866-R
Periodo/Period:	31-12-2016 / 31-12- 2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	332.750 €
Investigador responsable/Research head:	Angel Barranco Quero / Francisco Yubero Valencia
Componentes/Research group:	Ana Isabel Borrás Martos, Juan Pedro Espinós Manzorro, Fabián Frutos Rayego (US), Germán de la Fuente Leis (ICTP), Fernando Lahoz Zamarro (IPNA), Ricardo Molina Mansilla (IQAC), Alberto Palmero Acebedo, Victor Joaquin Rico Gavira, Agustín R. González-Elipe

RESUMEN / ABSTRACT

NANOFlow es un projecto multidisciplinar que persigue el desarrollo de nuevos dispositivos optofluídicos mediante la integración de materiales nanostructurados multifuncionales. El proyecto está sólidamente fundamentado en la experiencia de los componentes del grupo de investigación en campos como la síntesis de películas multifuncionales, procesos avanzados de modificación de superficies y en el desarrollo de dispositivos fotónicos multicapa. El objetivo principal de este proyecto de investigación es combinar e integrar los distintos métodos avanzados de síntesis y procesado disponibles en la fabricación de dispositivos optofluídicos singulares que sean capaces de responder a la presencia de líquidos mediante un cambio físico. La combinación de este tipo de procesos de integración junto con el desarrollo de nuevos métodos de de detección fotónica, el uso de microplasmas de gran área como fuentes de luz y de sustratos flexibles que incorporan elementos sensores definen un conjunto muy rico de posibilidades de desarrollo de aplicaciones prácticas que se explorarán en el proyecto. Además, en el proyecto también se llevarán a cabo estudios de simulación de crecimiento de películas delgadas que, en combinación con estudios

de diagnosis, permitirán ajustar los procesos de crecimiento para conseguir la funcionalidades requeridas. De esta forma, el proyecto NANOFlow intenta cubrir toda la cadena tecnológica que va desde el desarrollo de nuevos materiales hasta la aplicación final incluyendo estudios de caracterización, rutas sintéticas flexibles, búsqueda de procesos alternativos de bajo costo y alto rendimiento (por ejemplo métodos de fabricación empleando plasmas a presión atmosférica), integración de dispostivos y test de éstos en condiciones reales.

Las actividades de desarrollo planteadas en NANOFlow culminan en el desarrollo de tres tipos de dispositivos innovadores como son: las etiquetas inteligentes con actividad sensora y posibilidad de usarse como sistemas de trazabilidad y anticopia (por ejemplo integrados en el empaquetado de productos alimentarios), un dispositivos optofluídico multisensor versátil y un sistema de limpieza optofluídico fotocatalítico que integra una microplasma emisor de luz, interruptores ópticos de luz UV y visible actuados por líquidos y una superficie fotocatalítica nanoestructurada. Los tres dispositivos funcionan sobre la base de una actuación o respuesta optofluídica y están diseñados para poder usarse de forma directa en sistemas de detección, manipulación y monitorización de líquidos.

Las actividades de investigación en los distintos paquetes de trabajo del proyecto y los dispositivos finales se han propuesto para responder al Reto nº 2 de la convocatoria referida a "Seguridad y calidad alimentaria". Además, algunas de las actividades del proyecto, por ejemplo el tercer dispositivo, están también relacionados con el Reto nº 3 "Energía Segura, eficiente y limpia". Es interesante indicar que las actividades propuestas en NANOFlow son de particular relevancia en el contexto geográfico de Andalucía donde la agricultura, la producción de alimentos y la energía son tres de los más relevantes sectores estratégicos.

NANOFlow is a multidisciplinary Project that aims the development of novel optofluidics sensing devices integrating advanced multifunctional nanostructured materials. The project is solidly grounded in the research group experience in the synthesis of nanoestructured functional thin films, advance surface treatments and development of planar photonic structures The main objective of the project is to combine and integrate the available synthetic and processing methodologies in the fabrication of optofluidic components capable of modifying their physical behavior when they are exposed to liquids. The integration of these optofluidic components together with accessory technologies based on new principles of photonic detection, large surface area microplasmas discharge as light sources or flexible substrates for the fabrication of sensing tags define an ambitious landscape of applications that will be explored in the project. Besides, the modeling of thin film growth in combination with advanced deposition diagnosis methodologies will be combined to adjust the thin film deposition processes to the desired functionalities.Therefore, NANOFlow aims to cover all the scientific-technological chain from the materials development to the final applications including advanced characterization, flexible synthetic routes, alternative low-cost and high throughput process (e.g. atmospheric plasma synthesis), device integration and testing of devices in real conditions.

The NANOFlow research activities will culminate in the development of three innovative devices, namely smart labels for sensing, traceability and anticounterfeiting applications (e.g. smart labels incorporated in food-packaging), a versatile optofluidic multisensing device and an optofluidic photocatalytic cleaning system that will integrate a large area microplasma source, liquid actuated UV/Visible optical switches and a photocatalytic nanostructured surface. All of these devices will operate under the basis of an optofluidic actuation and/or response and are

designed to present clear potentialities for direct application in liquid sensing, manipulation and monitoring.

The NANOFlow research activities in the different work-packages and, particularly, the final devices are intended to have a direct impact in the Theme 2 (Seguridad and Calidad Alimentaria) of the "RETOS" defined in the call covering this project proposal.. Besides, some of the activities proposed, in particular the third device are also connected with the Theme 3 (Energía segura eficiente y limpia) of the call. It is very interesting to stress that these activities are of particular relevance in the geographical context of Andalucia where Agriculture, Food production and Energy are three of the most relevant strategic sectors.

Recubrimientos para aplicaciones en energía y alta temperatura High temperatura energy application coatings

Código/Code:	MAT2015-65539-P
Periodo/Period:	01-01-2016 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	83.006 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Teresa Cristina Rojas Ruiz, Maria Belinda Sigüenza Carballo, Iñigo Braceras Izaguirre, Marta Brizuela Parra

RESUMEN / ABSTRACT

La protección de las superficies frente a la temperatura, los fenómenos de oxidación o el desgaste ha logrado un progreso substancial mediante el desarrollo de nuevos materiales y recubrimientos con propiedades mejoradas tales como dureza extrema, baja fricción y tasas de desgaste, elevada resistencia ante la temperatura y la oxidación. Estas mejoras suponen un enorme ahorro de energía y reducción de costes debido a la vida media de los componentes mecánicos sin necesidad de sustitución, así como, a una reducción del impacto medioambiental. Este campo de investigación tiene una profunda repercusión en una gran variedad de sectores industriales (energía, herramientas de mecanizado, automoción, aeronáutico, metalurgia, etc.). El reto para la mayoría de estos procesos de funcionalización superficial residen en un control estricto de la micro y nanoestructura de la superficie y de las intercaras que hagan posible la aparición de nuevas propiedades y aplicaciones que la nanotecnología ofrece.

En este proyecto, se prepararán recubrimientos nanoestructurados para la protección de componentes sometidos a altas temperaturas y ambientes agresivos buscando un comportamiento mejorado. Este objetivo será abordado para tres diferentes aplicaciones que contribuirían a procesos energéticos más eficientes, energías renovables y soluciones para disminuir el impacto medioambiental. Basándonos en el sistema Cr-Al-N, se depositarán diferentes recubrimientos mediante la técnica de pulverización catódica reactiva cambiando la

composición química (contenido en metal, incorporación de dopantes tales como Y o Si); microestructura; distribución de fases; arquitectura (multicapa/nanocomposite) o estructuras más complejas (tándem, multicapa en gradiente) sobre los sustratos apropiados dependiendo de la aplicación prevista: a) resistencia a la oxidación a alta temperatura (hasta 1000ºC) para herramientas; b) absorbedores solares selectivos estables térmicamente a medias (300-500ºC) y alta temperatura (>600ºC); resistencia a la corrosión para componentes en turbinas de vapor supercríticos (650ºC/100% vapor).

La investigación sobre los mecanismos de oxidación, transformaciones de fases, modificaciones estructurales, etc. serán objeto de un estudio detallado sobre los sustratos definidos para lograr un conocimiento fundamental sobre los procesos de degradación y los efectos protectores. El establecimiento de correlaciones entre las propiedades iniciales y el comportamiento funcional permitirá una mejor comprensión de los mecanismos de protección y por ende, una optimización de tales sistemas en forma de recubrimientos nanoestructurados para las aplicaciones previstas.

The protection of surfaces from thermal, wear and oxidation phenomena has reached a substantial progress by developing new materials and coatings with improved properties as extreme hardness, low friction and wear rates, increased thermal and oxidation resistance. These improvements suppose a huge energy-saving and cost reduction due to the increased life-time of mechanical components without needs of replacement as well as a reduction in the environmental impact. This field of research has a deep impact in a large variety of industrial sectors (energy, machining tools, automotive, aeronautic, metallurgy, etc.). The challenge for most of these surface functionalization procedures is to get a strict control of the micro and nanostructure of the surface and interfaces that make possible the advent of new properties and applications that nanotechnology concept offers.

In this project, tailored nanostructured coatings for protection of components submitted to high temperature and aggressive environments are prepared seeking for an improved performance. This goal will be explored for three different applications that would contribute to an energy efficiency, renewable energies and solutions to decrease environmental impact. Based on the Cr-Al-N system, different coatings will be prepared by reactive magnetron sputtering technology changing chemical composition (metal content, incorporation of dopants like Y or Si); microstructure; phase distribution; architecture (multilayer/ nanocomposite) or more complex structures (tandem, multilayer gradient) on appropriated substrates depending on the foreseen application: a) oxidation resistance at high temperature (up to 1000ºC) for tool components; b) thermal stable solar selective absorber coating for mid (300-500ºC) and high temperature (>600ºC); c) corrosion resistant coating for supercritical turbine components (650ºC and 100% steam atmosphere).

The investigation of the oxidation mechanisms, phase transformations, structural modifications, etc. will be object of a careful study directly over the defined substrates for these applications to get fundamental knowledge on the degradation phenomena and protective effects. The establishment of the relationships between the initial properties and observed functional performance will enable the better understanding of the protection mechanisms and the optimization of such nanostructured coating systems for the selected application.

Desarrollo de catalizadores y soportes para procesos de almacenamiento químico de energía neutros en CO2 basados en líquidos orgánicos portadores de hidrógeno Development of catalysts and supports for CO2 neutral chemical energy storage processes based on liquid organic hydrogen carriers

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:	Mª 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 H2). 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 C02 y del H2 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 H2 (y la energía) proveniente de fuentes renovables. Estos combustibles alternativos se pueden quemar o usarse para generar H2 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 (Le. 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-estructuradesempeño 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 Ti02-TiOx 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 eHect 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 tuels. This makes the storage of energy produced by renewable sources (which are ¡ntermittent) 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 (H2 cycle). In this new project the research group propose to move into the implementatlon of the liquid organic hydrogen carriers (LOHC) as a promising way of comblning the C02 and de H2 cycles leading to a sustainable energy storage in a carbon neutral cycle.

Small organic molecules, IIke formic acid or methanol, can be used to store the H2 (and energy) coming from renewable sources. These alternative fuels can be combusted themselves or be used to generate H2 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 Iwo 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 aboye 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 etchlng for the development of nanostructured catalytic coatings and supported nanoparticles. Porous carbon foams 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. Ti02-TiOx photocatalytic films with Pt (and/or gold) as co-catalysts will be

investlgated for the photo-reforming 01 methanol.

OTROS PROYECTOS / OTHER PROJECTS

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

Código/Code:	6079
Periodo/Period:	31-10-2019 / 30-10-2020
Organismo Financiador/Financial source:	Junta de Andalucia
Importe total/Total amount:	70.117 €
Investigador responsable/Research head:	Angel Barranco Quero

Red de Terapia Cellular (TERCEL)

Código/Code:	RD16/0011/0022
Periodo/Period:	01-01-2016 / 31-12-2020
Organismo Financiador/Financial source:	Instituto de Salud Carlos III
Importe total/Total amount:	182.286,50 €
Investigador responsable/Research head:	José Becerra Ratia (UMA)
Componentes/Research group:	Daniel Amat Trujillo (CIBER-BBN), Maria Aránzazu Díaz Cuenca, Leonor Santos Ruiz (CIBER-BBN)

Desarrollo de capas finas y nanoestructuradas porosas mediante técnicas de evaporación y plasmas para el desarrollo de materiales y sistemas de detección avanzados

Código/Code:	201560E055
Periodo/Period:	01-07-2015 / 30-06-2019
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	229.723 €
Investigador responsable/Research head:	Agustín R. González-Elipe

Sostenibilidad de las líneas estratégicas Materiales Nanoestructurados y Microestructura (NANOMAT)

Código/Code:	201760E002
Periodo/Period:	01-01-2017 / 31-12-2019
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	325.481 €
Investigador responsable/Research head:	Asunción Fernández Camacho

NanoDispositivos 3D (NanoD3D): Nanohilos y nanoárboles para la nueva generación de nanodispositivos autoalimentados

Código/Code:	EUIN2017-89059
Periodo/Period:	01-01-2018 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	10.000 €
Investigador responsable/Research head:	Ana Isabel Borras Martos

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:	Ministerio de Economía y Competitividad

Importe total/Total amount: 208.029 €
Investigador responsable/Research head: Agustín R. González-Elipe

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: Angel 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: 106.480 €
Investigador responsable/Research head: Agustin R. Gonzalez-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: 121.00 €
Investigador responsable/Research head: Agustin R. Gonzalez-Elipe

Caracterización superficial de membranas de ósmosis inversa

Periodo/Period: 06-04-2018 / 01-04-2020
Organismo Financiador/Financial source: ACCIONA AGUA, S.A.U.
Importe total/Total amount: 11.371 €
Investigador responsable/Research head: Juan Pedro Espinós Manzorro

Desarrollo de Capas PVD

Periodo/Period: 08-05-2017 / 07-05-2019
Organismo Financiador/Financial source: FLUBETECH, S.L.
Importe total/Total amount: 7.260 €
Investigador responsable/Research head: Juan Carlos Sánchez López

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Preparación de dispositivos microfluídicos magnetoópticos basados en películas delgadas magnéticos porosas

Código/Code:	COOPB20349
Periodo/Period:	01-01-2018 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad (Programa CSIC de Cooperación Científica para el desarrollo I-COOP+)
Importe total/Total amount:	19.900 €
Investigador responsable/Research head:	Francisco Yubero Valencia

Universidad Nacional de Ciencia y Tecnología de Rusia (National University of Science and Technology «MISiS») y la Agencia Estatal Consejo Superior de Investigaciones Científicas (CSIC) a través del Instituto de Ciencia de Materiales de Sevilla (ICMS)

Periodo/ Period:	02-12-2016 / 01-02-2019
Código/Code:	C-ES-2016-101
Referencia VATC:	20171119
Investigador responsable/Research head:	Juan Carlos Sánchez López

PATENTES / PATENTS

Composición biopolimérica, procedimiento su preparación y uso de la misma

Inventores: Sara Borrego González, María Aránzazu Díaz Cuenca, Berta De la Cerda Haynes (CABIMER)
Tipo de Patente: Nacional
Número de Solicitud: 201930963
Fecha Solicitud: 5 de noviembre de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas

Optofluidic device to monitor the evolution of liquid products by near-infrared spectroscopy

Inventores: Jorge Gil Rostra, Francisco Yubero Valencia, Agustín R. González-Elipe
Tipo de Patente: Nacional
Número de Solicitud: 19382834
Fecha Solicitud: 27 de septiembre de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas

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

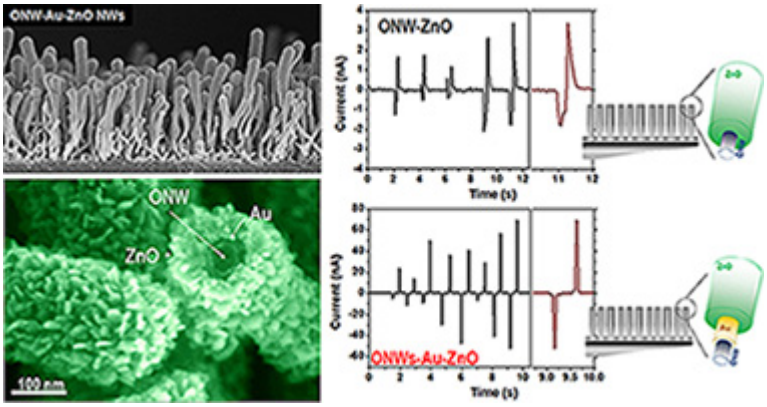
Inventores: Asunción Fernández Camacho, M. Carmen Jiménez de Haro, Dirk Hufschimdt, Vanda Fortio Godinho
Tipo de Patente: Internacional
Número de Solicitud: PCT/ES19/070729
Fecha Solicitud: 28 de octubre de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

3D core-multishell piezoelectric nanogenerators

A. Nicolas Filippin, Juan R.Sanchez-Valencia, Xabier Garcia-Casas, Victor Lopez-Flores, Manuel Macias-Montero, Fabian Frutos, Angel Barranco, Ana Borras
Nano Energy, **58** (2019) 476-483
Abril, 2019 | **DOI:** 10.1016/j.nanoen.2019.01.047

The thin film configuration presents obvious practical advantages over the 1D implementation in energy harvesting systems such as easily manufacturing and processing, and long-lasting and stable devices. However, ZnO-based piezoelectric nanogenerators (PENGs) generally rely on the exploitation of single-crystalline nanowires because of their self-orientation in the c-axis



direction and ability to accommodate long deformations resulting in high piezoelectric performance. Herein, we show an innovative approach to produce PENGs by combining polycrystalline ZnO layers fabricated at room temperature by plasma-assisted deposition with supported small-molecule organic nanowires (ONWs) acting as 1D scaffolds. Such hybrid nanostructures present convoluted core-shell morphology, formed by a single-crystalline organic nanowire conformally surrounded by a poly-crystalline ZnO shell and combine the organic core mechanical properties with the ZnO layer piezoelectric response. In a step forward towards the integration of multiple functions within a single wire, we have also developed ONW-Au-ZnO nanoarchitectures including a gold shell acting as inner electrode achieving output

piezo-voltages up to 170 mV. The synergistic combination of functionalities in the ONW-Au-ZnO devices promotes an enhanced performance generating piezo-currents one order of magnitude larger than the ONW-ZnO nanowires and superior to the thin film nanogenerators for equivalent and higher thicknesses.

Plasma Enabled Conformal and Damage Free Encapsulation of Fragile Molecular Matter: from Surface-Supported to On-Device Nanostructures

Alcaire, M, Aparicio, FJ, Obrero, J, Lopez-Santos, C, Garcia-Garcia, FJ, Sanchez-Valencia, JR, Frutos, F, Ostrikov, K, Borras, A, Barranco, A
Advanced Functional Materials, **29** (2019) 1903535
Septiembre, 2019 | **DOI:** 10.1002/adfm.201903535

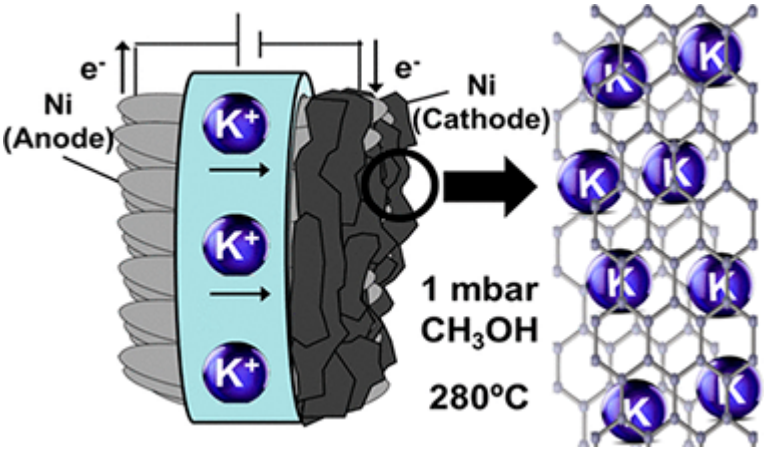
Damage-free encapsulation of molecular structures with functional nanolayers is crucial to protect nanodevices from environmental exposure. With nanoscale electronic, optoelectronic, photonic, sensing, and other nanodevices based on atomically thin and fragile organic matter shrinking in size, it becomes increasingly challenging to develop nanoencapsulation that is simultaneously conformal at atomic scale and does not damage fragile molecular networks, while delivering added device functionality. This work presents an effective, plasma-enabled, potentially universal approach to produce highly conformal multifunctional organic films to encapsulate atomically thin graphene layers and metalorganic nanowires, without affecting their molecular structure and atomic bonding. Deposition of adamantane precursor and gentle remote plasma chemical vapor deposition are synergized to assemble molecular fragments and cage-like building blocks and completely encapsulate not only the molecular structures, but also the growth substrates and device elements upon nanowire integration. The films are insulating, transparent, and conformal at sub-nanometer scale even on near-tip high-curvature areas of high-aspect-ratio nanowires. The encapsulated structures are multifunctional and provide effective electric isolation, chemical and environmental protection, and transparency in the near-UV-visible-near-infrared range. This single-step, solvent-free remote-plasma approach preserves and guides molecular building blocks thus opening new avenues for precise, atomically conformal nanofabrication of fragile nanoscale matter with multiple functionalities.

Graphene Formation Mechanism by the Electrochemical Promotion of a Ni Catalyst

Espinos, JP, Rico, VJ, Gonzalez-Cobos, J, Sanchez-Valencia, JR, Perez-Dieste, V, Escudero, C, de Lucas-Consuegra, A, Gonzalez-Elipe, AR
ACS Catalysis, **9** (2019) 11447-11454
Diciembre, 2019 | **DOI:** 10.1021/acscatal.9b03820

In this work, we show that multilayer graphene forms by methanol decomposition at 280 degrees C on an electrochemically promoted nickel catalyst film supported on a K-beta Al2O3 solid electrolyte. In operando near ambient pressure photoemission spectroscopy and electrochemical measurements have shown that polarizing negatively the Ni electrode induces the electrochemical reduction and migration of potassium to the nickel surface. This elemental potassium promotes the catalytic decomposition of methanol into graphene and also stabilizes the graphene formed via diffusion and direct K-C interaction. Experiments reveal that adsorbed

methoxy radicals are intermediate species in this process and that, once formed, multilayer graphene remains stable after electrochemical oxidation and back migration of potassium to the solid electrolyte upon positive polarization. The reversible diffusion of ca. 100 equivalent



monolayers of potassium through the carbon layers and the unprecedented low-temperature formation of graphene and other carbon forms are mechanistic pathways of high potential impact for applications where mild synthesis and operation conditions are required.

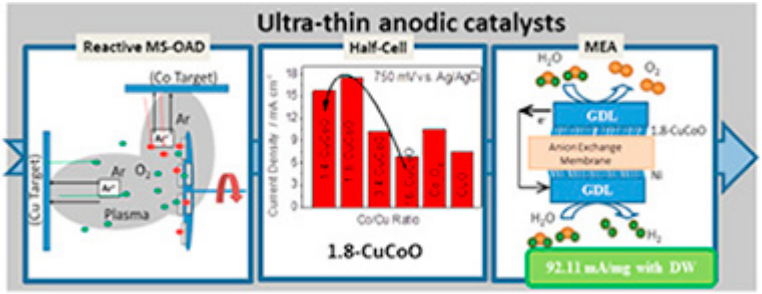
$Cu_xCo_{3-x}O_4$ ultra-thin film as efficient anodic catalysts for anion exchange membrane water electrolysers

Lopez-Fernandez, E, Gil-Rostra, J, Espinos, JP, Gonzalez-Elipe, AR, Yubero, F, de Lucas-Consuegra, A

Journal of Power Sources, **415** (2019) 136-144

Marzo, 2019 | **DOI:** 10.1016/j.jpowsour.2019.01.056

CuxCo3-xO4 ultra-thin films, deposited by magnetron sputtering at oblique angles have been used as anodic catalysts in anion exchange membrane water electrolysers. It has been demonstrated that the used deposition procedure provides porous and amorphous samples



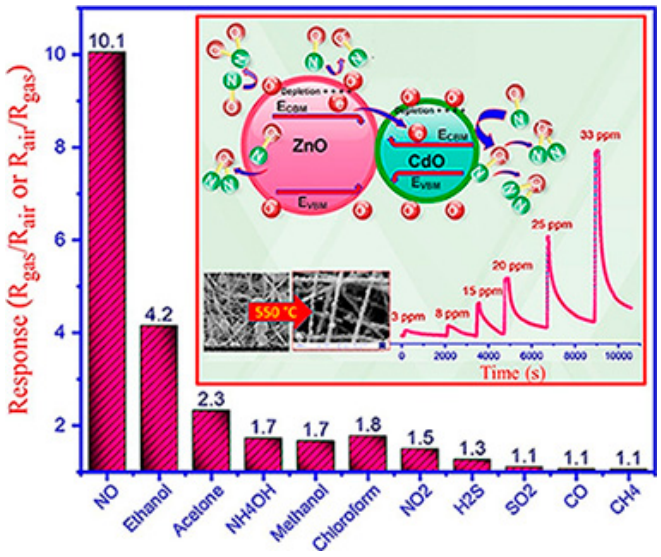
with a strict control of the total catalyst load and Co/Cu ratio. Electrocatalytic tests showed a maximum performance for the oxygen evolution reaction at Co/Cu atomic ratio around 1.8. The optimized anodic catalyst presented a long-term stability confirmed by accelerated lifetime tests together with the chemical surface analysis of the used samples. The effect of the crystallization of a single layer CuxCo3-xO4 and a multilayer (CuO/Co3O4)(n) anodic catalyst samples was also investigated. The observed loss of catalytic performance found in both cases may prove that a

particular local chemical environment around the Co and Cu sites acts as an efficient catalytic site for the oxygen evolution reaction. A catalyst film with the optimum Co/Cu atomic ratio was incorporated into a Membrane Electrode Assembly, using a sputtered Ni film as cathode. Current density values up to 100 mA cm(-2) at 2.0 V were obtained in 1.0 M KOH electrolyte. Upon normalization by the amount of catalyst, this performance is one of the highest reported in literature.

Highly selective few-ppm NO gas-sensing based on necklace-like nanofibers of ZnO/CdO n-n type I heterojunction

Naderi, H, Hajati, S, Ghaedi, M, Espinos, JP
Sensors and Actuators B-Chemical, **297** (2019) 126774
Octubre, 2019 | **DOI:** 10.1016/j.snb.2019.126774

Electrospinning method followed by calcination is applied to synthesize ZnO/CdO nanofibers. Characterization is performed by X-ray diffraction (XRD), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS), diffuse reflectance spectroscopy (DRS), X-ray photoelectron spectroscopy (XPS), ultraviolet



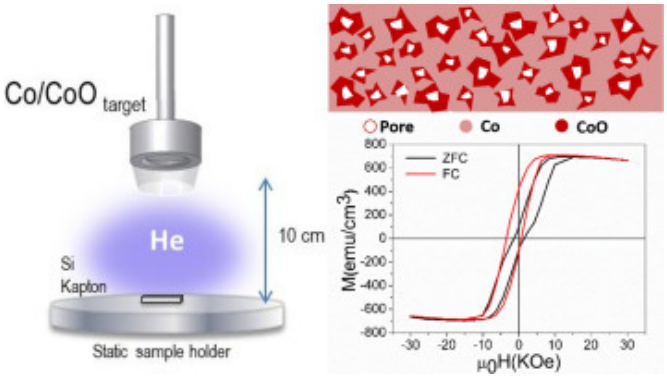
photoelectron spectroscopy (UPS) and reflection electron energy loss spectroscopy (REELS), which resulted in detailed analysis of the sensing material. For instance, it was found that the ZnO/CdO is n-n type I heterojunction which possesses straddling energy band gap, which could affect the mechanism of gas sensing. An electroless gold-plated interdigitated electrode with spacing 200 mu m is fabricated on alumina substrate to host the designed nanofibers being used as gas sensor. Gas-sensing activity of the heterojunction is investigated against NO, NO2, H2S, CH4, SO2 and CO in addition to VOCs such as ethanol, acetone, ammonia, methanol, and chloroform with high selectivity and response to NO gas by monitoring resistance changes. Detailed discussion on the mechanism of sensing is presented. The ZnO/CdO nanofibers are found to be highly sensitive to very low concentration range of NO gas (1.2-33 ppm) at optimal operating temperature of 215 degrees C. The influence of humidity (20-96%) on the sensor response was found to be ignorable. Additionally, good repeatability and long-term stability (45

days, every 5 days, SD = 0.7) was obtained for this sensor. Typically, short response times of 47 and 35 s are obtained versus 3 and 33 ppm of NO, respectively, making our sensor promisingly applicable for monitoring this toxic gas in polluting industries, metropolises and maybe in exhaled breath.

Exchange bias and two steps magnetization reversal in porous Co/CoO layer

Ovejero, JG, Godinho, V, Lacroix, B, Garcia, MA, Hernando, A, Fernandez, A
Materials & Design, **171** (2019) 107691
Junio, 2019 | **DOI:** 10.1016/j.matdes.2019.107691

In this paper Co/CoO thick layers (hundreds of nanometers) of different porosity and oxidation degree were prepared in a magnetron sputtering deposition processby tailoring the DC sputtering power, as well as the process gas and target composition. The control of the synthesis



parameters allowed the nanostructuration of the films with a singular distribution of closed pores and a controlled amount of CoO. We observed an exchange bias field of 2.8 KOe for porous Co/CoO composites, similar to Co/CoO bilayers but for coatings thicker than 300 nm. Besides, it was observed that the coating presents bistable magnetic features when cooled under zero field conditions as a result of the unusual exchange coupling.

Sodium ion storage performance of magnetron sputtered WO3 thin films

Garcia-Garcia, FJ, Mosa, J, Gonzalez-Elipe, AR, Aparicio, M
Electrochimica Acta, **321** (2019) 134669
Octubre, 2019 | **DOI:** 10.1016/j.electacta.2019.134669

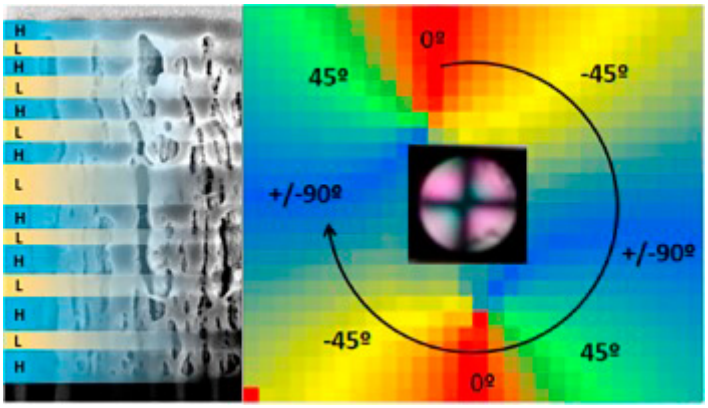
WO_3 thin film electrodes were successfully prepared by magnetron sputtering (MS) deposition under an oblique angle configuration (OAD). Intercalation of Na ions in the tungsten oxide layers has been studied using electrochemical techniques. Sample characterization before and after sodium intercalation has been carried out by Raman, XPS and XRD measurements. ToF-SIMS analysis has been also performed in order to analyze the element depth profiles along the electrode thickness. Electron microscopy evaluation of the cross section confirms the porous structure of the coatings. Batteries integrating these WO_3 electrodes have a discharge capacity of 120 mA h g(-1) at the initial cycles and show an adequate capacity retention upon 300 cycles. The WO_3-OAD thin-films are proposed as promising electrodes for Na-ion batteries.

Liquid switchable radial polarization converters made of sculptured thin films

Oliva-Ramirez, M, Rico, VJ, Gil-Rostra, J, Arteaga, O, Bertran, E, Serna, R, Gonzalez-Elip, AR, Yubero, F

Applied Surface Science, **475** (2019) 230-236
Mayo, 2019 | **DOI:** 10.1016/j.apsusc.2018.12.200

A radial polarization converter is a super-structured optical retarder that converts a conventional linearly polarized light beam into a structured beam with radial or azimuthal polarization. We present a new type of these sophisticated optical elements, which is made of porous nanostructured sculptured single thin films or multilayers prepared by physical vapor



deposition at an oblique angle. They are bestowed with an axisymmetric retardation activity (with the fast axis in a radial configuration). In particular, a Bragg microcavity multilayer that exhibits a tunable transmission peak in the visible range with a retardance of up to 0.35 rad has been fabricated using this methodology. Owing to the highly porous structure of this type of thin films and multilayers, their retardance could be switched off by liquid infiltration. These results prove the possibility of developing wavelength dependent (through multilayer optical design) and switchable (through vapor condensation or liquid infiltration within the pore structure) radial polarization converters by means of oblique angle physical vapor deposition.

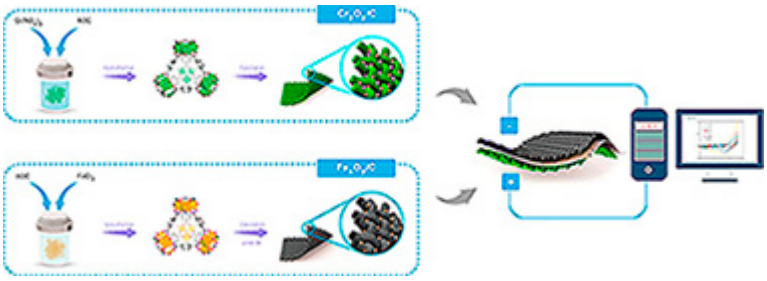
Controlled thermolysis of MIL-101(Fe, Cr) for synthesis of FexOy/porous carbon as negative electrode and Cr_2O_3/porous carbon as positive electrode of supercapacitor

Farisabadi, A, Moradi, M, Hajati, S, Kiani, MA, Espinos, JP

Applied Surface Science, **469** (2019) 192-203
Marzo, 2019 | **DOI:** 10.1016/j.apsusc.2018.11.053

In the present study, two kinds of metal oxide/carbon nanocomposite were prepared through calcination of MIL-101(Fe, Cr). The morphological and structural properties of the specimens were investigated using X-ray diffraction, Fourier-transform infrared spectroscopy, Brunauer, Emmett, and Teller analysis, energy dispersive Xray spectroscopy, X-ray photoelectron spectroscopy and scanning electron microscopy. The electrode materials were also electrochemically investigated using cyclic voltammetry, galvanostatic charge-discharge and electrochemical impedance spectroscopy techniques in 6 M KOH electrolyte. Because of synergistic effect of metal oxides and carbon, the obtained samples showed excellent

performance; in a way that Cr2O3/C and Fe Oy/C showed high specific capacitance of 420 F g(-1) and 114 F g(-1) at current density of 2 A g(-1), respectively. The Cr2O3/C electrode also displayed high rate capability even at scan rate of 1500 mV s(-1). Moreover, we successfully



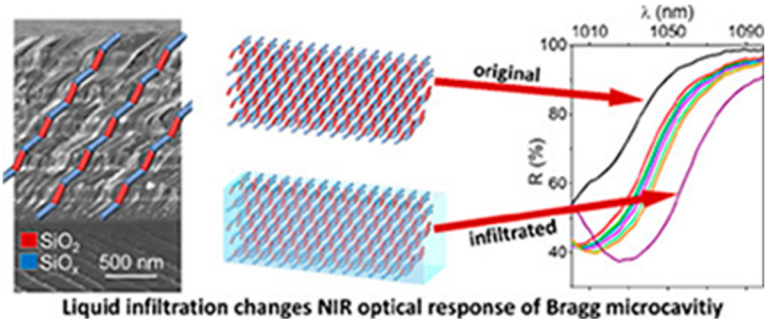
developed an asymmetric supercapacitor in which Cr2O3/C served as positive electrode and Fe Oy/C served as negative electrode. The asymmetric device can deliver an energy density of 9.6 W h kg(-1) and power density of 8000 W kg(-1), with 93% capacitance retention after 3000 charge-discharge cycles. These outcomes show that the MOF-derived metal oxide/carbon composite can be regarded as a promising development for advanced electrode materials for applying in supercapacitors.

SiOx by magnetron sputtered revisited: Tailoring the photonic properties of multilayers

Garcia-Valenzuela, A, Alvarez, R, Espinos, JP, Rico, V, Gil-Rostra, J, Palmero, A, Gonzalez-Elipe, AR

Applied Surface Science, **488** (2019) 791-800
Septiembre, 2019 | **DOI:** 10.1016/j.apsusc.2019.05.273

Traditionally porous silicon based photonic structures have been prepared by electrochemically etching of silicon. In this work, porous multilayers of nanocolumnar SiOx and SiO2 thin films acting as near infrared (NIR) 1D-photonic nanostructures are prepared by magnetron sputtering



Liquid infiltration changes NIR optical response of Bragg microcavitiy

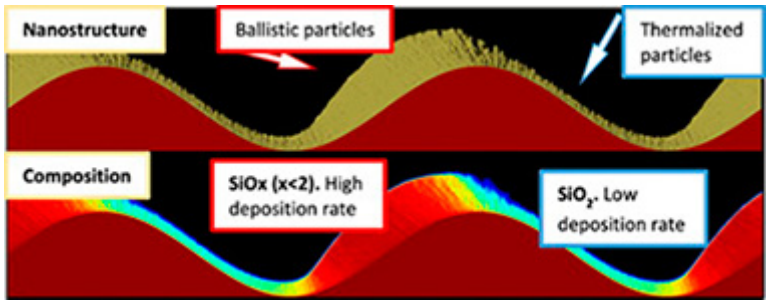
deposition at oblique angles (MS-OA). Simultaneous control of porosity and stoichiometry of the stacked films is achieved by adjusting the deposition angle and oxygen partial pressure according to a parametric formula. This new methodologoy is proved for the synthesis of SiOx thin films with x close to 0.4, 0.8, 1.2, 1.6 and nanostructures varying from compact (at 0 degrees deposition angle) to highly porous and nanocolumnar (at 70 degrees and 85 degrees deposition angles). The strict control of composition, structure and nanostructure provided by this technique permits a fine tuning of the absorption edge and refraction index at 1500 nm of the

porous films and their manufacturing in the form of SiOx-SiO2 porous multilayers acting as near infrared (NIR) 1D-photonic structures with well-defined optofluidic responses. Liquid tunable NIR Bragg mirrors and Bragg microcavities for liquid sensing applications are presented as proof of concept of the possibilities of this MS-OA manufacturing method as an alternative to the conventional electrochemical fabrication of silicon based photonic structures.

2D compositional self-patterning in magnetron sputtered thin films

Garcia-Valenzuela, A, Alvarez, R, Rico, V, Espinos, JP, Lopez-Santos, MC, Solis, J, Siegel, J, del Campo, A, Palmero, A, Gonzalez-Elipe, AR
Applied Surface Science, **480** (2019) 115-121
Junio, 2019 | **DOI:** 10.1016/j.apsusc.2019.02.206

Unlike topography patterning, widely used for numerous applications and produced by means of different technologies, there are no simple procedures to achieve surface compositional patterning at nanometric scales. In this work we have developed a simple method for 2D



patterning the composition of thin films. The method relies on the magnetron sputtering deposition at oblique angles onto patterned substrates made by laser induced periodic surface structures (LIPSS). The method feasibility has been demonstrated by depositing SiOx thin films onto LIPSS structures generated in Cr layers. A heterogeneous and aligned distribution of O/Si ratios (and different Sin+ chemical states) along the LIPSS structure in length scales of some hundreds nm's has been proven by angle resolved X-ray photoelectron spectroscopy and a patterned arrangement of composition monitored by atomic force microscopy-Raman analysis. The obtained results are explained by the predictions of a Monte Carlo simulation of this deposition process and open the way for the tailored one-step fabrication of surface devices with patterned compositions.

Study of the Interface of the Early Stages of Growth under Quasi-Equilibrium Conditions of ZnO on Graphene/Cu and Graphite

Morales, C., Black, A., Urbanos, F.J., Granados, D., Mendez, J., del Campo, A., Yubero, F., Soriano, L.
Advanced Materials Interfaces, **6** (2019) 1801689
Febrero, 2019 | **DOI:** 10.1002/admi.201801689

The study of the early stages of growth of ZnO on graphene supported on Cu and on highly oriented pyrolytic graphite by means of reactive thermal evaporation of metallic Zn at room temperature is presented. This growth method allows to go in depth in the study of the

fundamental interaction between ZnO and graphene at the interface in quasi-equilibrium conditions. Quantitative, chemical, and morphological analysis is performed using photoemission spectroscopy, atomic force, and scanning microscopies as experimental characterization techniques and factor analysis and inelastic peak shape analysis as modeling techniques. The growth of ZnO on a highly oriented pyrolytic graphite substrate is also studied using the same growth method for comparison. The results show that, in spite that the first atomic layer of both substrates is identical, the growth kinetics and morphology of the deposits are completely different. A model for the kinetics of the growth of ZnO on both substrates is proposed.

Environmentally Tight TiO_2-SiO_2 Porous 1D-Photonic Structures

Garcia-Valenzuela, A, Lopez-Santos, C, Rico, V, Alvarez, R, Palmero, A, Gonzalez-Elipe, AR
Advanced Materials Interfaces, **6** (2019) 1801212
Febrero, 2019 | **DOI:** 10.1002/admi.201801212

Although thin film porosity is the basis of many optical sensors, it can be deleterious for a stable optical behavior of passive optical elements due to the condensation of water and other vapors in their pores. This paper proposes a new strategy for the magnetron sputtering (MS) fabrication of environmentally tight SiO_2-TiO_2 porous multilayers. Thin films of these two oxides deposited in an oblique angle configuration (MS-OAD) present a nanocolumnar and highly porous nanostructure and, as a consequence, experience significant changes in their optical properties when exposed to water vapor. Similarly, the optical properties of Bragg reflectors and Bragg microcavities made of the stacking of porous and compact SiO_2 and TiO_2 thin films experience reversible changes when these 1D-photonic structures are exposed to water pressure. A key finding of this work is that a very thin capping layer of SiO_2 deposited on the surface of porous SiO_2 films in the stack, at the interlayer between the two oxides, efficiently seals the pores making the photonic structures environmentally tight. This capping layer approach is a useful strategy to incorporate porosity as an additional parameter to design the optical behavior of planar photonic structures while preserving optical and environmental stability.

On-Surface Synthesis and Characterization of Acene-Based Nanoribbons Incorporating Four-Membered Rings

Sanchez-Sanchez, C, Dienel, T, Nicolai, A, Kharche, N, Liang, LB, Daniels, C, Meunier, V, Liu, JZ, Feng, XL, Mullen, K, Sanchez-Valencia, JR, Groning, O, Ruffieux, P, Fasel, R
Chemistry-A European Journal, **25** (2019) 12074-12082
Septiembre, 2019 | **DOI:** 10.1002/chem.201901410

A bottom up method for the synthesis of unique tetracene-based nanoribbons, which incorporate cyclobutadiene moieties as linkers between the acene segments, is reported. These structures were achieved through the formal [2+2] cycloaddition reaction of ortho-functionalized tetracene precursor monomers. The formation mechanism and the electronic and magnetic properties of these nanoribbons were comprehensively studied by means of a multitechnique approach. Ultra-high vacuum scanning tunneling microscopy showed the occurrence of metal-coordinated nanostructures at room temperature and their evolution into nanoribbons through formal [2+2] cycloaddition at 475 K. Frequency-shift non-contact atomic

force microscopy images clearly proved the presence of bridging cyclobutadiene moieties upon covalent coupling of activated tetracene molecules. Insight into the electronic and vibrational properties of the so-formed ribbons was obtained by scanning tunneling microscopy, Raman spectroscopy, and theoretical calculations. Magnetic properties were addressed from a computational point of view, allowing us to propose promising candidates to magnetic acene-based ribbons incorporating four-membered rings. The reported findings will increase the understanding and availability of new graphene-based nanoribbons with high potential in future spintronics.

Ultrastable CoxSiyOz Nanowires by Glancing Angle Deposition with Magnetron Sputtering as Novel Electrocatalyst for Water Oxidation

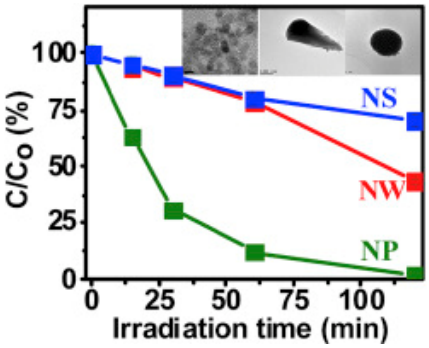
Cano, M, Garcia-Garcia, FJ, Rodriguez-Padron, D, Gonzalez-Elipe, AR, Giner-Casares, JJ, Luque, R
Chemcatchem, **11** (2019) 6111-6115
Diciembre, 2019 | **DOI:** 10.1002/cctc.201901730

Cobalt is one of the most promising non-noble metal as electrocatalyst for water oxidation. Herein, a highly stable silicon-cobalt mixed oxide thin film with a porous columnar nanostructure is proposed as electrocatalyst for oxygen evolution reaction (OER). CoOx and CoxSiyOz layers with similar thickness were fabricated at room temperature by magnetron sputtering in a glancing angle configuration (MS-GLAD) on tin-doped indium oxide (ITO) substrates. After characterization, a comparative study of the electrocatalytic performance for OER of both layers was carried out. The excellent long-term stability as electrocatalyst for OER of the porous CoxSiyOz thin film demonstrates that the presence of silicon on the mixed oxide network increases the mechanical stability of the Si/Co layer, whilst maintaining a considerable electrocatalytic response.

Morphological effects on the photocatalytic properties of SnO_2 nanostructures

Kar, A, Olszowka, J, Sain, S, Sloman, SRI, Montes, O, Fernandez, A, Pradhan, SK, Wheatley, AEH
Journal of Alloys and Compounds, **810** (2019) UNSP 151718
Noviembre, 2019 | **DOI:** 10.1016/j.jallcom.2019.151718

The photocatalytic properties of SnO_2 nanocrystals are tuned by varying their morphology and



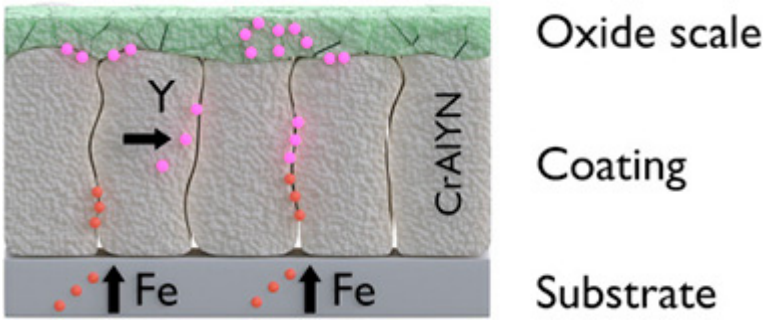
microstructure. SnO_2 nanoparticles and nanowedges have been synthesized using hydrothermal methods, while microwave irradiation techniques have given nanospheres. Detailed structural

and chemical characterization of these different morphologies has been accomplished. The influence of SnO_2 morphology on photocatalytic activity has been examined by monitoring the degradation of aqueous methylene blue dye. Results demonstrate that changing the morphology of the SnO_2 modulates both surface area and levels of surface defects and that these alterations are reflected in the photocatalytic properties of the materials. The degradation of methylene blue dye (98%) in the presence of SnO_2 nanoparticles under simulated solar irradiation is superior to previously reported photocatalyst performance and is comparable to that of standard TiO_2 (Degussa P-25). The SnO_2 nanoparticles perform better than both the nanowedges and nanospheres and this is attributed to the number of surface defects available to the high surface area material. They also reveal outstanding recyclability and stability.

Influence of Al and Y content on the oxidation resistance of CrAlYN protective coatings for high temperature applications: New insights about the Y role

Rojas, TC, Dominguez-Meister, S, Brizuela, M, Sanchez-Lopez, JC
Journal of Alloys and Compounds, **777** (2019) 1172-1181
Enero, 2019 | **DOI:** 10.1016/j.jallcom.2018.09.280

CrAlYN hard coatings with two different average Al contents: similar to 16 at.% and similar to 25 at.%, and Y concentration varying between 1.2 and 5.7 at.% were deposited by direct current reactive magnetron co-sputtering of mixed Cr-Al and Y targets on commercial M2 steel substrates. The samples were heated to 1000 degrees C in air during 2 h to study their oxidation



resistance and thermal stability. The Y content is critical and the coatings present different behaviour depending on the Al content. The best oxidation resistance and thermal stability are obtained for the coating with similar to 16 at.% Al and 3.4 at.% Y. The initial film microstructure and the cubic phase (fcc-CrAlN) were retained, and a thin (Cr,Al)(2)O-3 oxide protective scale was formed. At lower Y content (1.2 at.%) iron, from the substrate crosses the coating, while a higher content (4.6 at.%) avoided the iron diffusion at the expense of a thicker oxide scale with new oxide phases. The coatings with higher Al content (similar to 25 at. %) were not thermally stable at 1000 degrees C. A good oxidation resistance was obtained for 2.6 at.% of Y although new phases (hcp-AlN and Cr-Fe) were formed. Higher amount of yttrium (similar to 5.7 at. %) led to the complete oxidation of the coating.

Influence of Titanium Oxide Pillar Array Nanometric Structures and Ultraviolet Irradiation on the Properties of the Surface of Dental Implants-A Pilot Study

Santiago, JA, Fernandez-Martinez, I, Wennberg, A, Molina-Aldareguia, JM, Castillo-Rodriguez, Leon-Ramos, JR, Diosdado-Cano, JM, Lopez-Santos, C, Barranco, A, Torres-Lagares, D, Serrera-Figallo, MA

Nanomaterials, **9** (2019) 1458
Octubre, 2019 | **DOI:** 10.3390/nano9101458

Aim: Titanium implants are commonly used as replacement therapy for lost teeth and much current research is focusing on the improvement of the chemical and physical properties of their surfaces in order to improve the osseointegration process. TiO2, when it is deposited in the form of pillar array nanometric structures, has photocatalytic properties and wet surface control, which, together with UV irradiation, provide it with superhydrophilic surfaces, which may be of interest for improving cell adhesion on the peri-implant surface. In this article, we address the influence of this type of surface treatment on type IV and type V titanium discs on their surface energy and cell growth on them. Materials and methods: Samples from titanium rods used for making dental implants were used. There were two types of samples: grade IV and grade V. In turn, within each grade, two types of samples were differentiated: untreated and treated with sand blasting and subjected to double acid etching. Synthesis of the film consisting of titanium oxide pillar array structures was carried out using plasma-enhanced chemical vapor deposition equipment. The plasma was generated in a quartz vessel by an external SLAN-1 microwave source with a frequency of 2.45 GHz. Five specimens from each group were used (40 discs in total). On the surfaces to be studied, the following determinations were carried out: (a) X-ray photoelectron spectroscopy, (b) scanning electron microscopy, (c) energy dispersive X-ray spectroscopy, (d) profilometry, (e) contact angle measurement or surface wettability, (f) progression of contact angle on applying ultraviolet irradiation, and (g) a biocompatibility test and cytotoxicity with cell cultures. Results: The application of ultraviolet light decreased the hydrophobicity of all the surfaces studied, although it did so to a greater extent on the surfaces with the studied modification applied, this being more evident in samples manufactured in grade V titanium. In samples made in grade IV titanium, this difference was less evident, and even in the sample manufactured with grade IV and SLA treatment, the application of the nanometric modification of the surface made the surface optically less active. Regarding cell growth, all the surfaces studied, grouped in relation to the presence or not of the nanometric treatment, showed similar growth. Conclusions. Treatment of titanium oxide surfaces with ultraviolet irradiation made them change temporarily into superhydrophilic ones, which confirms that their biocompatibility could be improved in this way, or at least be maintained.

Antibacterial Nanostructured Ti Coatings by Magnetron Sputtering: From Laboratory Scales to Industrial Reactors

Alvarez, R, Munoz-Pina, S, Gonzalez, MU, Izquierdo-Barba, I, Fernandez-Martinez, I, Rico, V, Arcos, D, Garcia-Valenzuela, A, Palmero, A, Vallet-Regi, M, Gonzalez-Elipe, AR, Garcia-Martin, JM

Nanomaterials, **9** (2019) 1217
Septiembre, 2019 | **DOI:** 110.3390/nano9091217

Based on an already tested laboratory procedure, a new magnetron sputtering methodology to simultaneously coat two-sides of large area implants (up to similar to 15 cm(2)) with Ti nanocolumns in industrial reactors has been developed. By analyzing the required growth conditions in a laboratory setup, a new geometry and methodology have been proposed and tested in a semi-industrial scale reactor. A bone plate (DePuy Synthes) and a pseudo-rectangular bone plate extracted from a patient were coated following the new methodology, obtaining that their osteoblast proliferation efficiency and antibacterial functionality were equivalent to the coatings grown in the laboratory reactor on small areas. In particular, two kinds of experiments were performed: Analysis of bacterial adhesion and biofilm formation, and osteoblasts-bacteria competitive in vitro growth scenarios. In all these cases, the coatings show an opposite behavior toward osteoblast and bacterial proliferation, demonstrating that the proposed methodology represents a valid approach for industrial production and practical application of nanostructured titanium coatings.

Silver effect on the tribological and antibacterial properties of a-C:Ag coatings

Dominguez-Meister, S, Rojas, TC, Frias, JE, Sanchez-Lopez, JC
Tribology International, **140** (2019) UNSP 105837
Diciembre, 2019 | **DOI:** 10.1016/j.triboint.2019.06.030

a-C:Ag coatings (1.2-23.4 at.% of Ag) were deposited using magnetron sputtering. Ag nanoparticles appear embedded in the carbon matrix or segregated to the column boundaries or surface. The silver doping has not promoted significant changes of the sp(2)/sp(3) ratio although a decrease of the hardness is observed (from 17 to 7 GPa). The tribological behavior did not show a clear dependence on the silver concentration in unlubricated or lubricated conditions (fetal bovinum serum) against alumina or UHMWPE balls. Ag nanoparticle dispersion enhanced the bactericide behavior as determined by the released Ag+ ion in the fluid media. There is no clear effect of friction rubbing on the released silver indicating that diffusion and top segregation are prevalent mechanisms for its dissolution.

The impact of photocatalytic Ag/TiO_2 and Ag/N-TiO_2 nanoparticles on human keratinocytes and epithelial lung cells

Rebleanu, D, Gaidau, C, Voicu, G, Constantinescu, CA, Sanchez, CM, Rojas, TC, Carvalho, S, Calin, M
Toxicology, **416** (2019) 30-43
Marzo, 2019 | **DOI:** 110.1016/j.tox.2019.01.013

The potential human health risks following the exposure to inorganic nanoparticles (NPs) is a very important issue for their application in leather finishing industry. The aim of our study was to investigate the cytotoxic effect of silver (Ag)/titanium dioxide (TiO_2) NPs on human cells. Photocatalytic NPs were prepared by electrochemical deposition of Ag on the surface of TiO_2 and nitrogen (N)-TiO_2 NPs and, subsequently, physicochemical characterized. Then, a set of experiments have been performed to study the cytotoxicity and cell death mechanisms involved, the changes in cell morphology and the production of ROS induced in human keratinocytes (HaCaT) and human lung epithelial cells (A549) by exposure to NPs. Moreover, the changes in major signaling pathways and the inflammatory response induced by Ag/N-TiO_2 NPs

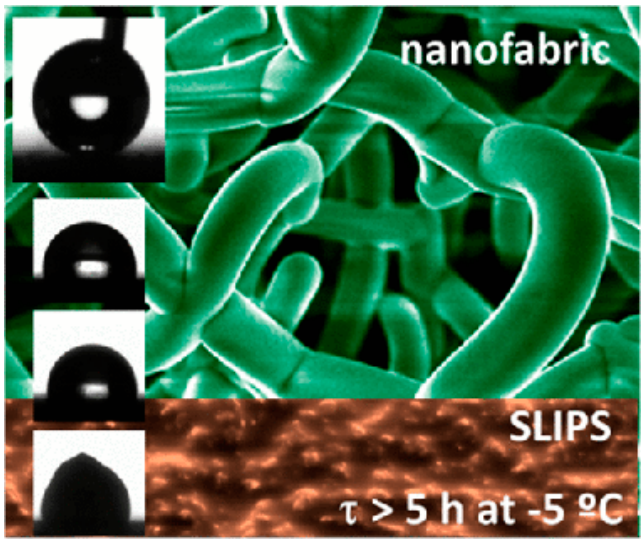
in A549 cells were investigated. The data showed that cell death by late apoptosis/necrosis is induced in cells as function of the dose and the type of NPs and is characterized by morphological changes and cytoskeletal disorganization and an increase in reactive oxygen species (ROS) production. The exposure of A549 cells to Ag/N-TiO_2 NPs determine the activation of ERK1/2 MAP-kinase pathway and the release of pro-inflammatory mediators CXCL1, GM-CSF and MIF, known to be involved in the recruitment of circulating neutrophils and monocytes.

3D Organic Nanofabrics: Plasma-Assisted Synthesis and Antifreezing Behavior of Superhydrophobic and Lubricant-Infused Slippery Surfaces

Alcaire, M, Lopez-Santos, C, Aparicio, FJ, Sanchez-Valencia, JR, Obrero, JM, Saghi, Z, Rico, VJ, de la Fuente, G, Gonzalez-Elipe, AR, Barranco, A, Borras, A

Langmuir, **35** (2019) 16876-16885
Diciembre, 2019 | **DOI:** 10.1021/acs.langmuir.9b03116

Herein, we present the development of supported organic nanofabrics formed by a conformal polymer-like interconnection of small-molecule organic nanowires and nanotrees. These organic nanostructures are fabricated by a combination of vacuum and plasma-assisted deposition techniques to generate step by step, single-crystalline organic nanowires forming one-



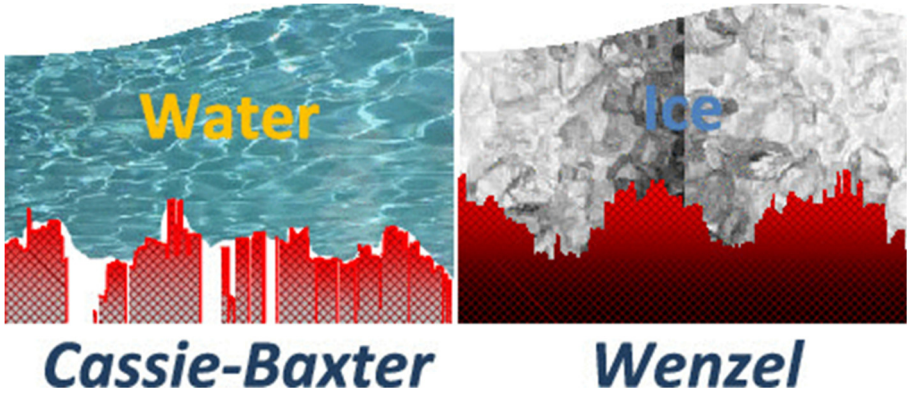
dimensional building blocks, organic nanotrees applied as three-dimensional templates, and the polymer-like shell that produces the final fabric. The complete procedure is carried out at low temperatures and is compatible with an ample variety of substrates (polymers, metal, ceramics; either planar or in the form of meshes) yielding flexible and low solid-fraction three-dimensional nanostructures. The systematic investigation of this progressively complex organic nanomaterial delivers key clues relating their wetting, nonwetting, and anti-icing properties with their specific morphology and outer surface composition. Water contact angles higher than 150° are attainable as a function of the nanofabric shell thickness with outstanding freezing-delay times (FDT) longer than 2 h at −5 °C. The role of the extremely low roughness of the shell surface is settled as a critical feature for such an achievement. In addition, the characteristic interconnected microstructure of the nanofabrics is demonstrated as ideal for the fabrication of slippery liquid-infused porous surfaces (SLIPS). We present the straightforward deposition of the

nanofabric on laser patterns and the knowledge of how this approach provides SLIPS with FDTs longer than 5 h at −5 °C and 1 h at −15 °C.

Hydrophobicity, Freezing Delay, and Morphology of Laser-Treated Aluminum Surfaces

Rico, VJ, Lopez-Santos, C, Villagra, M, Espinos, JP, de la Fuente, GF, Angurel, LA, Borras, A, Gonzalez-Elipe, AR
Langmuir, **35** (2019) 6483-6491
Mayo, 2019 | **DOI:** 10.1021/acs.langmuir.9b00457

Until recently, superhydrophobicity was considered as a hint to predict surface icephobicity, an association of concepts that is by no means universal and that has been proven to depend on different experimental factors and material properties, including the actual morphology and chemical state of surfaces. This work presents a systematic study of the wetting and freezing



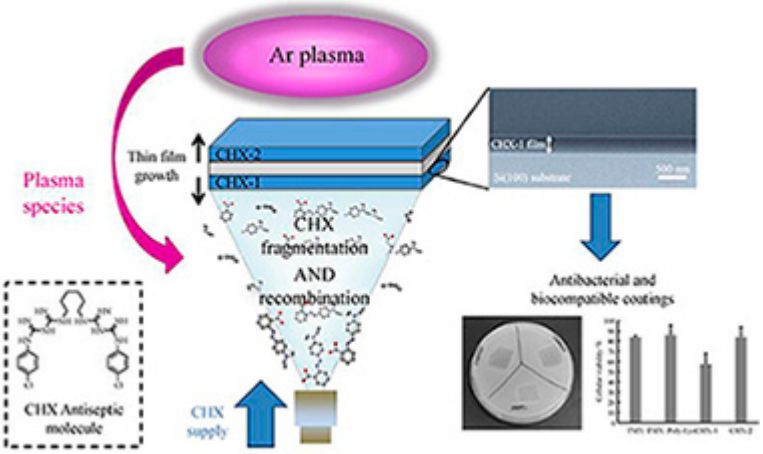
properties of aluminum Al6061, a common material widely used in aviation, after being subjected to nanosecond pulsed IR laser treatments to modify its surface roughness and morphology. All treated samples, independent of their surface finishing state, presented initially an unstable hydrophilic wetting behavior that naturally evolved with time to reach hydrophobicity or even superhydrophobicity. To stabilize the surface state and to bestow the samples with a permanent and stable hydrophobic character, laser-treated surfaces were covered with a thin layer of CFx prepared by plasma-enhanced chemical vapor deposition. A systematic comparison between freezing delay (FD) and wetting properties of water droplets onto these plasma-/polymer-modified laser-treated surfaces that, under conditions where a heterogeneous nucleation mechanism prevails, surface morphology rather than the actual value of the surface roughness parameter the key feature for long FD times. In particular, it is found that surface morphologies rendering a Cassie-Baxter wetting regime longer FDs than those characterized by a Wenzel-like wetting state. It is that laser treatment, with or without additional coverage with thin CFx coatings, affects wetting and ice formation behaviors and might be an efficient procedure to mitigate icing problems on metal surfaces.

Multifunctional antimicrobial chlorhexidine polymers by remote plasma assisted vacuum deposition

Mora-Boza, A, Aparicio, FJ, Alcaire, M, Lopez-Santos, C, Espinos, JP, Torres-Lagares, D, Borras, A, Barranco, A

Frontiers of chemical science and engineering, **13** (2019) 330-339
Junio, 2019 | **DOI:** 10.1007/s11705-019-1803-6

Novel antibacterial materials for implants and medical instruments are essential to develop practical strategies to stop the spread of healthcare associated infections. This study presents the synthesis of multifunctional antibacterial nanocoatings on polydimethylsiloxane (PDMS) by remote plasma assisted deposition of sublimated chlorhexidine powders at low pressure and



room temperature. The obtained materials present effective antibacterial activity against Escherichia coli K12, either by contact killing and antibacterial adhesion or by biocide agents release depending on the synthetic parameters. In addition, these multifunctional coatings allow the endure hydrophilization of the hydrophobic PDMS surface, thereby improving their biocompatibility. Importantly, cell-viability tests conducted on these materials also prove their non-cytotoxicity, opening a way for the integration of this type of functional plasma films in biomedical devices.

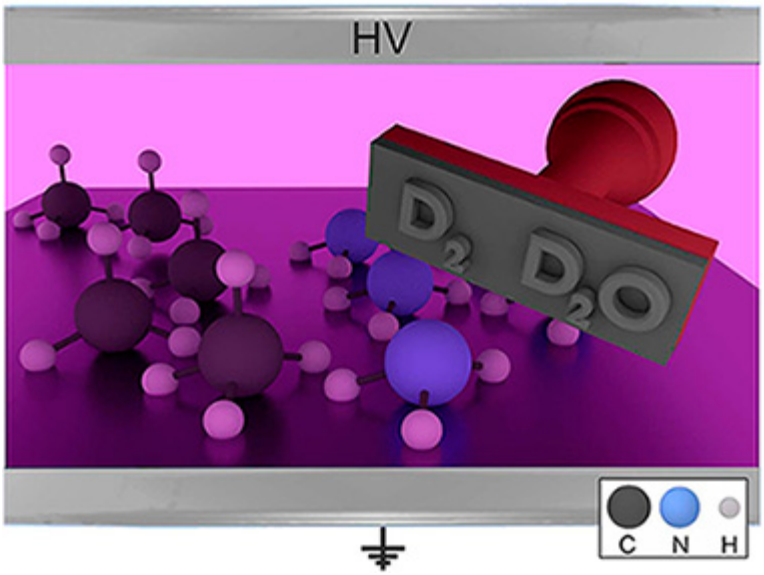
Isotope Labelling for Reaction Mechanism Analysis in DBD Plasma Processes

Navascues, P, Obrero-Perez, JM, Cotrino, J, Gonzalez-Elipe, AR, Gomez-Ramirez, A

Catalysts, **91** (2019) 45
Enero, 2019 | **DOI:** 10.3390/catal9010045

Dielectric barrier discharge (DBD) plasmas and plasma catalysis are becoming an alternative procedure to activate various gas phase reactions. A low-temperature and normal operating pressure are the main advantages of these processes, but a limited energy efficiency and little selectivity control hinder their practical implementation. In this work, we propose the use of isotope labelling to retrieve information about the intermediate reactions that may intervene during the DBD processes contributing to a decrease in their energy efficiency. The results are shown for the wet reforming reaction of methane, using D2O instead of H2O as reactant, and for the ammonia synthesis, using NH3/D-2/N-2 mixtures. In the two cases, it was found that a

significant amount of outlet gas molecules, either reactants or products, have deuterium in their structure (e.g., HD for hydrogen, CDxHy for methane, or NDxHy for ammonia). From the analysis of the evolution of the labelled molecules as a function of power, useful information has been



obtained about the exchange events of H by D atoms (or vice versa) between the plasma intermediate species. An evaluation of the number of these events revealed a significant progression with the plasma power, a tendency that is recognized to be detrimental for the energy efficiency of reactant to product transformation. The labelling technique is proposed as a useful approach for the analysis of plasma reaction mechanisms.

Laser-induced coloration of ceramic tiles covered with magnetron sputtered precursor layers

Rico, VJ, Lahoz, R, Rey-Garcia, F, de Francisco, I, Gil-Rostra, J, Espinos, JP, de la Fuente, GF, Gonzalez-Elipe, AR

Journal of the American Ceramic Society, **102** (2019) 1589-1598

Abril, 2019 | **DOI:** 10.1111/jace.16022

This paper reports a new methodology for the coloring of glazed ceramic tiles consisting of the near infrared pulsed laser processing of copper containing oxide coatings prepared by magnetron sputtering. As a second approach, the employ for the same purpose of a novel laser furnace technique is also described. Changing the laser parameters and using the laser furnace to treat the tiles at high temperature during irradiation has resulted in a wide color palette. The optical characterization of the modified tiles by UV-Vis spectroscopy has been complemented with their microstructural and compositional analysis by Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and Time Of Flight Secondary Ion Mass Spectrometry (TOF-SIMS). The chemical composition of the surface was obtained by X-ray Photoemission Spectroscopy (XPS) and its structure determined by X?ray diffraction (XRD). The chemical resistance was characterized by several tests following the norm ISO 10545-13. Color changes

have been attributed to surface microstructural and chemical transformations that have been accounted for by simple models involving different ablation, melting, diffusion, and segregation/agglomeration phenomena depending on the laser treatments employed.

Holmium doped fiber thermal sensing based on an optofluidic Fabry-Perot microresonator

Lahoz, F, Martin, IR, Soler-Carracedo, K, Caceres, JM, Gil-Rostra, J, Yubero, F
Journal of Luminescence, **206** (2019) 492-497
Febrero, 2019 | **DOI:** 10.1016/j.jlumin.2018.10.103

An optical temperature sensor suitable for label free liquid sensing has been designed and characterized. The sensor combines the photochemical stability of rare earth doped glasses and the high sensitivity of interferometric resonators. It is formed by a planar Eabry-Perot (FP) microcavity filled with the liquid to be monitored. A Ho3+ doped tapered optical fiber has been placed inside the microcavity surrounded by the fluid medium. An external laser is focused on the optical fiber inside the cavity to induce the luminescence of the Ho3+ ions, which couples to the FP optical resonances. The spectral position of the FP resonances is highly sensitive to the refractive index of the cavity medium. A second laser is co-aligned with the first one to locally heat the liquid medium around the optical fiber. An average blue shift of the FP resonances around 32 pm/degrees C is measured. The limit of detection of the laser induced heating of the liquid medium is about 0.3 degrees C in the biological temperature range. Alternatively, a hot-plate is used to heat the system. Interestingly, a red shift of the FP modes is observed with 75 pm/degrees C dependence and 0.12 degrees C limit of detection features.

Large gap atmospheric pressure barrier discharges using ferroelectric materials

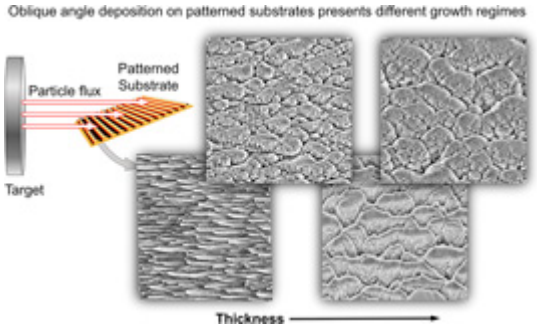
Navascues, P., Gonzalez-Elipe, A. R., Cotrino, J., Gomez-Ramirez, A.
Plasma Sources Sciences & Tecnology, **28** (2019) 075002
Julio, 2019 | **DOI:** 10.1088/1361-6595/ab28ce

This work reports a phenomenological comparative study of atmospheric pressure barrier plasmas using ferroelectric (ferroelectric barrier discharge (FBD)) and dielectric (dielectric barrier discharge (DBD)) plates to moderate the discharge. For FBD operation and large inter-electrode distances, experiments with helium carried out in a parallel plate reactor as a function of applied voltage have shown an enhancement of one order of magnitude in the charge transferred through the circuit. In a similar way to DBDs, FBDs rendered a laterally localized arrangement of discrete columnar discharges with a pattern distribution and an overall current intensity that depended on operation conditions. However, unlike the regular columnar pattern found for DBD operation, discharge columns in the FBD mode appear randomly and inhomogeneously distributed on the ferroelectric surface. This geometrical behavior of FBD plasma columns, as well as the singular variation of current with applied voltage and the particular shape characteristics of the current discharge curves have been accounted for by the high capacity of ferroelectric surfaces to randomly accumulate charge and to promote the emission of secondary electrons in the presence of a plasma.

Growth of nanocolumnar thin films on patterned substrates at oblique angles

Garcia-Valenzuela, A, Munoz-Pina, S, Alcala, G, Alvarez, R, Lacroix, B, Santos, AJ, Cuevas-Maraver, J, Rico, V, Gago, R, Vazquez, L, Cotrino, J, Gonzalez-Elipe, AR, Palmero, A
Plasma Processes and Polymers, **16** (2019) e1800135
Febrero, 2019 | **DOI:** 10.1002/ppap.201800135

The influence of one dimensional substrate patterns on the nanocolumnar growth of thin films deposited by magnetron sputtering at oblique angles is theoretically and experimentally studied. A well-established growth model has been used to study the interplay between the



substrate topography and the thin film morphology. A critical thickness has been defined, below which the columnar growth is modulated by the substrate topography, while for thicknesses above, the impact of substrate features is progressively lost in two stages; first columns grown on taller features take over neighboring ones, and later the film morphology evolves independently of substrate features. These results have been experimentally tested by analyzing the nanocolumnar growth of SiO2 thin films on ion-induced patterned substrates.

Higher hydration performance and bioactive response of the new endodontic bioactive cement MTA HP repair compared with ProRoot MTA white and NeoMTA plus

Jimenez-Sanchez, Maria Del Carmen, Segura-Egea, Juan Jose, Diaz-Cuenca, Aranzazu
Journal of biomedical materials research. Part B, Applied biomaterials, **107** (2019) 2109-2120
Agosto, 2019 | **DOI:** 10.1002/jbm.b.34304

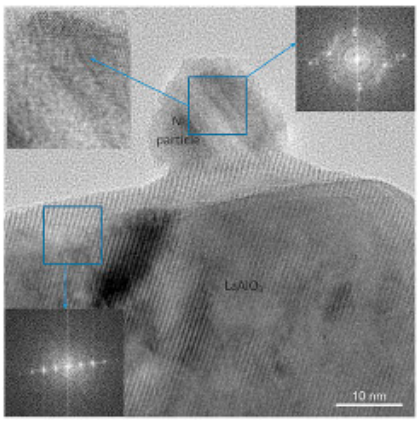
The aim of this study was to characterize the hydration performance and the bioactive response of the new bioactive endodontic cement MTA HP repair (HP), comparing its physicochemical parameters with those of ProRoot MTA White (Pro) and NeoMTA Plus (Neo). Un-hydrated precursor materials were characterized by X-ray fluorescence, laser diffraction, N2 physisorption and field emission gun scanning electron microscopy (FEG-SEM). Setting time was assessed according to ASTM specification C 266. Hydrated materials were analyzed by X-ray diffraction, Fourier transform infrared spectroscopy (FT-IR) and (FEG-SEM). Bioactivity evaluation in vitro was carried out, by soaking processed cement disk in simulated body fluid (SBF) during 168 h. The cements surface was studied by FT-IR, FEG-SEM, and energy dispersive X-ray. Release to the SBF media of ionic degradation products was monitored using inductively coupled plasma atomic emission spectroscopy. HP showed shorter initial setting time compared to Pro and Neo and produce a quick and effective bioactive response in vitro in terms of phosphate phase surface coating formation. This higher bioactive response for HP is correlated with increasing

calcium aluminate content, increasing surface area of un-hydrated powder precursor and the increasing release capacity of Si ionic products of the final hydrated product. The higher bioactive response of MTA HP repair highlights this material, as very interesting to further investigate its performance to improve the outcome of vital pulp therapy procedures.

Surface nickel particles generated by exsolution from a perovskite structure

Aguero, FN, Beltran, AM, Fernandez, MA, Cadus, LE
Journal of Solid State Chemistry, **273** (2019) 75-80
Mayo, 2019 | **DOI:** 10.1016/j.jssc.2019.02.036

LaAl1-xNixO3 (with x = 0.05 and 0.2) perovskite oxides were successfully synthesized and its behavior under reduction atmosphere was studied. HRTEM and STEM studies, coupled to HAADF and EDX detection, allowed to evidence the Ni exsolution process to the surface of the



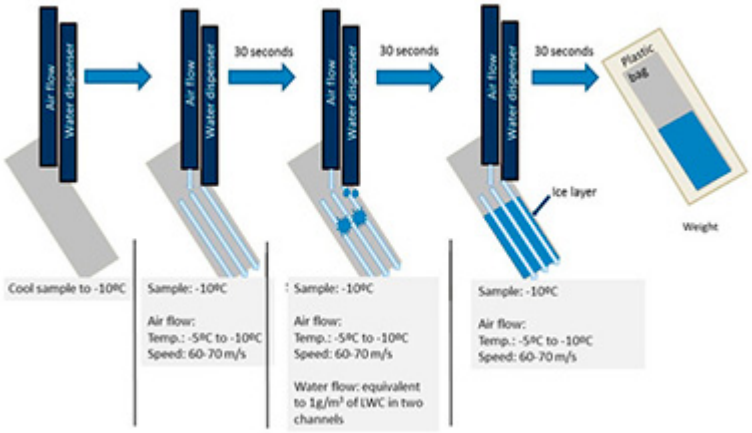
solid and to build nano-catalytic centers. The size of these centers is independent of the reduction conditions in the range studied. The high specific surface of the raw material, its porosity and the structure defects could be responsible of the low temperature at which the exsolution process starts. The content of Ni dopants allows the control of Ni centers size on the surface and the synthesis method provides Ni-nanoparticles strongly anchored to the resultant support.

Hydrophobic and Icephobic Behaviour of Polyurethane-Based Nanocomposite Coatings

Przybyszewski, B, Boczkowska, A, Kozera, R, Mora, J, Garcia, P, Aguero, A, Borras, A
Coatings, **9** (2019) 811
Diciembre, 2019 | **DOI:** 10.3390/coatings9120811

In this paper, hydrophobic nanocomposite coatings based on polyurethane (PUR) modified by nano-silica and silane-based compounds were manufactured by spraying. The main challenge was to assess and improve the hydrophobic as well as anti-icing properties of initially hydrophilic polymer coatings. The prepared nanocomposite coatings were characterized by means of scanning electron microscopy (SEM), optical profilometry and X-ray photoelectron spectroscopy (XPS). The results obtained showed that in order to achieve hydrophobicity, appropriate amounts of nano-silica must be incorporated in the coating, and complete coverage by nano-

silica particles is necessary for achieving hydrophobicity. Coating adhesion and the durability of the hydrophobic behaviour were also studied by scratch test and frosting/defrosting cycles, respectively. The results show that use of both nano-silica and silane-based compounds improve



the hydrophobic and anti-icing properties of the coating as compared to a non-modified PUR topcoat. A synergistic effect of both additives was observed. It was also found that the anti-icing behaviour does not necessarily correlate with surface roughness and the materials' wetting properties.

Comparative studies on electrochemical energy storage of NiFe-S nanoflake and NiFe-OH towards aqueous supercapacitor

Naseri, M, Moradi, M, Hajati, S, Espinos, JP, Kiani, MA
Journal of Materials Science-Materials in Electronics, **30** (2019) 4499-4510
Mayo, 2019 | **DOI:** 10.1007/s10854-019-00738-x

In this study, electrochemical energy storage performances of an efficient Ni-Fe sulfide and hydroxide supported on porous nickel foam are compared. X-ray diffraction (XRD), X-rayphotoelectron spectroscopy (XPS) and energy-dispersive X-ray spectrometer (EDS) results confirmed the formation of Ni-Fe-S and Ni-Fe-OH electrodes. In addition, Brunauer-Emmett Teller (BET) was used to determine the specific surface area of the prepared materials. Moreover, the morphologies were observed by scanning electron microscopy (SEM). The brilliant characteristics of Ni-Fe-S could be attributed to transport acceleration in electrolyte ions and electrons, occurrence of redox reactions as well as the higher conductivity of the sample. From stand point of comparison, the capacitance of Ni-Fe-S is more than that of Ni-Fe-OH. Therefore, the exchange of O2- with S2- in Ni-Fe-OH lattice obviously improves the electrochemical performance. The as-fabricated Ni-Fe sulfide electrode exhibits a tremendous specific capacitance of 884.9Fg(-1) at 1A g(-1). Furthermore, an assembled asymmetric supercapacitor device using the activated carbon as negative electrode and this smart configuration (Ni-Fe-S) as positive electrode also provided a maximum specific power and specific energy of 8000Wkg(-1), 37.9 Whkg(-1), respectively. Also, it shows cycling stability with 88.8% capacitance retention after 1700 cycles in aqueous electrolyte, demonstrating its potential application in the next-generation high-performance supercapacitors used for energy storage.

An innovative approach for micro/nano structuring plasma polymer films

Thiry, D, Vinx, N, Aparicio, FJ, Moerman, D, Lazzaroni, R, Cossement, D, Snyders, R
Thin Solid Films, **672** (2019) 26-32
Febrero, 2019 | **DOI:** 10.1016/j.tsf.2018.12.050

This work aims at presenting an innovative method for tailoring the morphology of functionalized plasma polymer films (PPF). The approach is based on the formation of a plasma polymer bilayer system in which the two layers differ by their chemical composition and cross-linking degree. As a case study, propanethiol-based plasma polymer films have been investigated. As revealed by a much higher S/C ratio than in the propanethiol precursor (i.e. 0.83 vs 0.33), it has been demonstrated that the bottom layer contains a large fraction of trapped sulfur-based molecules (e.g. H2S). When further covered by a denser PPF formed at higher energetic conditions, a three-dimensional morphological reorganization takes place giving rise to the micro/nano structuration of the organic material. The shape, the dimensions as well as the density of the generated structures are found to depend on the thickness of both coatings involved in the bilayer structure, offering a great flexibility for surface engineering. Annealing experiments unambiguously confirm the major role played by the sulfur-based trapped molecules for inducing the reshaping process. The whole set of data clearly paves the way for the development of an innovative approach for finely tailoring the morphology of functionalized PPF at the micro/nano scale.

XPS primary excitation spectra of Zn 2p, Fe 2p, and Ce 3d from ZnO, α-Fe_2O_3, and CeO_2

Pauly, N., Yubero, F., Espinós, J.P., Tougaard, S.
Surface and Interface Analysis, **51** (2019) 353-360
Marzo, 2019 | **DOI:** 10.1002/sia.6587

Metal oxides are important for current development in nanotechnology. X-ray photoelectron spectroscopy(XPS) is a widely used technique to study the oxidation states of metals, and a basic understanding of the photoexcitation process is important to obtain the full information from XPS. We have studied core level excitations of Zn 2p, Fe 2p, and Ce 3d photoelectron emissions from ZnO, α-Fe_2O_3, and CeO_2. Using an effective energy-differential XPS inelastic-scattering cross section evaluated within the semiclassical dielectric response model for XPS, we analysed the experimental spectra to determine the corresponding primary excitation spectra, ie, the initial excitation processes. We find that simple emission (Zn 2p) as well as complex multiplet photoemission spectra (Fe 2p and Ce 3d) can be quantitatively analysed with our procedure. Moreover, for α-Fe_2O_3, it is possible to use the software package CTM4XAS (Charge Transfer Multiplet program for X-ray Absorption Spectroscopy) to calculate its primary excitation spectrum within a quantum mechanical model, and it was found to be in good agreement with the spectrum determined by analysis of the experiment.

MTA HP Repair stimulates in vitro an homogeneous calcium phosphate phase coating deposition

Jiménez-Sánchez, M.D.C., Segura-Egea, J.J., Díaz-Cuenca, A.
Journal of Clinical and Experimental Dentistry, **11** (2019) e322-e326
Agosto, 2019 | **DOI:** 10.4317/jced.55661

Background: To study the mineralization capacity in vitro of the bioceramic endodontic material MTA HP Repair. Material and Methods: Bioactivity evaluation in vitro was carried out, by soaking processed cement disk in simulated body fluid (SBF) during 168 h. The cement surface was studied by Fourier transform infrared spectroscopy (FTIR), field emission gun scanning electron microscopy (FEG-SEM) and energy dispersive X-ray analysis (EDX). Release to the SBF media of ionic degradation products was monitored using inductively coupled plasma atomic emission spectroscopy (ICP-AES). Results: FT-IR showed increasing formation of phosphate phase bands at 1097, 960, 607 and 570 cm -1 with prolonged SBF soaking. FEG-SEM analysis reveals that HP produces a effectively surface covering consisting in homogeneous spherical phosphate phase aggregates with an average diameter of 0.5 -1 .0 µm. EDX analysis comparing un-treated (hydrated), 24 h and 72 h SBF treated surfaces of MTA HP Repair revealed phosphate deposition after 24 h, with high phosphorous/silicon element ratio signal measured after 24 h, indicating a very high phosphate phase deposition for this material. Conclusions: The study shows that MTA HP Repair produces a quick and effective bioactive response in vitro in terms of crystalline calcium phosphate surface coating formation. The high bioactive response of MTA HP Repair makes it an interesting candidate for endodontic use as repair cement.

Physicochemical parameters - hydration performance relationship of the new endodontic cement MTA Repair HP

Jiménez-Sánchez, M.D.C., Segura-Egea, J.J., Díaz-Cuenca, A.
Journal of Clinical and Experimental Dentistry, **11** (2019) e739-e744
Agosto, 2019 | **DOI:** 10.4317/jced.56013

Background: To characterize the chemical composition and textural parameters of the MTA Repair HP precursor powder and their influence to hydration performance. Material and Methods: Un-hydrated precursor material was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), X-ray fluorescence (XRF), laser diffraction (LD), N2 physisorption and field emission gun scanning electron microscopy (FEG-SEM). Setting time was assessed according to ASTM specification C 266. Hydrated material was analysed by XRD, FT-IR, energy dispersive X-ray (EDX) analysis and FEG-SEM. Results: Ca3SiO5 and Ca2SiO4, in addition to CaWO4 as radiopacifier are the main compositional phases. Other measured parameters indicate high specific surface area of 4.8 m2 g-1, high aluminium content of 1.7 wt.% and low initial and final setting times of 12 and 199 min, respectively. Singular microstructural features consisting of high aspect ratio nanoparticles are main constituents of un-hydrated precursor. Besides, FEM-SEM observation shows notably growth of hexagonal shaped plate-like morphologies homogeneously distributed along the sample during hydration process. Conclusions: The short setting time measured for HP Repair, is correlated with high surface area of precursor powder, high Al content and the absence of compositional sulphate phases.

Test of a He-3 target for transfer reactions in inverse kinematics

Carozzi, G, Valiente-Dobon, JJ, Gadea, A, Siciliano, M, Mengoni, D, Fernandez, A, Godinho, V, Hufschmidt, D, Di Nitto, A
Nuovo cimento c-colloq uia and communications in physics, **42** (2019) 94
Marzo, 2019 | **DOI:** 10.1393/ncc/i2019-19094-9

With the aim of studying exotic nuclei close to the proton dripline, an innovative He-3 target was produced and tested in a collaboration between the Materials Science Institute of Seville (Spain) and the Legnaro National Laboratories (Italy). The target was manufactured with a new technique that aims to reduce the costs while providing high quality targets. The target was tested at the Legnaro National Laboratories. The results of this test are presented in this contribution.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

Roughness of deposited carbon-based coatings and its statistical characteristics at nano and microscales
F.M. Borodich, E. Brousseau, A. Clarke, A. Pepelyshev, J.C Sanchez-Lopez
Front. Mech. Eng., **5** (2019) 24
Mayo, 2019 | **DOI:** 10.3389/fmech.2019.00024

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

IBERTRIVA 2019: Conference Proceedings
Editors: J.A. Martín Gago y J.C. Sánchez López
Productor: Instituto de Ciencia de Materiales de Sevilla (ICMS)
Designed by: Elena López Guijarro
Published by: IBERTRIVA2019
Printed by: Gráficas Mafra, S.L.
ISBN: 978-84-09-12421-3
Número de Páginas: 258

Relationship between the scratch and wear behavior of 3Y-TZP reinforced with graphene nanoplatelets
J.A. Santiago, I. Fernández-Martínez, A. Wennberg, J.M. Molina-Aldareguia, M.A. Monclús, V. Bellido-González, R. González-Arrabal, T.C. Rojas, J.C. Sánchez-López
"IBERTRIVA 2019 Conference Proceedings", (2019) Editorial IBERTRIVA2019, J.A. Martín Gago y J.C. Sánchez López, págs. 198
ISBN: 978-84-09-12421-3

Unraveling the nanostructure of nanoporous films fabricated by magnetron sputtering using Helium as process gas
A. Fernandez, B. Lacroix, V. Godinho, F.J. Ferrer, M.C. Jiménez de Haro
"IBERTRIVA 2019 Conference Proceedings", (2019) Editorial IBERTRIVA2019, J.A. Martín Gago y J.C. Sánchez López, págs. 135-136
ISBN: 978-84-09-12421-3

Study of TiC/a-C(:H) Coatings before and after Friction by Nanoindentation
Elena Torskaya, Tamara Muravyeva, Juan Carlos Sánchez López
"Proceedings of the 4th International Conference on Industrial Engineering (ICIE 2018)", (2019) Editorial Springer, Eds. A.A Radionov, O.A. Kravchenko, V.I. Guzeev, Y.V. Rozhdestvenskiy, págs. 1011-1018
ISBN: 978-3-319-95629-9

Fracture of TiC/a-C(:H) Coatings in Friction Contact
Elena Torskaya, Alexey Mezrin, Juan Carlos Sánchez-López
"Proceedings of the 4th International Conference on Industrial Engineering (ICIE 2018)", (2019) Editorial Springer, Eds. A.A Radionov, O.A. Kravchenko, V.I. Guzeev, Y.V. Rozhdestvenskiy, págs. 1003-1010
ISBN: 978-3-319-95629-9

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

EMRS Spring meeting 2019
27 mayo [Francia]

Understanding the high-temperature oxidation behavior of CrAlYN-based protective coatings
J.C. Sánchez
Conferencia Invitada

X Iberian Conference on Tribology / XI Iberian Vacuum Conference | IBERTRIVA 2019
26 – 28 junio [Sevilla, España]

Unraveling the nanostructure of nanoporous films fabricated by magnetron sputtering using Helium as process gas
A. Fernandez, B. Lacroix, V. Godinho, F.J. Ferrer, M. C. Jiménez de Haro
Comunicación oral

Hard Cr-doped DLC coatings deposited by low-frequency HiPIMS with enhanced tribomechanical behavior at high temperature.
J.A. Santiago, I. Fernández-Martínez, A. Wennberg, J.M. Molina-Aldareguia, M.A. Monclús, V. Bellido-González, R. González-Arrabal, T.C. Rojas, J.C. Sánchez-López
Poster

V Simposio Nacional de Ingeniería de Superficies y Tribología (V-SNISyT)
9 – 11 octubre [Mexico]

Control de la nanoestructuración en capas lubricantes de calcogenuros metálicos para aplicaciones en vacío y aeroespaciales
J.C. Sánchez-López, S. Dominguez-Meister, T.C. Rojas
Conferencia Invitada

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

40 Congreso Nacional de Endodoncia
31 octubre – 2 noviembre [Valencia, España]

Biomateriales Compuestos de Biopolímero y Vidrio BioactivoMesoporoso Funcionalizados con Zinc para Aplicación en Endodoncia Regenerativa
MC Jiménez-Sánchez, F Lundy, A Díaz-Cuenca, JJ Segura-Egea
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Recubrimientos multicapa de Cr-Al-N-O como absorbedores solares selectivos usando la técnica de HiPIMS**
Autor:	Jaume Carreras Buscató
Directores:	Juan Carlos Sánchez López
Calificación:	Sobresaliente “Cum Laude”
Centro:	Universidad de Sevilla
Fecha:	17 de julio de 2019

Título:	**Control Nanoestructural en Capas y Multicapas Porosas mediante Pulverización Catódica: Procesado y Aplicaciones**
Autor:	Aurelio García Valenzuela
Directores:	Agustín Rodríguez González-Elipe y Alberto Palmero Acebedo
Calificación:	Sobresaliente "Cum Laude"
Centro:	Universidad de Sevilla
Fecha:	19 de julio de 2019

Título:	**Materiales compuestos de vidrio bioactivo mesoporoso funcionalizado con zinc para aplicación en endodoncia regenerativa**
Autor:	María del Carmen Jiménez Sánchez
Directores:	María Aránzazu Díaz Cuenca
Calificación:	Sobresaliente "Cum Laude"
Centro:	Universidad de Sevilla
Fecha:	18 de noviembre de 2019

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Estudio de la reacción de descomposición catalítica del ácido fórmico como medio de producción de hidrógeno**
Autor:	Adrián Fernández Vega
Directores:	Gisela Mariana Arzac Di Tomaso y Ana María Beltrán Custodio
Grado:	Trabajo Fin de Grado
Año Académico:	2018-2019 (10 de enero 2019)

Título:	**Recubrimientos anti-rayadura para lentes oftálmicas**
Autor:	Diego Bermúdez Domínguez
Directores:	Victor Lopez Flores y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (4 julio 2019)

Título:	**Nanopartículas magnéticas para hipertermia en tratamientos contra el cáncer**
Autor:	Cristina Suárez Rufo
Directores:	Victor Lopez Flores y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2018-2019 (4 julio 2019)

Título:	**Synthesis and characterization of three-dimensional ITO nanoelectrodes**
Autor:	Javier Castillo Seoane

Directores: Ana Isabel Borras Martos, Leidy Marcela Martínez Tejada y Jorge Gil Rostra
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (9 julio 2019)

Título: **Estructuras fotónicas multicapa para la detección selectiva de compuestos orgánicos volátiles**
Autor: Hermine Berthon
Directores: Ana Isabel Borras Martos, Leidy Marcela Martínez Tejada y Jorge Gil Rostra
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (9 julio 2019)

Título: **Láminas delgadas nanoestructuradas mediante aplicación de ondas acústicas superficiales**
Autor: José Andrés Espino Román
Directores: Anna Dimitrova Penkova y Agustín R. González-Elipe
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2018-2019 (9 julio 2019)

Título: **Recubrimientos multicapa de Cr-Al-N-O como absorbedores solares selectivos usando la técnica de HiPIMS**
Autor: Jaume Carreras Buscató
Directores: Juan Carlos Sánchez López, Marco A. Pérez Martínez, Manuel D. Abad Roldán
Grado: Trabajo Fin de Master
Centro: Universitat Ramón Llull (Barcelona)
Año Académico: 2018-2019 (17 julio 2019)

Título: **Rediseño de una cámara de vacío destinada al crecimiento de películas delgadas por pulverización catódica: modelado y optimización**
Autor: María del Alba Vivas Sánchez
Directores: María Asunción Fernández Camacho
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (17 de septiembre 2019)

Título: **Modificación superficial de recubrimientos nanoestructurados de Pd-C por técnicas de plasma**
Autor: Aida Laureano Niñez
Directores: Vanda Cristina Fortio Godinho
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (25 de septiembre 2019)

DOCENCIA / TEACHING

Máster Oficial y Doctorado en Biotecnología Avanzada
Nanotecnología
María Aránzazu Díaz Cuenca
Lugar: Universidad Internacional de Andalucía. Universidad de Málaga

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

Instituto de Investigaciones en Materiales. Universidad Nacional Autónoma de México
México **Juan Carlos Sánchez López** 7/10/2019 al 8/10/2019

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS
PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Universitá Degli Studi di Salerno
Salerno, Italia **Luigi Panzone** 24/09/2018 al 10/02/2019

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de Magnetron Sputttering 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)
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- Bomba híbrida de vacío resistente a químicos VACUUBRAND RC-6
- Software y sondas para monitorización para equipo de liofilización CHRIST EPSILON 2-4 LSC
- 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)

- Malvern, Modelo Zetasizer Nano: para determinar mediante "Dynamic light scattering" la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos 1-3000 nanómetros.
  Este equipo también es adecuado para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico) mediante medidas de Movilidad Electroforética.
- 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 extención 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 ºC (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).

<u>Laboratorio de síntesis y catálisis</u>

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 heterogenea solido-gas.

Reactores catalíticos para catálisis heterogenea 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.
Buretas automáticas para medición de gases.

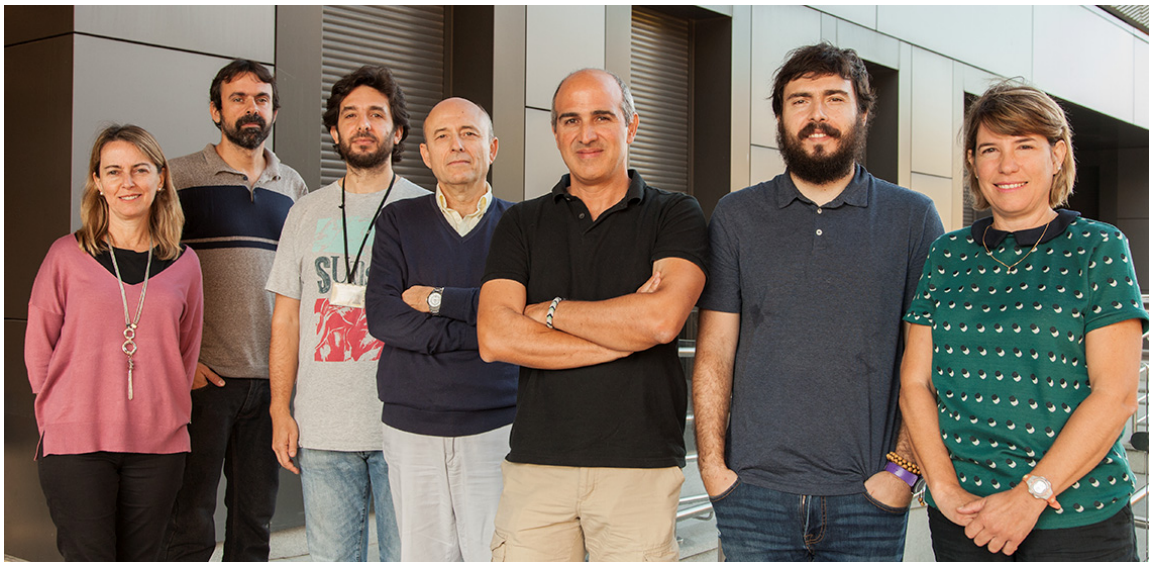
Cámaras de deposición de recubrimientos y películas delgadas y tratamientos con plasma.
Sistema de tratamiento con plasmas Diener.
Tres cámaras de deposición por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación total de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables, calentables y "biased".
Una cámara de deposición por la técnica de pulverización catódica con fuente HIPIMS.

Equipamiento para microscopía electrónica
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.

Propiedades mecánicas de superficies y recubrimientos
Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.
Tribómetro de alta temperatura (hasta 800ºC) –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.

Propiedades eléctricas de materiales
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.

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 O. Núñez Álvarez

Investigadores Distinguidos

Dr. Juan F. Galisteo López

Investigadores Contratados

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Lda. Victoria Esteso Carrizo (hasta octubre)
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PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Materiales ópticos basados en nanofósforos para la próxima generación de dispositivos emisores de luz Nanophosphor-based photonic materials for next generation light-emitting devices NANOPHOM

Código/Code:	H2020-ERC-STG/0259
Periodo/Period:	1-04-2017 / 31-03-2022
Organismo Financiador/Financial source:	European Commission STARTING GRANT
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.

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

Código/Code:	RTI2018-094426-B-I00
Periodo/Period:	01-01-2019 / 31-12-20201
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Manuel Ocaña Jurado y Ana Isabel Becerro Nieto
Componentes/Research group:	Nuria O. Nuñez Alvarez

RESUMEN / ABSTRACT

El proyecto persigue la preparación de nanoparticulas (NPs) multifuncionales con propiedades mejoradas y caracteristicas (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 clinico 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 (Nd3+ o Er3+fYb3+ o Tm3+fYb3+), 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 ZnGa204:Cr3+ y Y3AI2Ga3012:Ce3+,Cr3+,Nd3+, para los que es prioritario desarrollar nuevos métodos de sintesis 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 asi obtener un diagnóslico 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 asi 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 sintesis de NPs inorgánicas basadas en tierras raras y dispone de la mayoria 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 (Nd3+ or Er3+lYb3+ or Tm3+lYb3+), 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 pro bes 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 ZnGa204:Cr3+ and Y3AI2Ga3012:Ce3+,Cr3+,Nd3+, 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 Advanced optical materials for more efficient optoelectronic devices

Código/Code:	MAT2017-88584-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Juan F. Galisteo López, Mauricio E. Calvo Roggiani, Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El proyecto MODO busca optimizar el diseño óptico de dispositivos optoelectrónicos, ya sean fotovoltáicos 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 principial 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 fotovoltáicas. Por otro lado, presentan elevados rendimientos cuánticos de fotoemisión en el verde y el rojo, lo que también las convierte en candidadatas 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 presente ahondar en la compresión de los fenómenos que intervienen en la degradación fotoinducida de estos materials 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 fotovoltáica, propuesta muy recientemente, basada en nanocristales semiconductores de AgBiS2, de muy bajo impacto ambiental pero con una absorción óptica aún muy inferior al resto de celdas 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 estodos excitados a escala macroscópica y la de observar de 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 incluídas 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
Verification of the existence of macroscale repulsive Casimir forces in suspended self-standing films

Código/Code:	FIS2017-91018-EXP
Periodo/Period:	01-11-2018 / 31-10-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	36.300 €
Investigador responsable/Research head:	Hernán 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 centraá 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, readicalmente novedosa, hace uso de técnicas de espectroscoìa 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 tefón, 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 macroscópicas de fuerzas repulsivas de Casimir-Lifshitz, nunca reportada anteriormente, mediante medidas de espectroscopía óptica constituiria 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 levation phenomena can be observed and characterized due to the balance between the latter and gravity force. This radically new approcah 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 glycorel 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 selft-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.

Adquisición de un equipo de medida de espectroscopía ultrarrápida de estados transitorios para el servicio de espectroscopías del ICMS

Código/Code: EQC2018-004413-P
Periodo/Period: 01-01-2018 / 31-12-2020
Organismo Financiador/Financial source: Ministerio de Ciencia e Innovación
Importe total/Total amount: 441.843,34 €
Investigador responsable/Research head: Hernán Míguez García

RESUMEN / ABSTRACT

La técnica que pretende implementarse, la espectroscopía ultrarrápida de absorción de estados transitorios, es actualmente una de las más potentes para estudiar los procesos de excitación de materiales y compuestos empleados en numerosos campos tales como energía fotovoltaica, iluminación, fotosíntesis o catálisis, lo que explica la diversidad temática de los grupos que apoyan esta propuesta. Pese a su sofisticación, es una técnica ya imprescindible para la mayor parte de grupos de relevancia internacional en los ámbitos mencionados. Andalucía, aunque cuenta con grupos de gran reconocimiento en esos campos, no dispone de un laboratorio de referencia en este tipo de caracterización que dé servicio a la comunidad científica relacionada, lo que supone una clara desventaja competitiva con respecto a otros países. La adquisición de este equipo a través del Servicio General de Espectroscopías del ICMS, permitirá solucionar esta desventaja. El ICMS, a través de su Unidad Estructural de Materiales Ópticos, cuenta con personal permanente recientemente incorporado al centro altamente cualificado y con experiencia en este tipo de caracterización, adquirida en estancias postdoctorales en centros nacionales y extranjeros de gran prestigio.

Ultrafast absorption spectroscopy of transient states is nowadays one of the most powerful ones to analyze excitation processes in materials and compounds used in numerous fields such as photovoltaics, lighting, photosynthesis or catalysis, which explains the great diversity of groups supporting this application. In spite of its sophistication, it is already an irreplaceable technique for most groups of international relevance in these fields. Andalucía, although it counts with renowned groups in these areas, does not have an open laboratory that can provide support to the related scientific community, which implies a competitive disadvantage with respect to other countries. The acquisition of this equipment through the General Spectroscopy Service of the ICMS will help to solve this situation. The ICMS, through its Department of Optical Materials, counts with recently incorporated permanent scientific staff skilled and trained in this type of characterization, attained during postdoctoral stays in national and international centers of great prestige.

Sistema de caracterización óptica avanzada en atmósfera inerte

Código/Code:	EQC2019-005556-P
Periodo/Period:	01-01-2019 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	121.917,11 €
Investigador responsable/Research head:	Juan F. Galisteo López

RESUMEN / ABSTRACT

Esta solicitud pretende dotar al Servicio de Espectroscopías (SE) del Instituto de Ciencia de Materiales de Sevilla (ICMS) de un equipo para realizar una caracterización óptica avanzada in-situ de materiales y dispositivos fabricados en atmósfera inerte. Este equipamiento, de carácter único en la Comunidad Autónoma Andaluza, permitirá monitorizar la preparación de materiales de gran relevancia en distintos ámbitos tales como la optoelectrónica de tercera generación o la catálisis basada en complejos órgano-metálicos. El amplio campo de aplicación de este equipamiento se refleja en el apoyo de grupos cuyo ámbito de trabajo es muy diverso y que pertenecen a distintos centros de investigación y universidades del entorno. El SE a cargo del equipo dispone de personal cualificado para su manejo y abrirá su uso a la comunidad científica interesada. Este equipamiento permitirá consolidar el SE del ICMS como referencia en el ámbito del estudio de las propiedades ópticas en Andalucía y como uno de los centros más avanzados de este tipo en España. La propuesta está avalada por numerosos proyectos de investigación tanto de ámbito estatal (proyectos MAT, CTQ, redes temáticas de ámbito nacional, etc) como europeo (proyectos ERC Starting Grant) en los que se requiere de una caracterización de este tipo. Al mismo tiempo, potenciará las ya intensas relaciones con los agentes productivos del entorno, materializada a través de numerosos contratos de investigación, desarrollo e innovación.

This application seeks to endow the Spectroscopy Facility (SF) of the Institute of Materials Science of Seville (ICMS) with a setup to carry out the in-situ advanced optical characterization of materials and devices prepared in an inert atmosphere. This equipment, unique in the Andalucía Autonomous Community, will allow monitoring the preparation of highly relevant materials for different applications such as third-generation optoelectronics or organo-metallic complexes for catalysis. The wide application scope of this equipment is evidenced by the support shown by groups whose research topics are very different and that belong to different research centers and universities of the environment. The Spectroscopy Service is managed by qualified staff for its operation, and will provide open access to the interested scientific community regardless their affiliation. This infrastructure will help to consolidate the SF of the ICMS as a reference in the analysis of optical properties of materials in Andalucia and as one of the most advanced ones in Spain. This grant proposal is endorsed by the scientific and technical research activity carried out in numerous projects both at national (projects MAT, CTQ, national thematic networks, etc) and international level (ERC Starting Grant) in which a characterization of the sort herein pursued is required. At the same time, this infrastructure will boost and reinforce relations with industry and companies with which the groups supporting .

OTROS PROYECTOS / OTHER PROJECTS

Fotofísica de perovskitas híbridas de metal-haluro

Código/Code:	201860I068
Periodo/Period:	22-11-2018 / 21-11-2019
Organismo Financiador/Financial source:	CSIC
Importe total/Total amount:	5.000 €
Investigador responsable/Research head:	Juan F. Galisteo López

Arquitecturas fotónicas para fuentes de luz más versatiles

Código/Code:	201860I067
Periodo/Period:	22-11-2018 / 21-11-2019
Organismo Financiador/Financial source:	CSIC
Importe total/Total amount:	5.000 €
Investigador responsable/Research head:	Gabriel S. Lozano Barbero

Desarrollo de Dispositivos Emisores de Luz basados en Perovskita Nanoestructurada

Código/Code:	201960E090
Periodo/Period:	01-09-2019 / 31-08-2022
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	120.345 €
Investigador responsable/Research head:	Hernán Míguez García

Nanopartículas basadas en matrices inorgánicas de tierras raras para aplicaciones biotecnolóticas

Código/Code:	201560E056
Periodo/Period:	01-06-2015 / 01-05-2019
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	75.789 €
Investigador responsable/Research head:	Ana Isabel Becerro Nieto

Transparent Ceramic Phosphors

Código/Code:	PIC2016FR1
Periodo/Period:	01-01-2017/ 31-12-2019
Organismo Financiador/Financial source:	CSIC (PIC)
Importe total/Total amount:	10.000 €
Investigador responsable/Research head:	Ana Isabel Becerro Nieto

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

SOLAR BLUE
Periodo/Period: 01-01-2019 / 30-06-2020
Organismo Financiador/Financial source: CAPSUN TECHNOLOGIES, S.L.
Importe total/Total amount: 50.000 €
Investigador responsable/Research head: Hernán Míguez García

Evaluación de la aplicabilidad de diversas estructuras inorgánicas como matriz huésped en el desarrollo de pigmentos inorgánicos (PAINTINK
Periodo/Period: 01-03-2018 / 15-04-2019
Organismo Financiador/Financial source: AL-FARBEN, S.A.
Importe total/Total amount: 82.764 €
Investigador responsable/Research head: Manuel Ocaña Jurado

Recubrimientos ópticos para sistemas fotovoltaicos de concentración
Periodo/Period: 15-05-2014 / 26-04-2019
Organismo Financiador/Financial source: Abengoa Solar New Technologies, S.A.
Importe total/Total amount: 181.500 €
Investigador responsable/Research head: Hernán Míguez García

PATENTES / PATENTS

Efficient transparent white light emitting layered phosphor structure of tunable shade, process for obtaining said structure and uses
Inventores: Dongling Geng, Hernán R. Míguez García, Gabriel S. Lozano Barbero
Tipo de Patente: Internacional
Número de Solicitud: PCT/EP19/070218
Fecha Solicitud: 26 de julio de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas

ABX3 compounds with perovskite crystalline structure infiltrated within a porous metal oxide film
Inventores: Andrea Rubino, Mauricio E. Calvo Roggiani, Hernán R. Míguez García, Juan F. Galisteo López
Tipo de Patente: PCT/EP19/062850
Número de Solicitud: 18382341
Fecha Solicitud: 17 de mayo de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas y Universidad de Sevilla

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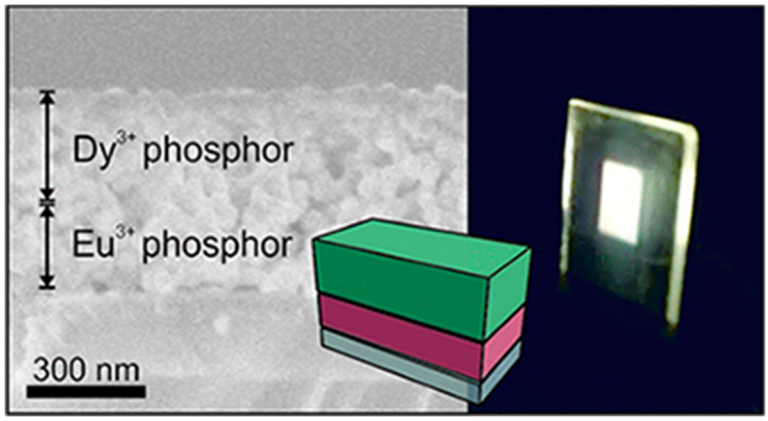
Inventores: Alberto Jiménez Solano, Hernán R. Míguez García
Tipo de Patente: Sotware
Número de Solicitud: 4755/2019
Fecha Solicitud: 11 de junio de 2019
Entidad Titular: Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Highly Efficient Transparent Nanophosphor Films for Tunable White-Light-Emitting Layered Coatings

Geng, DL, Lozano, G, Miguez, H
ACS Applied Materials & Interfaces, **11** (2019) 4219-4225
Enero, 2019 | **DOI:** 10.1021/acsami.8b17368

Bright luminescence in rare-earth (RE) nanocrystals, the so-called nanophosphors, is generally achieved by choosing a host that enables an effective excitation of the RE activator through charge or energy transfer. Although tungstate, molybdate, or vanadate compounds provide the aforementioned transfer, a comparative analysis of the efficiency of such emitters remains



elusive. Herein, we perform a combined structural and optical analysis, which reveals that the tetragonal GdVO4 matrix gives rise to the highest efficiency among the different transparent nanophosphor films compared. Then, we demonstrate that by a sequential stacking of optical quality layers made of Eu3+- and Dy3+-doped nanocrystals, it is possible to attain highly transparent white-light-emitting coatings of tunable shade with photoluminescence quantum yields above 35%. Layering provides a precise dynamic tuning of the chromaticity based on the photoexcitation wavelength dependence of the emission of the nanophosphor ensemble without altering the chemical composition of the emitters or degrading their efficiency. The total extinction of the incoming radiation along with the high quantum yields achieved makes these thin-layered phosphors one of the most efficient transparent white converter coatings ever developed.

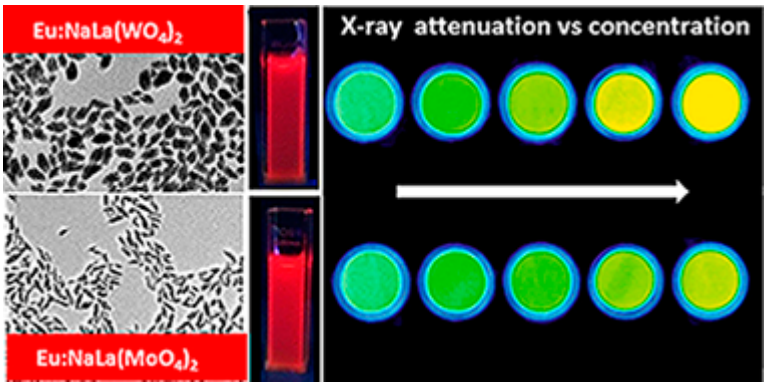
Synthesis, functionalization and properties of uniform europium-doped sodium lanthanum tungstate and molybdate (NaLa(XO4)(2), X = Mo,W) probes for luminescent and X-ray computed tomography bioimaging

Laguna, M, Nunez, NO, Becerro, AI, Lozano, G, Moros, M, de la Fuente, JM, Corral, A, Balcerzyk, M, Ocana, M

Journal of Colloid and Interface Science, **554** (2019) 520-530

Octubre, 2019 | **DOI:** 10.1016/j.jcis.2019.07.031

A one-pot simple procedure for the synthesis of uniform, ellipsoidal Eu3+-doped sodium lanthanum tungstate and molybdate (NaLa(XO4)(2), X = W, Mo) nanophosphors, functionalized



with carboxylate groups, is described. The method is based on a homogeneous precipitation process at 120 degrees C from appropriate Na+ Ln(3+) and tungstate or molybdate precursors dissolved in ethylene glycol/water mixtures containing poly acrylic acid. A comparative study of the luminescent properties of both luminescent materials as a function of the Eu3+ doping level has been performed to find the optimum nanophosphor, whose efficiency as X-ray computed tomography contrast agent is also evaluated and compared with that of a commercial probe. Finally, the cell viability and colloidal stability in physiological pH medium of the optimum samples have also been studied to assess their suitability for biomedical applications.

Flexible nanophosphor films doped with Mie resonators for enhanced out-coupling of the emission

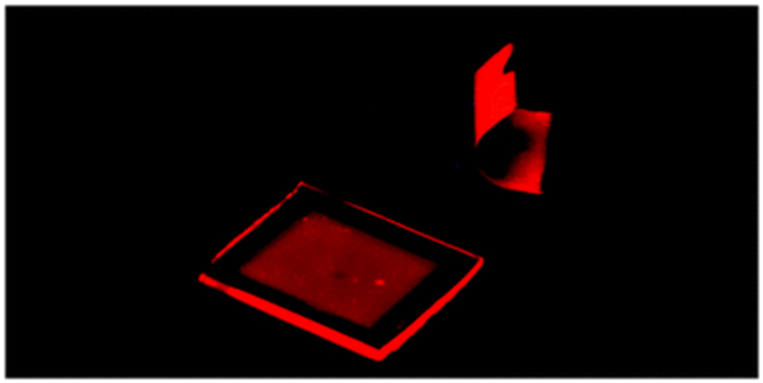
Miranda-Munoz, JM, Geng, DL, Calvo, ME, Lozano, G, Miguez, H

Journal of Materials Chemistry C, **7** (2019) 267-274

Enero, 2019 | **DOI:** 10.1039/C8TC05032E

Herein we present a combined study of the evolution of both the photoluminescence (PL) and the surface chemical structure of organic metal halide perovskites as the environmental oxygen pressure rises from ultrahigh vacuum up to a few thousandths of an atmosphere. Analyzing the changes occurring at the semiconductor surface upon photoexcitation under a controlled oxygen atmosphere in an X-ray photoelectron spectroscopy (XPS) chamber, we can rationalize the rich variety of photophysical phenomena observed and provide a plausible explanation for light-induced ion migration, one of the most conspicuous and debated concomitant effects detected during photoexcitation. We find direct evidence of the formation of a superficial layer of negatively charged oxygen species capable of repelling the halide anions away from the

surface and toward the bulk. The reported PL transient dynamics, the partial recovery of the

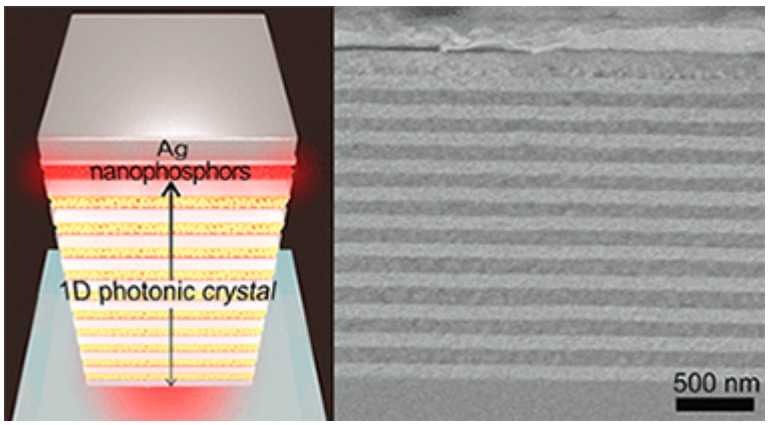


initial state when photoexcitation stops, and the eventual degradation after intense exposure times can thus be rationalized.

Tamm Plasmons Directionally Enhance Rare-Earth Nanophosphor Emission

Geng, DL, Cabello-Olmo, E, Lozano, G, Miguez, H
ACS Photonics, **6** (2019) 634-641
Marzo, 2019 | **DOI:** 10.1021/acsphotonics.8b01407

Rare-earth-based phosphors are the materials on which current solid-state lighting technology is built. However, their large crystal size impedes the tuning, optimization, or manipulation of emitted light that can be achieved by their integration in nanophotonic architectures. Herein we

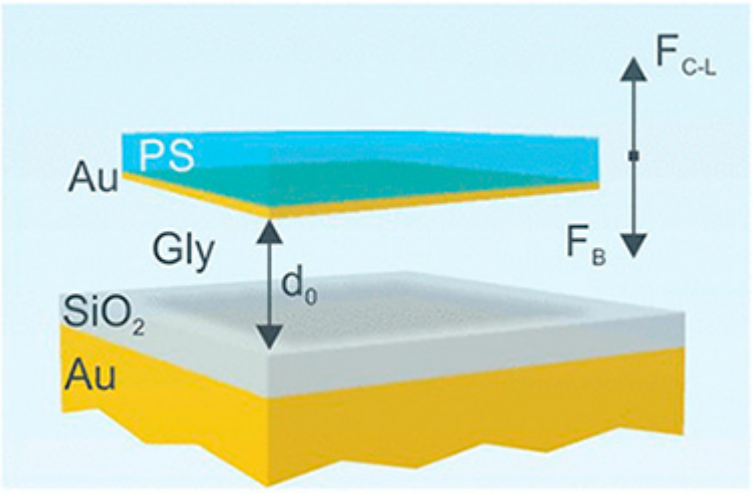


demonstrate a hybrid plasmonic-photonic architecture capable of both channeling in a specific direction and enhancing by eight times the emission radiated by a macroscopically wide layer of nanophosphors. In order to do so, a slab of rare-earth-based nanocrystals is inserted between a dielectric multilayer and a metal film, following a rational design that optimizes the coupling of nanophosphor emission to collective modes sustained by the metal-dielectric system. Our approach is advantageous for the optimization of solid-state lighting systems.

Casimir-Lifshitz Force Based Optical Resonators

Esteso, V, Carretero-Palacios, S, Miguez, H
Journal of Physical Chemistry Letters, **10** (2019) 5856-5860
Octubre, 2019 | **DOI:** 10.1021/acs.jpclett.9b02030

We theoretically investigate the building of optical resonators based on the levitation properties of thin films subjected to strong repulsive Casimir-Lifshitz forces when immersed in an adequate medium and confronted with a planar substrate. We propose a design in which cavities



supporting high Q-factor optical modes at visible frequencies can be achieved by means of combining commonly found materials, such as silicon oxide, polystyrene or gold, with glycerol as a mediating medium. We use the balance between flotation and repulsive Casimir-Lifshitz forces in the system to accurately tune the optical cavity thickness and hence its modes. The effects of other forces, such as electrostatic, that may come into play are also considered. Our results constitute a proof of concept that may open the route to the design of photonic architectures in environments in which dispersion forces play a substantial role and could be of particular relevance for devising novel microfluidic optical resonators.

From structure to luminescence investigation of oxyfluoride transparent glasses and glass-ceramics doped with Eu3+/Dy3+ ions

Walas, M, Lisowska, M, Lewandowski, T, Becerro, AI, Lapinski, M, Synak, A, Sadowski, W, Koscielska, B
Journal of Alloys and Compounds, **896** (2019) 1410-1418
Octubre, 2019 | **DOI:** 10.1016/j.jallcom.2019.07.017

Glasses and glass-ceramics with nominal composition 73 TeO2- 4BaO-3Bi(2)O(3)-18SrF(2)-2RE(2)O(3) (where RE = Eu, Dy) have been synthesized by conventional melt-quenching technique and subsequent heat treatment at 370 degrees C for 24 h in air atmosphere. Various Eu3+ to Dy3+ molar ratio have been applied to investigate luminescence properties in both glass and glass-ceramic matrices. Especially, white light emission through simultaneous excitation of Eu3+ and Dy3+ has been studied in detail. Influence of crystalline SrF2 phase on luminescence kinetics has been determined by luminescence decay time measurements. Presence of

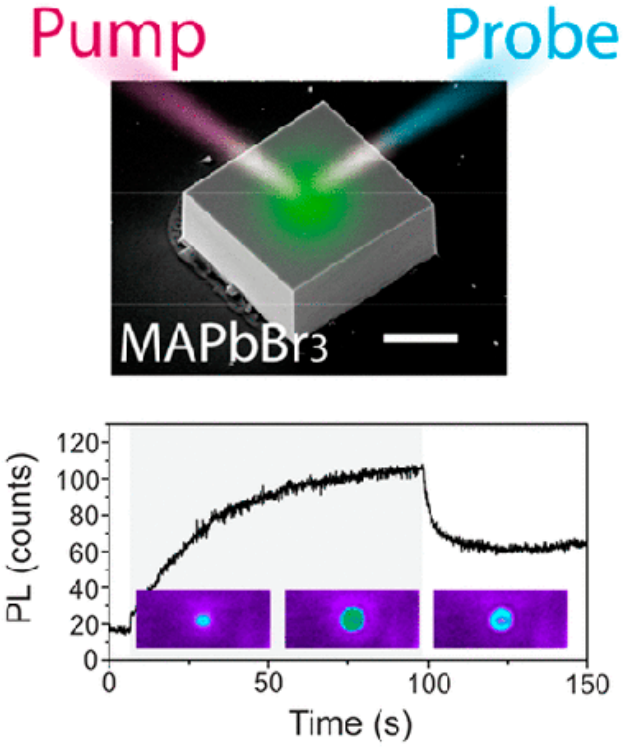
crystalline SrF2 phase has been confirmed by X-ray diffraction technique XRD and transmission electron microscopy TEM. X-ray photoelectron spectroscopy XPS and Fourier-transform infrared spectroscopy FTIR have been applied to get further insight into structural properties of glass and glass-ceramic materials. Color tunable white light emission has been obtained using UV excitation. Influence of the SrF2 crystallization on luminescence properties of prepared materials have been described in detail. Moreover, luminescence properties and especially emission color dependence on the Eu3+ to Dy3+ molar ratio have been exhaustively studied. Color-tunable white light emission has been observed as a result of simultaneous radiative transition of both, Eu3+ and Dy3+ ions when applying UV excitation. Judd - Ofelt and other optical parameters have been calculated based on luminescence emission spectra. Achieved results confirm that tellurite glass-ceramics containing SrF2 nanocrystals are good hosts for RE3+ ions and they can be considered as new phosphors for white light emitting diodes WLEDs.

Spatially Resolved Analysis of Defect Annihilation and Recovery Dynamics in Metal Halide Perovskite Single Crystals

Galisteo-Lopez, JF, Calvo, ME, Miguez, H

ACS Applied Energy Materials, **2** (2019) 6967-6972
Octubre, 2019 | **DOI:** 10.1021/acsaem.9b01335

The spectacular advances in efficiency of optoelectronic devices based on lead-halide perovskites have been accompanied by detailed structural and optical studies regarding the



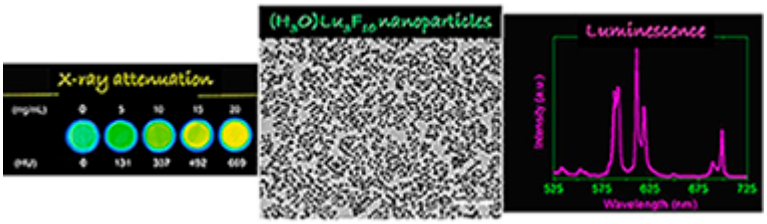
instability presented by these materials, which constitute their main bottleneck for commercialization. Following a pump and probe scheme in a laser scanning confocal microscope, we resolve the photoinduced emission activation/deactivation dynamics in

CH3NH3PbBr3 single crystals with millisecond and sub-micrometer resolution. This is complemented with a study of spectral variations and interpreted in the framework of light-induced ion migration and associated defect passivation. Our results point to the presence of photoinduced structural changes accompanying the migration of ions.

Luminescence and X-ray Absorption Properties of Uniform Eu3+:(H3O)Lu3F10 Nanoprobes

Gonzalez-Mancebo, D, Becerro, AI, Corral, A, Balcerzyk, M, Ocana, M
Nanomaterials, **9** (2019) 1153
Agosto, 2019 | **DOI:** 10.3390/nano9081153

Due to the high atomic number of lutetium and the low phonon energy of the fluoride matrix, Lu-based fluoride nanoparticles doped with active lanthanide ions are potential candidates as bioprobes in both X-ray computed tomography and luminescent imaging. This paper shows a

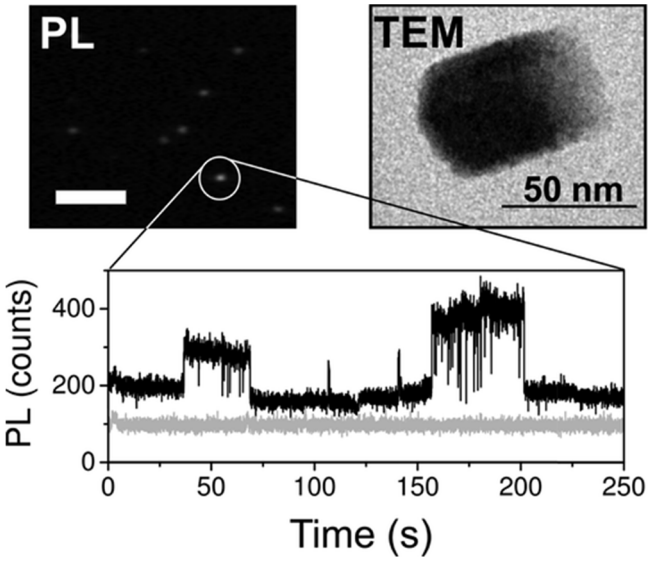


method for the fabrication of uniform, water-dispersible Eu3+:(H3O)Lu3F10 nanoparticles doped with different Eu contents. Their luminescent properties were studied by means of excitation and emission spectra as well as decay curves. The X-ray attenuation capacity of the phosphor showing the highest emission intensity was subsequently analyzed and compared with a commercial contrast agent. The results indicated that the 10% Eu3+-doped (H3O)Lu3F10 nanoparticles fabricated with the proposed polyol-based method are good candidates to be used as dual probes for luminescent imaging and X-ray computed tomography.

Mechanism of Photoluminescence Intermittency in Organic-Inorganic Perovskite Nanocrystals

Galisteo-Lopez, JF, Calvo, ME, Rojas, TC, Miguez, H
ACS Applied Materials & Interfaces, **11** (2019) 6344-6349
Febrero, 2019 | **DOI:** 10.1021/acsami.8b17122

Lead halide perovskite nanocrystals have demonstrated their potential as active materials for optoelectronic applications over the past few years. Nevertheless, one issue that hampers their applicability has to do with the observation of photoluminescence intermittency, commonly referred to as "blinking", as in their inorganic counterparts. Such behavior, reported for structures well above the quantum confinement regime, has been discussed to be strongly related to the presence of charge carrier traps. In this work, we analyze the characteristics of this intermittency and explore the dependence on the surrounding atmosphere, showing evidence for the critical role played by the presence of oxygen. We discuss a possible mechanism

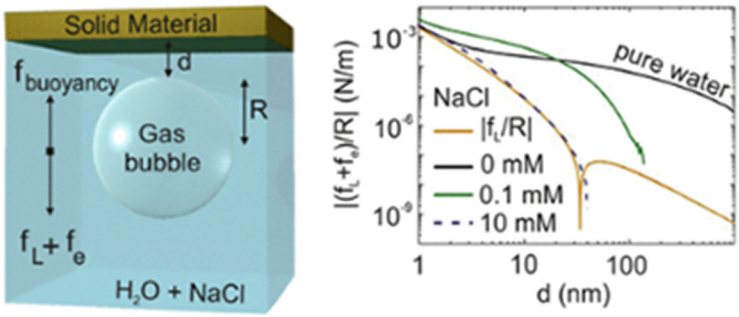


in which a constant creation/annihilation of halide-related carrier traps takes place under light irradiation, with the dominant rate being determined by the atmosphere.

Trapping of Gas Bubbles in Water at a Finite Distance below a Water-Solid Interface

Esteso, V, Carretero-Palacios, S, Thiyam, P, Miguez, H, Parsons, DF, Brevik, I, Bostrom, M
Langmuir, **35** (2019) 4218-4223
Marzo, 2019 | **DOI:** 10.1021/acs.langmuir.8b04176

Gas bubbles in a water-filled cavity move upward because of buoyancy. Near the roof, additional forces come into play, such as Lifshitz, double layer, and hydrodynamic forces. Below uncharged



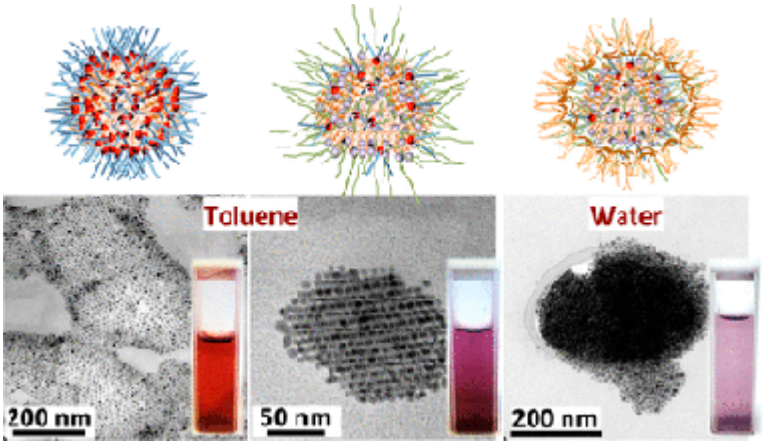
metallic surfaces, repulsive Lifshitz forces combined with buoyancy forces provide a way to trap micrometer-sized bubbles. We demonstrate how bubbles of this size can be stably trapped at experimentally accessible distances, the distances being tunable with the surface material. By contrast, large bubbles (>= 100 mu m) are usually pushed toward the roof by buoyancy forces and adhere to the surface. Gas bubbles with radii ranging from 1 to 10 mu m can be trapped at equilibrium distances from 190 to 35 nm. As a model for rock, sand grains, and biosurfaces, we consider dielectric materials such as silica and polystyrene, whereas aluminium, gold, and silver

are the examples of metal surfaces. Finally, we demonstrate that the presence of surface charges further strengthens the trapping by inducing ion adsorption forces.

Monodisperse Gold Cuboctahedral Nanocrystals Directly Synthesized in Reverse Micelles: Preparation, Colloidal Dispersion in Organic Solvents and Water, Reversible Self-Assembly and Plasmonic Properties

Luna, C, Castaneda-Rodriguez, D, Barriga-Castro, ED, Nunez, NO, Mendoza-Resendez, R
Langmuir, **34** (2019) 14291-14299
Noviembre, 2019 | **DOI:** 10.1021/acs.langmuir.9b02374

The synthesis of organic-solvent-dispersible gold nanoparticles in reverse micelles of didodecyldimethylammonium bromide (DDAB) is revisited in the present investigation. Some parameters of synthesis, specifically the reaction volume and the concentration of the reducing agent, were slightly modified obtaining directly monodisperse gold nanocrystals (AuNCs)



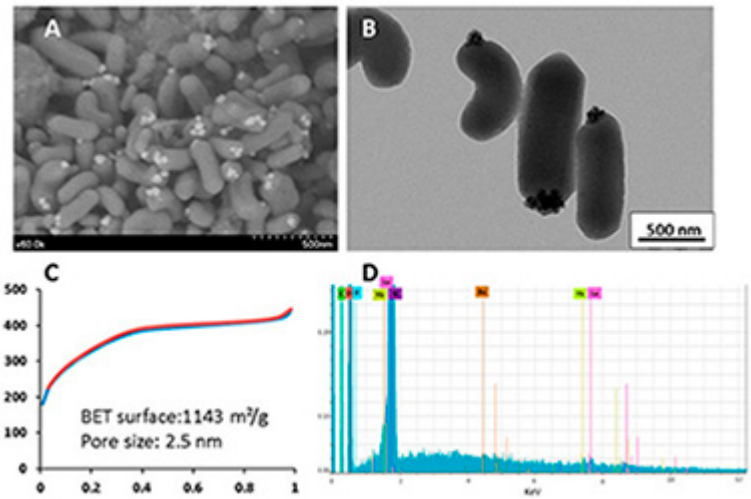
without the need to use additional active surfactants or additional treatments such as digestive ripening. Interestingly, most of the obtained AuNCs display the same exposed crystalline faces composed of six bounding facets (four {111} faces and two {002} faces), corresponding to single-crystalline face-centered cubic nanoparticles with a cuboctahedron shape. When these AuNCs are subsequently functionalized with 1-decanethiol (C10H21SH) or 1-dodecanethiol (C12H25SH), they don't experience significant changes in their size or crystalline texture, however, they self-aggregate directly in the suspension at room temperature into faceted supramolecular structures and exhibit collective plasmonic excitations. Such self-organization is reversible under heating treatments allowing the observation of the influence of the AuNCs aggregation state on their plasmonic properties. Fourier transform infrared spectroscopy reveals that thiols only replace partially the DDAB molecules, and thus, DDAB molecules remain present in the thiol-capped AuNCs. To turn the thiol-capped nanocrystals into water-dispersible nanocrystals and extend their technological potential, they are stabilized with poloxamer 407 obtaining highly stable purple colloids in water.

Encapsulation of Upconversion Nanoparticles in Periodic Mesoporous Organosilicas

Rahmani, S, Jimenez, CM, Aggad, D, Gonzalez-Mancebo, D, Ocana, M, Ali, LMA, Nguyen, C, Nieto, AIB, Francolon, N, Oliveiro, E, Boyer, D, Mahiou, R, Raehm, L, Gary-Bobo, M, Durand, JO, Charnay, C

Molecules, **24** (2019) 22
Noviembre, 2019 | **DOI:** 10.3390/molecules24224054

(1) Background: Nanomedicine has recently emerged as a promising field, particularly for cancer theranostics. In this context, nanoparticles designed for imaging and therapeutic applications are of interest. We, therefore, studied the encapsulation of upconverting nanoparticles in mesoporous organosilica nanoparticles. Indeed, mesoporous organosilica nanoparticles have been shown to be very efficient for drug delivery, and upconverting nanoparticles are interesting

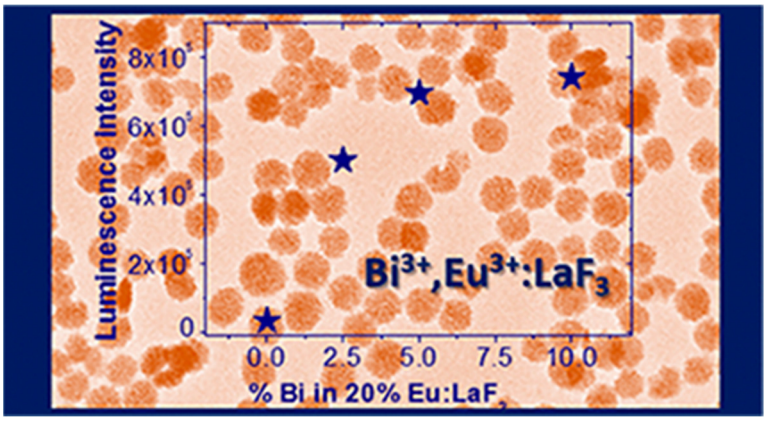


for near-infrared and X-ray computed tomography imaging, depending on the matrix used. (2) Methods: Two different upconverting-based nanoparticles were synthesized with Yb3+-Er3+ as the upconverting system and NaYF4 or BaLuF5 as the matrix. The encapsulation of these nanoparticles was studied through the sol-gel procedure with bis(triethoxysilyl)ethylene and bis(triethoxysilyl)ethane in the presence of CTAB. (3) Results: with bis(triethoxysilyl)ethylene, BaLuF5: Yb3+-Er3+, nanoparticles were not encapsulated, but anchored on the surface of the obtained mesoporous nanorods BaLuF5: Yb3+-Er3+@Ethylene. With bis(triethoxysilyl)ethane, BaLuF5: Yb3+-Er3+ and NaYF4: Yb3+-Er(3+)nanoparticles were encapsulated in the mesoporous cubic structure leading to BaLuF5: Yb3+-Er3+@Ethane and NaYF4: Yb3+-Er3+@Ethane, respectively. (4) Conclusions: upconversion nanoparticles were located on the surface of mesoporous nanorods obtained by hydrolysis polycondensation of bis(triethoxysilyl)ethylene, whereas encapsulation occurred with bis(triethoxysilyl)ethane. The later nanoparticles NaYF4: Yb3+-Er3+@Ethane or BaLuF5: Yb3+-Er3+@Ethane were promising for applications with cancer cell imaging or X-ray-computed tomography respectively.

Enhancing Luminescence and X-ray Absorption Capacity of Eu3+:LaF3 Nanoparticles by Bi3+ Codoping

Mancebo, DG, Becerro, AI, Corral, A, Moros, M, Balcerzyk, M, de la Fuente, JM, Ocana, M
ACS Omega, **4** (2019) 765-774
Enero, 2019 | **DOI:** 10.1021/acsomega.8b03160

Bi3+ codoping has been proposed in this work with a twofold objective, namely, enhancing the luminescence emission of Eu3+:LaF3 nanoparticles (NPs) and increasing their X-ray attenuation capacity, with the purpose of obtaining a bimodal bioprobe for luminescence bioimaging and X-



ray computed tomography. The synthesis method, reported here for the first time for LaF3 particles, allowed obtaining uniform, nonaggregated NPs using a homogeneous precipitation in polyol medium at room temperature in just 2 h. The simplicity of the synthesis method allows the large-scale production of NPs. LaF3 NPs with different Eu3+ contents were first synthesized to find the critical Eu3+ concentration, producing the highest emission intensity. This concentration was subsequently used to fabricate Bi3+-Eu3+-codoped LaF3 NPs using the same method. The emission intensity of the codoped NPs increased in more than one order of magnitude, thanks to the possibility of excitation through the Bi3+. Eu3+ energy-transfer band. The luminescence properties of the codoped NPs were analyzed in detail to find the mechanism responsible for the emission enhancement. Finally, it was demonstrated that the high atomic number of Bi3+, higher than that of lanthanides, was an added value of the material because it increased its X-ray attenuation capacity. In summary, the LaF3 NPs codoped with Eu3+ and Bi3+ presented in this work are promising candidates as a bimodal bioprobe for luminescence bioimaging and X-ray computed tomography.

Biocompatibility assessment of up-and down-converting nanoparticles: implications of interferences with in vitro assays

Pem, Barbara, Gonzalez-Mancebo, Daniel, Moros, Maria, Ocana, Manuel, Becerro, Ana Isabel, Pavicic, Ivan, Selmani, Atida, Babic, Michal, Horak, Daniel, Vinkovic Vrcek, Ivana
Methods and Applications in Fluorescence, **7** (2019) 014001
Enero, 2019 | **DOI:** 10.1088/2050-6120/aae9c8

The safety assessment of nanoparticles (NPs) is crucial during their design and development for biomedicine. One of the prerequisite steps during this evaluation is in vitro testing that employs cell-based assays not always validated and well-adapted for NPs. Interferences with in vitro

assays may arise due to the nano-related optical, oxidative, fluorescent, surface and catalytic properties of NPs. Thus, proper validation of each assay system has to be performed for each NP type. This study aimed to evaluate the applicability of the most common in vitro cytotoxicity assays for the safety assessment of up- and down-converting lanthanide-doped NPs. Conventional cell viability tests and fluorescence-based assays for oxidative stress response were selected to determine the biological effects of up- and down-converting NPs to human brain cells. Comparison with known silver and iron oxide NPs was made for verification purposes. Both the plate reader and flow cytometric measurements were examined. The obtained results indicated that both types of Ln-doped NPs interfered to a much lesser extent than metallic NPs. In addition, the great potential of both up- and down-converting NPs for biomedicine was manifested due to their biocompatibility and low toxicity.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

Enhancing Nanoparticle Bragg reflectors: A smart analytical tool for biosensing
González-Pedro, Victoria, Calvo, Mauricio E, Míguez, H., Maquieira, Ángel
Biosensors and Bioelectronics: X, **1**, (2019), p. 100012
April, 2019 | **DOI**: 10.1016/j.biosx.2019.100012

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

International Conference on Perovskite Photonics and Optoelectronics (NIPHO19)
25 – 27 febrero [Jerusalén, Israel]

Photoemission Properties of Perovskite Thin Films, Microcrystals and Nanocrystals
Hernán Míguez
Conferencia Invitada

International Conference on Interfaces in Organic and Hybrid Thin-Film Optoelectronics | INFORM192
5 – 7 febrero [Valencia, España]

Perovskite Nanocrystals in Mesostructured Media: from Materials to Devices
Hernán Míguez
Conferencia Invitada

2019 MRS Spring Meeting & Exhibit
22 abril [Estados Unidos de América]

Photophysical Properties of Perovskite Thin Films, Microcrystals and Nanocrystals
Hernán Míguez
Comunicación oral

11th International Conference on Hybrid and Organic Photovoltaics
12 mayo [Roma, Italia

Mechanism of Photoluminescence Intermittency in Organic-Inorganic Perovskite Nanoparticles
Juan Francisco Galisteo López
Comunicación oral

International Conference on Hybrid and Organic Photovoltaics - HOPV19
12 – 15 mayo [Roma, Italia]

APbX3 Perovskite Nanocrystals in Porous Matrices: Size Control and New Potential Applications
Andrea Rubino, Juan F. Galisteo-López, Mauricio Calvo, Hernan Miguez
Poster

Improving the Bulk Emission Properties of CH3NH3PbBr3 by Modifying the Halide-Related Defect Structure
David O. Tiede, Juan F. Galisteo-López, Maurico E. Calvo, Hernán Míguez
Poster

FOTOFUEL Workshop: Current challenges in solar fuels production
13 – 14 mayo [Madrid, España]

Optical material for Solar energy applications
Hernán Míguez
Conferencia Invitada

EMRS Spring meeting 2019
27 mayo [Niza, Francia]

ABX3 perovskite nanocrystals in porous matrices
Mauricio E. Calvo Roggiani
Comunicación oral

23rd International Symposium on Radiopharmaceutical Sciences
26 -31 Mayo [Beijing, China]

18F-fluorination of BaGdF5 nanoparticles for multimodal imaging and PET/CT biodistribution in mouse. S 166
L. Fernandez-Maza, A. Corral, A. Becerro, D. Gonzalez, A. Parrado, M. Balcerzyk, M. Ocana
Poster

NanoSpain
28 – 31 mayo [Barcelona, España]

HoF3 and DyF3 nanoparticles as dual contrast agents for MRI and CT
A. I. Becerro, D. González-Mancebo, A. Corral, M. Balcerzyk, M.L. García-Martín, J.M. de la Fuente, M. Ocaña
Comunicación oral

PIERS 2019: Progress In Electromagnetics Research Symposium
17-20 junio [Roma, Italia]

Forces at the Nanoscale for Trapping Gas Bubbles inWater at a Finite Distance below a Water-solid Inter-face
Carretero-Palacios, Sol, Esteso Victoria, Priyadarshini Thiyam, Hernán Míguez, Drew F. Parsons, Iver Bre-vik, Mathias Bostrom
Conferencia invitada

Controlling the Casimir-Lifshitz Force between PlaneParallel Systems with Multi-layered Dielectric Nanos-tructures
Carretero-Palacios, Sol, Esteso Victoria, Hernán Míguez
Comunicación Oral

2019 OSA Advanced Photonics Congress
29 julio – 1 agosto [Estados Unidos de América]

Transparent Nanophosphor Films for Efficient White-Light Generation
Gabriel Lozano
Comunicación oral

Nanophotonics Tunes Rare-Earth Nanophosphor Emission
Gabriel Lozano
Comunicación oral

Metal halide perovskite semiconductors, from basic physics to applications
19 – 20 agosto [Suecia]

Photophysical properties of metal halide perovskite thin films, microcrystals and nanocrystals
Hernán Míguez
Conferencia Invitada

23rd International Symposium on Radiopharmaceutical Sciences
28 octubre – 1 noviembre [Vienna, Austria]

Labeling of [18F]BaGdF5 nanoparticles for multimodal imaging and PET/CT biodistribution in mice
L. Fernandez-Maza, A. Corral, A.I. Becerro, D. Gonzalez, A. Parrado, M. Balcerzyk, M. Ocaña
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

PEROVSKITA19 | Research Network. Perovskites for solar energy conversion and optoelectronics
06 mayo [Castellón, España]

Temperature-dependent photoluminescence of MAPbI3 nanocrystals
Andrea Rubino, Mauricio Calvo, Hernán Míguez
Comunicación oral

Optical properties and devices with templated perovskite nanocrystals
Mauricio Calvo, Hernán Míguez
Comunicación oral

XXXVII Reunión Bienal de la Real Sociedad Española de Física
15 - 19 julio [Zaragoza, España]

Multifunctional Optical Materials
Hernán Míguez
Conferencia Invitada

Tamm Plasmons for Controlled Emission of Nanophosphors
Elena Cabello-Olmo, Dongling Geng, Gabriel Lozano, Hernán Míguez
Comunicación Oral

Flexible optically disordered materials for LED coatings
José María Miranda-Muñoz , Dongling Geng , Mauricio E. Calvo, Gabriel Lozano , Hernán Míguez
Comunicación Oral

Optical interference effects on the Casimir-Lifshitz force between plane-parallel systems with multilayer nanostructures
Hernán R. Míguez García, Sol Carretero Palacios,, Victoria Esteso Carrizo
Póster-Flash Talk

Tailoring the absorption and emission properties of nanomaterials through their photonic Environment
Alberto Jiménez-Solano , Juan F. Galisteo-López , Hernán Míguez
Invited symposio

PEROVSKITA19 | Perovskites for solar energy conversion and optoelectronics
27 septiembre [Madrid, España]

Photophysical properties of perovskite microcrystals and nanocrystals
Hernán Míguez
Conferencia Invitada

Temperature-dependent photoluminescence of MAPbI3 nanocrystals
Andrea Rubino, Mauricio Calvo, Hernan Miguez
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Partículas uniformes de materiales luminiscentes basados en molibdatos y volframatos de tierras raras y alcalinotérreos para aplicaciones optoelectrónicas y biomédicas**
Autor:	Mariano Laguna Moreno
Directores:	Manuel Ocaña Jurado y Nuria Ofelia Nuñez Alvarez
Calificación:	Sobresaliente “Cum Laude”
Centro:	Universidad de Sevilla
Fecha:	19 de marzo de 2019

Título:	**Integration of optically random media into optoelectronic devices**
Autor:	José María Miranda Muñoz
Directores:	Gabriel Lozano Barbero y Hernán Míguez García
Calificación:	Sobresaliente “Cum Laude”

Centro: Universidad de Sevilla
Fecha: 12 de diciembre de 2019

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Optimización de recubrimientos luminiscentes de perovskita para LEDs**
Autor: Carlos Romero Pérez
Directores: Mauricio Calvo Roggiani y Hernán Míguez García
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (2 julio 2019)

Título: **Diseño, preparación y caracterización de materiales emisores nanoestructurados para el control espectral y direccional de la luz emitida**
Autor: Elena Cabello Olmo
Directores: Gabriel Lozano Barbero y Hernán Míguez García
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (2 julio 2019)

Título: **Procesos fotofísicos en materiales semiconductores de interés en sensores**
Autor: David Otto Tiede
Directores: Juan F. Galisteo López y Hernán Míguez García
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (2 julio 2019)

Título: **Fabricación y caracterización fotovoltaica de celdas solares de perovskita**
Autor: José Luis Olivas Sánchez
Directores: Laura Calio y Hernán Míguez García
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (2 julio 2019)

Título: **Síntesis, caracterización y propiedades ópticas de partículas uniformes de Ln: BaWO4 (Ln: Eu, Dy)**
Autor: María Escamilla Rebollo
Directores: Nuria O. Núñez Álvarez , José Antonio Navío Santos
Grado: Trabajo Fin de Grado
Año Académico: 2018-2019 (24 septiembre 2019)

Título: **Agentes de contraste para el diagnóstico médico por imagen**
Autor: Diego Martínez Gutiérrez
Directores: Manuel Ocaña Jurado, Ana Isabel Becerro Nieto
Grado: Trabajo Fin de Master
Año Académico: 2018-2019 (12 diciembre 2019)

Título:	**Nanopartículas luminiscentes con aplicaciones tecnológicas**
Autor:	Beatriz Medrán Barranco
Directores:	Manuel Ocaña Jurado, Ana Isabel Becerro Nieto
Grado:	Trabajo Fin de Master
Año Académico:	2018-2019 (12 diciembre 2019)

Título:	**Síntesis y funcionalización de nanopartículas uniformes de NaLn(MoO4)2 (Ln: Dy3+, Ho3+)**
Autor:	Miguel Omar Escudero Cortines
Directores:	Nuria O. Núñez Álvarez
Grado:	Trabajo Fin de Master
Año Académico:	2018-2019 (12 diciembre 2019)

Título:	**Síntesis y propiedades luminiscentes de nanopartículas de NaY(MoO4)2 dopadas con Eu3+**
Autor:	Carmen María Berraquero Romero
Directores:	Nuria O. Núñez Álvarez, Manuel Ocaña Jurado
Grado:	Trabajo Fin de Master
Año Académico:	2018-2019 (12 diciembre 2019)

DOCENCIA / TEACHING

Máster Universitario en Ciencia y Teconología de Nuevos Materiales
Propiedades Términas, Dieléctricas y Ópticas
Hernán Míguez, Gabriel Lozano
Universidad de Sevilla

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Premio Real Maestranza de Caballería de Sevilla 2019
Alberto Jiménez Solano. Diciembre 2019

Premio Estraordinario de Doctorado. Universidad de Sevilla
Alberto Jiménez Solano. Junio 2019

XXXVII Bienal de la RSEF. Premio a la mejor flashtalk
1er premio. Victoria Esteso. Julio 2019

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

Instituto de Ciencia de Materiales de Barcelona. Grupo Optoelectronic Properties of Nanostructured Materials con el Prof. Alejandro Goñi
Bellaterra, Barcelona
Andrea Rubino 04-08 de febrero

Imperial College London al Grupo Complex Nanophotonics. Faculty of Natural Sciences, Department of Physics con el Dr. Riccardo Sapienza
Londres, Inglaterra
Andrea Rubino 04 marzo al 02 de abril

Conditions Extrêmes et Matériaux : Haute Température et Irradiation (CNRS)
Orleans (Francia)
Ana Isabel Becerro Nieto 1-31 de Julio 2019

Instituto de Ciencia de Materiales de Barcelona. Grupo Nanostructured Materials for Optoelectronics and Energy Harvesting con el Dr. Agustín Mihi.
Bellaterra, Barcelona
Elena Cabello 23 noviembre al 01 de diciembre

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS
PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Instituto de Ciencia de Materiales de Barcelona. Barcelona, España.
Prof. Alejandro Goñi. 15-16 enero

Instituto IMDEA Materiales, Madrid, España.
Dr. Rubén Costa Riquelme 30-31 enero

Instituto de Ciencia de Materiales de Barcelona. Barcelona, España.
Dr Agustín Mihi Cervelló 04-05 junio

Walter Schottky Institut. Technische Universität München. Múnich, Alemania.
Dr. Úrsula Wurstbauer. 15-16 julio

Universidad Autónoma de Madrid. Instituto IMDEA Materiales. Madrid. España.
Dr. Giovanni Bottari 10 diciembre

L'Institut Langevin, ondes et imagen. ESPCI Paris - PSL - CNRS París, Francia.
Prof. Rèmi Carminati 11-12 diciembre

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 360º 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

Catedrático

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

Profesor Titular

Dra. Josefa María Borrego Moro
Dra. M. del Carmen Gallardo Cruz
Dra. Ángela Gallardo López
Dr. Felipe Gutiérrez Mora
Dr. José María Martín Olalla
Dra. Ana Morales Rodríguez
Dr. Francisco Javier Romero Landa

Profesor Emérito

Dr. Alejandro Conde Amiano

Profesor Contratado

Dr. Jhon J. Ipus Bados
Dr. Victor Morales Flórez

Investigador postdoctoral

Dr. Luis M. Moreno Ramírez
Dra. JiaYan Law

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Gdo. Álvaro Díaz García
Gda. Cristina López Pernía
Gdo. Alejandro F. Manchón Gordón
Gda. Carmen Muñoz Ferreiro
Gdo. Pedro Rivero Antúnez
Gdo. Antonio Vidal Crespo

Investigador honorario

Dr. Jaime del Cerro
Dr. Arturo Domínguez Rodríguez
Dr. Justo Jiménez

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Modelado y Control de la Histéresis en Materiales Magnetocalóricos para Refrigeración y Conversión de Energía

Código/Code: MAT2016-77265-R
Periodo/Period: 30-12-2016 / 29-09-2020
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Investigador responsable/Research head: Victorino Franco García, Javier Sebastián Blázquez Gámez
Componentes/Research group: Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon J. Ipus Bados

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales Processing and characterization of ceramic composites with two-dimensional laminar nanomaterials (CMC-NANOLAM)

Código/Code: PGC2018-101377-B-I00
Periodo/Period: 01-01-2019 / 31-12-2022
Organismo Financiador/Financial source: Ministerio de Ciencia, Innovación y Universidades
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

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 analizar 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.

Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno Processing and microstructural, mechanical and electrical characterization of ceramic-graphene composites

Código/Code:	MAT2015-67889-P
Periodo/Period:	01-01-2016 / 31-09-2019
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	89.177 €
Investigador responsable/Research head:	Ángela Gallardo López, Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora

RESUMEN / ABSTRACT

En la actualidad se plantean interesantes expectativas sobre los compuestos cerámica-grafeno, propuestos para aplicaciones en catálisis, almacenamiento y conversión de energía, protección del medio ambiente y biotecnología. Pero aún se requieren importantes esfuerzos para dar respuesta a cuestiones abiertas. Hay que incidir en aspectos como la resistencia a la cizalla de las intercaras cerámica-grafeno -esencial para asegurar la transferencia de carga efectiva sobre las láminas de grafeno-, la distribución homogénea del grafeno en la matriz

cerámica y la relación de tamaños entre matriz y refuerzo, para maximizar el aumento de tenacidad y de conductividad eléctrica, así como también explorar sus propiedades mecánicas a alta temperatura.

En este proyecto se plantea un estudio sistemático de compuestos de matrices cerámicas con grafeno, desde la fabricación hasta la caracterización microestructural, mecánica y eléctrica, con el objetivo de mejorar la comprensión de los mecanismos que controlan estas propiedades al incorporar nanoestructuras de grafeno a una matriz cerámica. Se procesarán compuestos de dos matrices cerámicas diferentes, de alúmina y de circona tetragonal dopada con óxido de itrio (3YTZP), con grafeno mediante técnicas coloidales, prestando especial atención a la dispersión del grafeno en la matriz cerámica, aspecto no exento de dificultades y que es clave para conseguir la mejora de las propiedades. La sinterización se realizará en un horno de descarga de plasma (SPS, spark plasma sintering) de última generación, optimizando las condiciones para conseguir compuestos densos y de tamaño de grano nanométrico. Para el análisis microestructural se utilizarán técnicas como la difracción de rayos X, la espectroscopía Raman, y la microscopía electrónica de barrido y transmisión. Con ellas se evaluarán las fases cristalográficas presentes, el tamaño de grano, la distribución de las nanoestructuras de grafeno, etc.

Desde el punto de vista del diseño de materiales avanzados, es fundamental investigar la relación entre microestructura y propiedades mecánicas y eléctricas. Las propiedades mecánicas a temperatura ambiente (dureza, tenacidad a la fractura y resistencia a flexión) se abordarán mediante indentación y ensayos de flexión, a escalas macro y microscópica. A alta temperatura, se estudiará la deformación plástica de los compuestos cerámica-grafeno mediante ensayos de fluencia en atmósferas controladas. También se estudiará el comportamiento tribológico de los compuestos y se evaluará su conductividad eléctrica, una de las propiedades más interesantes ya que se modifica de forma notoria como resultado de la incorporación del grafeno a estos sistemas cerámicos. La respuesta eléctrica se analizará en un amplio rango de temperaturas, bien mediante espectroscopía de impedancia compleja, bien mediante medidas de conductividad en corriente continua en el caso de los compuestos menos resistivos.

Nowadays, interesting prospects are proposed for ceramic-graphene composites, in application fields such as catalysis, energy storage and conversion, environment protection and biotechnology. A great effort is still required to answer open questions. Issues such as shear resistance of the ceramic-graphene interface –essential to obtain an effective load transfer to the graphene sheets-, distribution of graphene in the ceramic matrix -to maximize the reinforcement mechanisms and electrical conductivity- and the high temperature mechanical properties in these composites need special attention.

A systematic study of ceramic matrix graphene composites, including processing and microstructural, mechanical and electrical characterization is proposed in this project, with the aim of improving the comprehension of mechanisms controlling these properties when adding graphene nanostructures to a ceramic matrix. Both alumina and yttria tetragonal zirconia (3YTZP) graphene composites will be processed by means of colloidal techniques. Special attention will be devoted to the dispersion of graphene in the ceramic matrix which is not a straightforward aspect, but is key to improve mechanical and functional properties. Sintering will be carried out by spark plasma sintering, SPS. Conditions will be optimized in order to obtain fully dense composites with nanometric grain size. Microstructural analysis will be performed by X ray diffraction, Raman spectroscopy, scanning and transmission electron microscopy (SEM

and TEM). The present crystallographic phases, grain size and distribution of graphene nanostructures will be evaluated.

In order to design advanced materials, it is necessary to study the relationship between microstructure and mechanical or electrical properties. Room temperature mechanical properties (hardness, fracture toughness and flexural resistance) will be characterized by indentation and bending tests at macro and microscopic scales. At high temperature, the plastic behavior of these ceramic-graphene composites will be assessed by creep tests under controlled atmosphere. Tribological behavior of the composites will also be studied to evaluate their resistance to wear. The electrical response will be assessed in a wide range of temperatures by means of complex impedance spectroscopy or by direct current conductivity measurements in the composites with lower resistivity. This is a most interesting property since it can be strongly increased when incorporating graphene to these ceramic systems.

Refuerzo Intragranular de Cerámicas con Fases de Baja Dimensionalidad

Código/Code:	PGC2018-094952-B-I00
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Plan Estatal 2017-2020 Generación Conocimiento - Proyectos I+D+i
Importe total/Total amount:	96.800 €
Investigador responsable/Research head:	Victor Morales Florez
Componentes/Research group:	Luis Esquivias Fedriani, Francisco de Paula Jiménez Morales, Florentino Sánchez Bajo

Procesado, caracterización y propiedades mecánicas de cerámicos nanoestructurados reforzados con nanotubos de carbono

Código/Code:	P12-FQM-1079 (Proyecto de Excelencia)
Periodo/Period:	30-01-2014 / 16-02-2019
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Arturo Domínguez Rodríguez
Componentes/Research group:	Luis María Esquivias Fedriani, Angela Gallardo López, Diego Gómez García, Felipe Gutiérrez Mora, Victor Morales Flórez, Ana Morales Rodríguez, Rodrígo Moreno Botella, Antonio Muñoz Bernabé, Rosalía Poyato Galán, Eugenio Zapata Solvas

PATENTES / PATENTS

CO_2 and SO_2 capture method
Inventores: Victor Manuel Morales Florez, Luis María Esquivias Fedriani
Tipo de Patente: Nacional
Número de Solicitud: 12781643
Fecha Solicitud: 26 de junio de 2019
Entidad Titular: Universidad de Sevilla, Universidad de Cádiz

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Tunable first order transition in La(Fe,Cr,Si)(13) compounds: Retaining magnetocaloric response despite a magnetic moment reduction
Moreno-Ramirez, LM, Romero-Muniz, C, Law, JY, Franco, V, Conde, A, Radulov, IA, Maccari, F, Skokov, KP, Gutfleisch, O
Acta Materialia, **175** (2019) 406-414
DOI: 10.1016/j.actamat.2019.06.022

Materials with a large magnetocaloric response require a large magnetic moment. However, we show in this paper that it is possible to retain both the isothermal entropy change and the adiabatic temperature change even using dopants that reduce the magnetic moment of the parent alloy, provided that the first order character of the transition is enhanced. In this work, a combination of first-principles calculations, experimental determination of the magnetocaloric response (direct and indirect) as well as a new criterion to determine the order of the phase transition are applied to Cr-doped La(Fe,Si)(13) compounds. Despite a reduction in magnetic moment, the magnetocaloric response is retained up to x approximate to 0.3 in LaFe11.6-xCrxSi1.4. Unlike other transition metal dopants, Cr occupy 8b sites and couple antiferromagnetically to Fe atoms. The cross-over of first to second order transition is achieved for a Cr content of x = 0.53, larger in comparison to other dopants (e.g. Ni or Mn). A direct relation between the first order character and the hysteresis is observed.

How concurrent thermomagnetic transitions can affect magnetocaloric effect: The Ni(49+)xMn(36-)xIn(15) Heusler alloy case
Law, JY, Diaz-Garcia, A, Moreno-Ramirez, LM, Franco, V, Conde, A, Giri, AK
Acta Materialia, **166** (2019) 459-465
DOI: 10.1016/j.actamat.2019.01.007

It is usually claimed that structural and magnetic transitions should coincide to improve the total entropy change. Here we show that overlapping structural and magnetic transitions can compete with each other, which gives rise to some signature of the competition misinterpreted as an experimental artifact of the measurements as observed in Ni49+xMn36-xIn15 (nominally x = 0, 0.5, 1.0, 1.5 and 2.0) investigated in this work. Their various magnetic phase transitions show a low temperature conventional magnetocaloric effect (MCE), an inverse MCE followed by another high temperature conventional MCE. For concurrent phase transitions with their

respective transition temperatures close to one another, an observed spike embedded in the high temperature conventional MCE region is found arising from the martensitic transformation. We show that a crossover of the magnetic field dependence of the transition temperatures is obtained for concurrent phase transitions occurring in a narrow temperature range. As an evidence of the complexity of overlapping phase transitions, a recently proposed quantitative method for determining the order of phase transition is applied to this compositional series, showing its applicability even to cases when first- and second-order phase transitions occur in a narrow temperature range.

Modification of the order of the magnetic phase transition in cobaltites without changing their crystal space group

Law, JY, Franco, V, Conde, A, Skinner, SJ, Pramana, SS
Journal of Alloys and Compounds, **777** (2019) 1080-1086
DOI: 10.1016/j.jallcom.2018.11.020

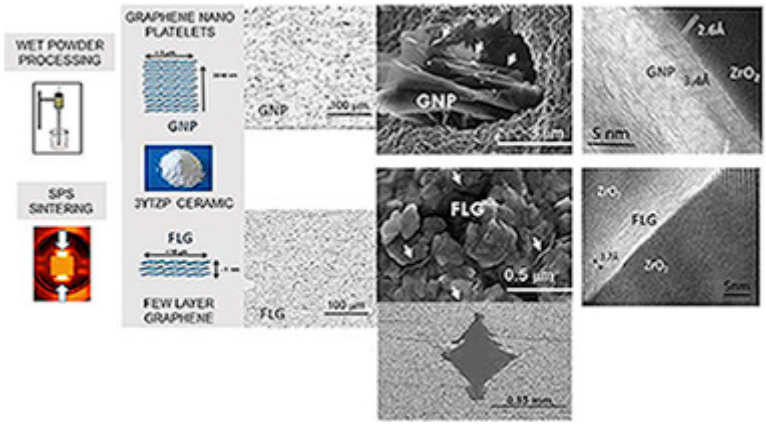
It has been found that GdBa1-xSrxCo2O6-delta can exhibit consecutive magnetic transitions: antiferromagnetic-ferromagnetic (AFM-FM) transition followed by ferromagnetic-paramagnetic transition (FM-PM), which give rise to a coexistence of inverse and conventional magnetocaloric effect (MCE), respectively. In the pristine compound (x = 0), its AFM-FM transition is shown to belong to a first-order phase transition and the FM-PM to a second-order type. Despite it is widely known that the properties of cobaltites are highly influenced by their oxygen content and type of doping carriers, in this work, further evaluation using magnetocaloric analysis (universal curve method and a quantitative criterion using magnetic field dependence of the magnetic entropy change) reveals that the first-order AFM-FM phase transition converts into a second-order character with just Sr doping of x = 0.1 (despite of having the same space group at room temperature and type of dopant carrier as x = 0), severely affecting its thermomagnetic properties. Moreover, the peaks of these two MCE span over a temperature range that is larger than those reported for cobaltite-type materials, making it closer to room temperature applications.

Microstructure, interfaces and properties of 3YTZP ceramic composites with 10 and 20 vol% different graphene-based nanostructures as fillers

Munoz-Ferreiro, C, Morales-Rodriguez, A, Rojas, TC, Jimenez-Pique, E, Lopez-Pernia, C, Poyato, R, Gallardo-Lopez, A
Journal of Alloys and Compounds, **777** (2019) 213-224
Marzo, 2019 | **DOI:** 10.1016/j.jallcom.2018.10.336

The graphene family comprises not only single layer graphene but also graphene-based nanomaterials (GBN), with remarkably different number of layers, lateral dimension and price. In this work, two of these GBN, namely graphene nanoplatelets (GNP) with n similar to 15-30 layers and few-layer graphene (FLG) with n < 3 layers have been evaluated as fillers in 3 mol% yttria stabilized tetragonal zirconia (3YTZP) ceramic composites. Composites with 10 and 20 vol% GNP or FLG have been fabricated by wet powder processing and spark plasma sintering (SPS) and the influence of the content and number of layers of the graphene-based filler has been assessed. For both graphene-based fillers, an intermediate zirconia oxycarbide has been

detected in the grain boundaries. The lower stacking degree and much more homogeneous distribution of the FLG, revealed by transmission electron microscopy (TEM), can improve load transfer between the GBNs and the ceramic matrix. However, high FLG contents lower



densification of the composites, due partly to the larger FLG interplanar spacing also estimated by TEM. The hardness (both Vickers and nanoindentation) and the elastic modulus decrease with increased GBN content and with improved graphene dispersion. The FLG greatly inhibit the crack propagation that occur perpendicular to their preferential orientation plane. The composites with thinner FLG have higher electrical conductivity than those with GNP. The highest electrical conductivity is achieved by composites with 20 vol% FLG in the direction perpendicular to the compression axis during sintering, sigma(perpendicular to) = 3400 +/- 500 Sm-1.

Graphene nanoplatelets for electrically conductive 3YTZP composites densified by pressureless sintering

Lopez-Pernia, C, Gallardo-Lopez, A, Morales-Rodriguez, A, Poyato, R
Journal of the European Ceramic Society, **39** (2019) 4435-4439
Noviembre, 2019 | **DOI:** 10.1016/j.jeurceramsoc.2019.05.067

3 mol% yttria tetragonal zirconia polycrystalline (3YTZP) ceramic composites with 2.5, 5 and 10 vol% graphene nanoplatelets (GNP) were pressureless sintered in argon atmosphere between 1350 and 1450 degrees C. The effects of the GNP content and the sintering temperature on the densification, microstructure and electrical properties of the composites were investigated. An isotropic distribution of GNP surrounding ceramic regions was exhibited regardless the GNP content and sintering temperature used. Electrical conductivity values comparable to the ones of fully dense composites prepared by more complex techniques were obtained, even though full densification was not achieved. While the composite with 5 vol% GNP exhibited electrical anisotropy with a semiconductor-type behaviour, the composite with 10 vol% GNP showed an electrically isotropic metallic-type behaviour.

Tribological behavior of graphene nanoplatelet reinforced 3YTZP composites

Gutierrez-Mora, F, Morales-Rodriguez, A, Gallardo-Lopez, A, Poyato, R
Journal of the European Ceramic Society, **39** (2019) 1381-1388
Abril, 2019 | **DOI:** 10.1016/j.jeurceramsoc.2018.11.005

The tribological behavior of graphene nanoplatelet (GNP) reinforced 3 mol% yttria tetragonal zirconia polycrystals (3YTZP) composites with different GNP content (2.5, 5 and 10 vol%) was analyzed and discussed. Their dry sliding behavior was studied using a ball-on-disk geometry with zirconia balls as counterparts, using loads between 2 and 20 N at ambient conditions and compared to the behavior of a monolithic 3YTZP ceramic used as a reference material. The composites showed lower friction coefficients and higher wear resistance than the monolithic 3YTZP. An outstanding performance was achieved at 10 N, where the friction coefficient decreased from 0.6 to 0.3 and the wear rates decreased 3 orders of magnitude in comparison with the monolithic ceramic. A layer adhered to the worn surface was found for all the composites, but it did not acted as a lubricating film. The composites with the lowest GNP content showed an overall improved tribological behavior.

Magnetocaloric effect and scaling analysis in superspinglass cobalt based nanoparticles

Zelenakova, A., Hrubovcak, P., Zelenak, V., Kovac, J., Franco, V.
Physical Review B, **99** (2019) 224101
DOI: 10.1103/PhysRevB.99.224101

We have combined high sensitivity, extra-low differential temperature scanning rate calorimetry, and acoustic emission (AE) measurements to study avalanches during the cubic <-> 18R martensitic transition of a Cu-Al-Be single crystalline shape memory alloy. Both AE and calorimetry corroborate a good power-law behavior for cooling with an exponent epsilon similar or equal to 1.6. For heating, a slope is observed in the maximum likelihood curves, which confirms that our data are affected by an exponential cutoff. An effective energy exponent, epsilon similar to 1.85, and a cutoff, lambda(-1) = 0.115(38) x 10(-3) aJ, were determined by fits of power-laws with exponential damping. The long tail observed in the low-temperature region by calorimetric measurements suggests the existence of significant elastic effects that constrain the progress of the transformation at low temperatures. While thermodynamic features such as transformation enthalpy and entropy are those expected for Cu-based shape-memory alloys undergoing a cubic <-> 18R transition, the critical behavior deviates from the corresponding behavior expected from this symmetry change. These deviations are a consequence of the elastic hardening induced by the interplay of the transformation with dislocation jamming, which has the effect of effectively reducing the number of pathways connecting the parent and martensitic phase.

Scale-invariant avalanche dynamics in the temperature-driven martensitic transition of a Cu-Al-Be single crystal

Romero, FJ; Martin-Olalla, JM; Gallardo, MC; Soto-Parra, D; Salje, EKH; Vives, E; Planes, A*Applied Physics Letters,* **115** (2019) 161904
DOI: 10.1063/1.5116753

Calorimetric, elastic, and polar properties of ferrolectric lead scandium tantalate PbSc0.5Ta0.5O3 with 65% cation order have been investigated in the vicinity of the paraelectric-ferroelectric transition at T-trans = 295 K. Comparison of temperature dependencies of the excess specific heat and elastic properties indicates that both anomalies stem from thermal

fluctuations of order parameters in three dimensions. These fluctuations are consistent with the tweed microstructure. This transition is driven by several coupled thermodynamic order parameters, as evidenced by a strongly nonlinear scaling of the excess entropy with the squared ferroelectric polarization.

Scale-invariant avalanche dynamics in the temperature-driven martensitic transition of a Cu-Al-Be single crystal

Romero, FJ; Martin-Olalla, JM; Gallardo, MC; Soto-Parra, D; Salje, EKH; Vives, E; Planes, A*Applied Physics Letters,* **115** (2019) 161904
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Correlations between elastic, calorimetric, and polar properties of ferroelectric PbSc0.5Ta0.5O3 (PST)

Gan, LY, Romero, FJ, Franco, V, Martin-Olalla, JM, Gallardo, MC, Salje, EKH, Zhou, YM, Aktas, O
Applied Physics Letters, **115** (2019) 161904
DOI: 10.1063/1.5116753

Calorimetric, elastic, and polar properties of ferrolectric lead scandium tantalate PbSc0.5Ta0.5O3 with 65% cation order have been investigated in the vicinity of the paraelectric-ferroelectric transition at T-trans = 295 K. Comparison of temperature dependencies of the excess specific heat and elastic properties indicates that both anomalies stem from thermal fluctuations of order parameters in three dimensions. These fluctuations are consistent with the tweed microstructure. This transition is driven by several coupled thermodynamic order parameters, as evidenced by a strongly nonlinear scaling of the excess entropy with the squared ferroelectric polarization.

A procedure to obtain the parameters of Curie temperature distribution from thermomagnetic and magnetocaloric data

Manchon-Gordon, AF, Moreno-Ramirez, LM, Ipus, JJ, Blazquez, JS, Conde, CF, Franco, V, Conde, A
Journal of Non-crystalline Solids, **520** (2019) 119460
DOI: 10.1016/j.jnoncrysol.2019.119460

We propose a procedure for the determination of the parameters of the Curie temperature distribution (TCD) in a compositionally inhomogeneous ferromagnetic material. Assuming a Gaussian TCD and using a mean field approach based on the Brillouin function, we report that with respect to the average value of the distribution: a) both inflection point of magnetization, T-inf, and temperature at maximum magnetic entropy change curves, T-pk(MCE), shift to lower temperatures and b) temperature at maximum paramagnetic susceptibility, T-pk(chi), shifts to higher temperatures. Using these evolutions as a function of the TCD broadening and fitting them to a second order polynomial function, a self-consistent procedure to determine the parameters of the distribution is supplied. These predictions have been experimentally tested for a ball milled Fe70Zr30 amorphous alloy using thermomagnetic and magnetocaloric measurements. The obtained parameters using the proposed procedure agree with those directly measured using Mossbauer spectrometry.

Mechanical Amorphization and Recrystallization of Mn-Co(Fe)-Ge(Si) Compositions

Vidal-Crespo, A, Ipus, JJ, Blazquez, JS, Conde, A
Metals, **9** (2019) 534
DOI: 10.3390/met9050534

Mechanical alloying using a planetary ball mill allowed us to obtain two homogeneous systems formed by units with nanometer size and MnCo0.8Fe0.2Ge1-xSix stoichiometry (x = 0 and 0.5). The phase evolution of the systems with the milling time was analyzed using X-ray diffraction. Thermal stability of the final products was studied using differential scanning calorimetry. Room temperature Fe-57 Mossbauer spectroscopy was used to follow the changes in the Fe environments. A paramagnetic Co-based amorphous phase developed in both alloys as milling progressed. However, while the presence of Si stabilized the Mn-type phase, mechanical recrystallization was observed in a Si-free composition leading to the formation of a MnCo(Fe)Ge intermetallic (Pnma space group) with a crystal size of 7 +/- 1 nm. Mossbauer results indicate that Fe atoms migrate from the initial bcc phase to the amorphous and intermetallic phases.

Influence of Thermal and Magnetic History on Direct Delta T-ad Measurements of Ni49+xMn36-xIn15 Heusler Alloys

Moreno-Ramirez, Luis M., Delgado-Matarin, Antonio, Law, Jia Yan, Franco, Victorino, Conde, Alejandro, Giri, Anit K.
Metals, **9** (2019) 1144
DOI: 10.3390/met9111144

In the present work, using Heusler Ni49+xMn36-xIn15 (with x = 0 and 0.5) alloys, it is shown that the choice of the appropriate measurement protocol (erasing the prior state of the sample in between experiments) in T-ad first shot characterization is crucial for obtaining reliable results. Unlike indirect measurements, for which incorrect protocols produce overestimates of the characteristics of the material, erroneous direct measurements underestimate T-ad in the region close to its first order phase transition. The error in T-ad is found to be dependent on the temperature step used, being up to 40% underestimation, including a slight shift in its peak temperature.

Intragranular carbon nanotubes in alumina-based composites for reinforced ceramics

Esquivias, Luis, Rivero-Antunez, Pedro, Zamora-Ledezma, Camilo, Dominguez-Rodriguez, Arturo, Morales-Florez, Victor
Journal of Sol-Gel Science and Technology, **90** (2019) 162-171
DOI: 10.1007/s10971-018-4834-4

The traditional methods for the synthesis of reinforced alumina-based matrix composites with carbon nanotubes (CNTs) have presented serious difficulties for obtaining well-dispersed and homogeneously distributed CNTs within the matrix. Besides this, the CNTs are typically found in the grain boundaries of the matrix. These features involve a non-optimal reinforcement role of the CNTs. With the aim of maximizing the efficiency of the reinforcement of the CNT, this work reconsiders a sol-gel-based procedure for ceramic composite fabrication with a two fold objective: to achieve a good dispersion of the CNTs and to promote the intragranular location of the CNTs. The mixing of precursors and CNTs has been developed under the presence of high-power ultrasounds, followed by a rapid in-situ gelation that froze the nanotubes inside the gel. The chemical and physical relationships between the ceramic matrix and the embedded reinforcing phase have been researched. First results confirm the success of the synthesis procedure for the preparation of alumina-based composite powders starting from a commercial boehmite sol and multiwalled carbon nanotubes. X-ray diffraction and Raman analyses confirmed the formation of the -Al2O3 and the persistence of the non-damaged nanotube structure. N-2 physisorption and electron microscopy were used to check the evolution of the nanostructure and to confirm the presence of intragranular carbon nanotube within the polycrystalline powder. Therefore, the alumina-based composite powder prepared by this new procedure is a good candidate for the preparation of reinforced ceramic matrix composites.

FORC study of the ferromagnetic impurities in Na and K feldspars of "El Realejo" mine

Antonio Montiel-Anaya, Jose, Franco, Victorino
AIP Advances, **9** (2019) 035038
DOI: 10.1063/1.5080081

Feldspar is a Na-K-Ca-Al tectosilicate, generally poor in iron or other elements with large magnetic moments. Being the most abundant constituent minerals in Earth's crust, feldspars are technologically used in a broad variety of applications, which include glass-manufacturing, fabrication of ceramics elements, fillers in paintings, enamels, floors, etc. However, most applications require the absence (or minimization) of Fe inclusions, being this a very relevant factor that controls the price of the mineral. Typically, Fe content in the mineral produced at a mine is determined by chemical analysis, which implies an off-site test and small sampling volume. Separation of magnetic inclusions is usually made by crushing the rocks and applying a magnetic field gradient that, in combination with gravity, guides the magnetic particles out from the production line. In this work we use FORC to determine the content of the magnetic phases and show that the conventional separation methods used in the mine, which indirectly affect the final price of the product, are selective in the extraction of magnetic particles, as evidenced by the different FORC distribution of the natural rock and that of the separated particles.

La0.59Li0.24TiO3 ceramics obtained by spark plasma sintering: Electric behavior analysis

Pereira, JS, Guerrero, F, Romaguera-Barcelay, Y, Anglada-Rivera, J, Sales, JCC, Silva, RS, Zulueta, Y, Poyato, R, Gallardo, A, Almeida, A, Moreira, JA, Leyet, Y
Materials Research Express, **6** (2019) 015504
Enero, 2019 | **DOI:** 10.1088/2053-1591/aae496

This work describes the electric study of Lithium lanthanum titanate (La0.59Li0.24TiO3) ceramics performed by Complex Impedance Spectroscopy. The nanoparticle powders have been synthesized through high energy ball milling and sintered via Spark Plasma Sintering technique. The experimental impedance data have been analyzed using the equivalent circuit model, the Extended Jonscher universal law and the derivative method. From these models, we have determined the dielectric response as well as the grain and grain boundary conductivity. The samples show ionic conductivity values between 10(-5) to 10(-4) S cm(-1) in the studied temperature range, and activation energy values 0.24 eV and 0.48 eV for grain and grain boundary, respectively. These results confirm the Li+ ions mobility through the crystalline structure of the material.

Tailoring Organic-Organic Poly(vinylpyrrolidone) Microparticles and Fibers with Multiwalled Carbon Nanotubes for Reinforced Composites

Narvaez-Munoz, Christian P., Carrion-Matamoros, Luis M., Vizuete, Karla, Debut, Alexis, Arroyo, Carlos R., Guerrero, Victor, Almeida-Naranjo, Cristina E., Morales-Florez, Victor, Mowbray, Duncan J., Zamora-Ledezma, Camilo
ACS Applied Nano Materials, **90** (2019) 4302-4312
DOI: 10.1021/acsanm.9b00758

Polymeric-based microparticles and fibers are tailorable for a wide range of common industrial and biomedical applications, while multiwalled carbon nanotubes (MWCNTs) are among the most useful macromolecules based on their outstanding electronic, mechanical, and optical properties at the nanoscale. If one combines these nanostructures with various polymeric precursors, their range of potential applications becomes even greater. One of the simplest and most affordable methods for fabricating micro- and nanostructures is electrospinning. Herein we a demonstrate how MWCNTs may be used to produce tailor-made organic-organic poly(vinylpyrrolidone) (PVP) micro-particles and fibers via electrospinning by studying their structural, vibrational, rheological, and mechanical properties' dependence on their solvent (ethanol (EtOH) or dimethylformamide (DMF)) and resulting morphology. Specifically, we find clear differences in morphologies from perfectly spherical and isolated microparticles to fibers mats, or a combination of fibers with entangled beads, with solvent type and concentration. On the basis of our findings, we propose that the mechanism governing the shape and size of the particles is a competition between the solvent's surface tension, dielectric constant, and viscoelastic properties. We show, based on both our experimental results and density functional theory (DFT) calculations, that OH functionalization of the MWCNTs is essential for achieving high PVP coverages and promoting the stability of the resulting PVP/MWCNT nanocomposite. Finally, by fabricating PVP/MWCNT fiber mats, we demonstrate that low concentrations (0.01-0.1 wt %) of MWCNTs led to a qualitative improvement (similar to 250%) in the resulting mechanical properties, i.e., a reinforced composite. These results show how by controlling the

solvent's dielectric constant, surface tension, and polymer concentration, one may produce tailor-made polymeric nanomaterials in combination with other organic/inorganic nanoparticles, i.e., silver, gold, or carbon allotropes, for next-generation applications.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

International Workshop INTRACER
18 diciembre [Sevilla, España]
Victor Morales Flórez

COMUNICACIONES / COMMUNICATIONS

International Workshop on Woman in Ceramic Science
7 – 9 abril [Budapest, Hungria]

GBN/zirconia composites: a study of the effect of powder processing method and sintering technique on the microstructural, mechanical and electrical properties
C. López-Pernía, C. Muñoz-Ferreiro, C. González-Orellana, A. Morales-Rodríguez, A. Gallardo-López, R. Poyato
Poster

3YTZP ceramic composites with different 2D nanomaterials
C. Muñoz-Ferreiro, A. Morales-Rodríguez, A. Gallardo-López, R. Poyato
Poster

6th International Congress on Microscopy & Spectroscopy
16 – 20 junio [Fethiye, Turquia]

Tailoring the Microstructure and Performance of Ceramic Composites with Graphene Nano-Platelets: Electron Microscopy as a Basic Tool
Á. Gallardo-López, C. López-Pernía, C. Muñoz-Ferreiro, A. Morales-Rodríguez, F. Gutiérrez-Mora, R. Poyato
Conferencia Invitada

16th Conference and Exhibition of the European Ceramic Society | 16-ECERS
16 – 20 junio [Turin, Italia]

Hardness and fracture toughness of alumina and zirconia composites with different amounts of reduced graphene-oxide sintered by Spark Plasma Sintering
R. Cano-Crespo, P. Rivero-Antúnez, R. Moreno, D. Gómez-García, A.Domínguez-Rodríguez
Comunicación oral

Microstructure and toughening mechanisms in zirconia composites with different graphene-based nanostructures and various powder processing routines
R. Poyato, J. Castillo-Seoane, A. Morales-Rodríguez, Á. Gallardo-López
Comunicación oral

Hydrothermal degradation in zirconia composites with different bidimensional graphene-based nanostructures
A. Morales-Rodríguez, C. Muñoz-Ferreiro, C. González-Orellana, R. Poyato, Á. Gallardo-López
Poster

X Iberian Conference on Tribology / XI Iberian Vacuum Conference | IBERTRIVA 2019
26 – 28 junio [Sevilla, España]

Relationship between the scratch and wear behavior of 3Y-TZP reinforced with graphene nanoplatelets
F. Gutiérrez-Mora, A. Gallardo-López, C. Muñoz-Ferreiro, A. Muñoz, J.C. Sánchez-López, R. Poyato
Comunicación oral

Scratch and wear behavior of isotropic zirconia matrix composites with homogenously distributed graphene nanoplatelets
C. Muñoz-Ferreiro, R. Poyato, A. Gallardo-López, M. Fides, P. Hvizdos, F. Gutiérrez-Mora
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

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

I Congreso de Estudiantes de Física e Ingeniería de Materiales de la Universidad de Sevilla (CEFIMUS)
7 marzo [Sevilla, España]
Ángela Gallardo López

COMUNICACIONES / COMMUNICATIONS

I Congreso de Estudiantes de Física e Ingeniería de Materiales de la Universidad de Sevilla (CEFIMUS)
7 marzo [Sevilla, España]

Estudio del procesado de compuestos de circona con nanoláminas de grafeno
C. Muñoz-Ferreiro*, R. Poyato, A. Morales-Rodríguez, Á. Gallardo-López
Comunicación oral

El grafeno en la degradación hidrotermal de la 3Y-TZP
Antonio A. Pérez-García*, C. González-Orellana, C. Muñoz-Ferreiro, R. Poyato, Á. Gallardo-López. A. Morales-Rodríguez
Poster

Preparación de compuestos de cerámica/grafeno mediante diferentes técnicas de sinterización
C. López-Pernía*, Á. Gallardo-López, A. Morales-Rodríguez, R. Poyato
Poster

Obtención de nanoláminas de grafeno a partir de grafito para su uso en compuestos cerámicos
R. Verdugo*, C. Muñoz-Ferreiro, Á. Gallardo-López, R. Poyato
Poster

I Jornada de Jóvenes Investigadores en Cerámica y Vidrio de Sevilla
20 marzo [Sevilla, España]

Efecto de la técnica de sinterización sobre las propiedades eléctricas de los compuestos de 3YTZP/GNP
C. López-Pernía*, R. Poyato, A. Morales-Rodríguez, Á. Gallardo-López
Comunicación oral

Compuestos de circona con nanoláminas de grafeno: optimización del procesado y estudio de propagación de fisuras.
C. Muñoz-Ferreiro*, R. Poyato, A. Morales-Rodríguez, Á. Gallardo-López
Comunicación oral

Estudio de la adición de estructuras bidimensionales en la degradación hidrotermal de la 3YTZP
Antonio A. Pérez-García*, C. Muñoz-Ferreiro, C. González-Orellana, R. Poyato, A. Morales-Rodríguez, A. Gallardo-López
Poster

Nanoláminas de grafeno a partir de la exfoliación electroquímica de una barra de grafito
M.R. Verdugo*, C. Muñoz-Ferreiro, A. Gallardo-López, R. Poyato
Poster

VIII Jornadas de Jóvenes Investigadores del Instituto de Cerámica y Vidrio (ICV-CSIC)
10 julio [Madrid, España]

Efecto de la técnica de sinterización sobre las propiedades eléctricas de los compuestos de 3YTZP/GNP
C. López-Pernía*, R. Poyato, A. Morales-Rodríguez, Á. Gallardo-López
Conferencia Invitada

30th International Conference on Diamond and Carbon Materials
8 – 12 septiembre [Sevilla, España]

Synthesizing graphene based nanomaterials for zirconia matrix composites
C. Muñoz-Ferreiro, R. Verdugo-Manzanares, A. Gallardo-López, A. Morales-Rodríguez, R. Poyato
Poster

Electrical conductivity of in-situ reduced graphene oxide-zirconia composites
C. López-Pernía, P. Luna, C. Muñoz-Ferreiro, A. Morales-Rodríguez, Á. Gallardo-López, R. Poyato
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Preparación y caracterización estructural y mecánica de andamios basados en alúmina para ingeniería tisular ósea**
Autor:	Yeray Pascual Rodríguez
Directores:	Victor Morales Flórez
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2019

DOCENCIA / TEACHING

Máster Profesorado de ESO y Bachillerato, FP
Complementos de formación disciplinar en Física y Química
Dr. Javier S. Blázquez
Lugar: Universidad de Sevilla

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Premio Extraordinario Fin de Estudios a **Carmen Muñoz Ferreiro**. Máster Universitario en Ciencias y Tecnologías de Nuevos - Curso 2017/2018
Premio Extraordinario Fin de Estudios a **Antonio Vidal Crespo**. Grado en Física y Grado en Ingeniería de Materiales - Curso 2017/2018
2ª Premio a la mejor comunicación oral a Cristina López Pernía en la I Jornada de Jóvenes Investigadores en Cerámica y Vidrio de Sevilla (España). 2019

Premio de la Facultad de Física (Universidad de Sevilla), al artículo de mes:

Scale-invariant avalanche dynamics in the temperature-driven martensitic transition of a Cu-Al-Be single crystal
Francisco Javier Romero, José-María Martín-Olalla, María Carmen Gallardo, Daniel Soto-Parra, Ekhard K. H. Salje, Eduard Vives, and Antoni Planes
Premio de la Facultad de Física al artículo del mes, junio 2019.

Microstructure, interfaces and properties of 3YTZP ceramic composites with 10 and 20 vol% different graphene-based nanostructures as fillers
Munoz-Ferreiro, C, Morales-Rodriguez, A, Rojas, TC, Jimenez-Pique, E, Lopez-Pernia, C, Poyato, R, Gallardo-Lopez, A

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS
PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Universidad Yachay-Tech		
Ecuador	**Dr. Camilo Zamora Ledezma**	3/6/2019 al 3/8/2019
Université de Montpellier		
Francia	**Prof. Eric Anglaret**	17/12/2019 al 20/12/2019

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/cm2 y campo eléctrico hasta 800V/cm
- Calorímetro de conducción, rango 80-400K, campo eléctricos 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia

Servicios Generales
General Services

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 momentam. 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 Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel 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 Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA ÓPTICA EN EL RANGO VIOLETA, VISIBLE E INFRARROJO CERCANO / ULTRAVIOLET-VISIBLE 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 Spectroscopy (UV-Vis) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
There are equipments in the laboratory, which work in the wavelength range of 190 nm to 900 nm. It can operate in the Transmission mode or in Diffuse Reflectance Modes.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- SHIMADZU UV-2101 PC
  SHIMADZU UV-2101 PC
- 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 Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA DE EMISIÓN ATÓMICA / ATOMIC EMISSION SPECTROSCOPY

La espectroscopía 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.
Inductively coupled plasma atomic emission spectroscopy (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.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)
  iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)

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. Gerado 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 1500ºC, 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 1500ºC, 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 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

Responsable Científico/ Scientific Responsible: Dr. Luis A. Pérez Maqueda
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

TAMAÑO DE PARTÍCULAS Y POTENCIAL Z / PARTICLE SIZE AND Z POTENTIAL ANALYSIS

Se dispone de las técnicas de Dispersión Dinámica de Luz y de Difracción Láser (LD), que permiten determinar la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos que van de 3 a 3000 nanómetros (DLS) o de 0.05 a 900 micras (LD).

Así mismo, se dispone de la técnica de Análisis de Movilidad Electroforética para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico).

Dynamic light scattering (DLS) and Laser diffraction (LD) are available for the determination of particle size distributions of colloidal systems (dispersed in aqueous or organic dispersions solutions) in the range 3-3000 nanometers (DLS) and 0.05-900 microns (LD).

Electrophoretic mobility measurements can be also performed for the evaluation of Z potential in colloidal systems (aqueous or organic dispersions).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Malvern modelo Zetamaster (DLS) y MalvernSizer (LD)
  Malvern model Zetamaster (DLS) and MalvernSizer (LD)

Responsable Científico/ Scientific Responsible: Dr. Manuel Ocaña Jurado
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" (ver sección abajo)
  Additional equipment in the "electron microscopy samples preparation laboratory" (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
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 selecta y microscopía electrónica de alta resolución así como análisis elemental de area selecta. 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” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (cicCartuja) y 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 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 mas avanzados, como pueden ser:

- Seguir las transformaciones de fase "in situ" provocadas por calentamientos en atmósfera inerte (vacio, 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 ºC) ANTON PAAR HTK 1200
  Diffractometer PHILIPS X'PERT PRO with high temperature chamber (1200ºC) 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: D. 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<800ºC) 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 (< 800C) 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 ºC, 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 800ºC (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 < 150ºC). 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 < 150ºC). 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/ MECHANIZED 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. Ofreciendo 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 y D. Manuel Perea Domínguez

Actividades Divulgativas y Formativas

Outreach and Teaching Activities

DOCENCIA / TEACHING

MÁSTER / MASTER

MÁSTER EN CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES

Organizado por la Universidad de Sevilla
Coordinador: Dra. Regla Ayala Espinar
Fecha de Celebración: Curso Académico 2018-19
Créditos necesarios: 60
Dirigido a: Licenciados 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
http://masteroficial.us.es/materiales/

Aplicaciones Tecnológicas de Materiales Funcionales (Créditos: 5) | Dr. Victorino Franco García
Catalizadores para la Energía y el Medio Ambiente (Créditos: 5) | Dr. Alfonso Caballero Martínez
Corrosión y Recubrimientos Protectores (Créditos: 5) | Dr. Leidy Marcela Martínez Tejada | Antonio Paul Escolano (US)
Fisica del Estado Sólido (Créditos: 5) | Dra. Angela Gallardo López | Dr. Manuel Jiménez Melendo
Materiales con Funcionalidad Superficial (Créditos: 5) | Dr. Luis Bobadilla Baladrón| Dra. Leidy Marcela Martínez Tejada
Modelización Aplicada a la Caracterización Estructural de Medios Condensados (Créditos: 5) | Dr. Carlos López Cartes (US)| Dra. Regla Ayala Espinar
Propiedades Magnéticas de Materiales (Créditos: 5) | Dr. Vctorino Franco García
Procesado de Materiales Estructurales (Créditos: 5) | Dr. Felipe Gutiérrez Mora | Dr. José Antonio Rodríguez Ortíz (US)
Propiedades Térmicas, Dieléctricas y Ópticas (Créditos: 5) | Dr. Francisco J. Romero Landa| Dra. María del Carmen Gallardo Cruz
Química del Estado Sólido (Créditos: 5) | Dr. Antonio Parejón Pazo | Dr. José Manuel Córoba Gallego
Recuperación y Transformación de Materiales (Créditos: 5) | Dr. Antoni Perejón Pazo | Dr. Leidy Marcela Martínez Tejada | Dra. Svetlana Ivanova
Síntesis de Materiales y Nanoestructuras (Créditos: 7) | Dra. María Dolores Alcalá | Dr. José Manuel Córdoba Gallego | Dra. Svetlana Ivanova
Técnicas de Caracterización de Materiales (Créditos: 8) | Dra. Ana Morales Rodríguez |Dra. Anna Dimitrova Penkova | Dr. Joaquín Ramírez Rico | Dr. Juan Manuel Montes Martos (US)
Trabajo Fin de Máster (Créditos: 10) | Dra. Ana Morales Rodríguez | Dr. Diego Gómez García | Dr. Felipe Gutiérrez Mora | Dr. Javier Blázquez Gámez | Dr. Jhon Jairo Ipus Bados | Dr. Joaquín Ramírez Rico | Dr. Víctor Morales Flórez

MÁSTER INTERUNIVERSITARIO "LÁSER, PLASMA Y TECNOLOGÍA DE SUPERFICIES"

Organizado por la Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Sevilla y el Instituto de Ciencia de Materiales de Madrid

Fecha de Celebración: Curso Académico 2018-19
Créditos necesarios: 60
Dirigido a: Licenciados 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

Profesorado ICMS

Principios Básicos
Interaccion de partículas y radiación con la materia. Láseres
Asunción Fernández Camacho
Materiales e Ingeniería de Superficies
Ana Isabel Becerro Nieto, Alfonso Caballero Martínez, Agustín R. González-Elipe, Francisco Yubero Valencia

Tecnologias
Plasmas y Tecnología de superficies
Alberto Palmero, José Cotrino Bautista
Tecnologías de lámina delgada
Ana Isabel Borrás Martos, Agustín R. González-Elipe, María del Carmen López Santos
Técnicas de caracterización de superficies y láminas delgadas
T. Cristina Rojas Ruiz, Francisco Yubero Valencia

Aplicaciones
Nanotecnología de superficies y sus aplicaciones
Angel Barranco Quero, Juan Ramón Sánchez Valencia
Nuevos materiales para dispositivos (fotónicos, electrónicos, magnéticos y aprovechamiento Energ)
Ana Isabel Borrás Martos
Funcionalización de superficies para aplicaciones mecánicas, protectoras y de bioactividad controladas
Juan Carlos Sánchez-López, María Carmen López Santos, 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 ha impartido cursos regulares de primer y segundo ciclos de las correspondientes Licenciaturas y/o Grados en Química, Física y Farmacia e Ingeniería de Materiales y Biología en la Universidad de Sevilla.

CURSOS / COURSES

ÚLTIMOS AVANCES EN MICRO-NANOESTRUCTURACIÓN DE SUPERFICIES
PATTERNING AND STRUCTURATION OF SURFACES

Organizado por el Instituto de Ciencia de Materiales de Sevilla
Director : Isabel Montero, ICMM
Co-directores: Juan Carlos Sánchez y Agustín R. González-Elipe
Fecha de Celebración: 25 de junio de 2019
Créditos Europeos: 2.5 ETCS
Dirigido a: Estudiantes de C. Físicas, Químicas, y de estudios técnicos: Ingenieros, Arquitectos.
Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.

CONFERENCIAS INVITADAS IMPARTIDAS POR PERSONAL DEL ICMS
INVITED CONFERENCES BY PERSONNEL OF THE ICMS

23 de julio 2019 | **Mecanismos y fuentes de financiación para la cooperación de la PUJ con la Universidad de Sevilla**
Miguel Angel Centeno Gallego
Lugar: Departamento de Química de la Facultad de Ciencias de la Pontificia Universidad Javeriana (Colombia)

23 de julio 2019 | **Aspectos claves para la movilidad y generación de cotutorias entre España y Colombia**
Miguel Angel Centeno Gallego
Lugar: Departamento de Química de la Facultad de Ciencias de la Pontificia Universidad Javeriana (Colombia)

24 de julio 2019 | **Aplicaciones de la técnica de infrarrojo en Catálisis**
Miguel Angel Centeno Gallego
Lugar: Departamento de Química de la Facultad de Ciencias de la Pontificia Universidad Javeriana (Colombia)

8 de octubre 2019 | **Ingeniería en la nanoescala: aplicaciones al diseño de recubrimientos funcionales**
Juan Carlos Sánchez López
Lugar: Instituto de Investigaciones en Materiales, Universidad Nacional Autonoma de Mexico

9 de octubre 2019 | **Structured Catalytic Systems for Biofuels Production**
José Antonio Odriozola Gordón
Lugar: Huanzhong University of Science and Technology, Wuhan (China)

16 de octubre 2019 | **Microchannel reactors in Catalysis**
José Antonio Odriozola Gordón
Lugar: Huanzhong University of Science and Technology, Wuhan (China)

23 de octubre 2019 | **Nanocatalysis: overruling the particle size effect**
José Antonio Odriozola Gordón
Lugar: Huanzhong University of Science and Technology, Wuhan (China)

1 de noviembre 2019 | **Overruling the size effect of nanocatalysts:the role of the interface**
José Antonio Odriozola Gordón
Lugar: Beijing Forestry University, Beijing (China)

CONFERENCIAS Y SEMINARIOS IMPARTIDOS EN EL ICMS
CONFERENCES AND SEMINAR IN THE ICMS

4 de abril | **Descubrimiento y Comercialización de una Nueva Familia de Catalizadores**
Dr. Javier García Martínez
Catedrático de Química Inorgánica de la Universidad de Alicante y Director del Laboratorio de Nanotecnología Molecular (NANOMOL)

El pequeño tamaño de los microporos de las zeolitas dificulta la entrada y salida de las moléculas voluminosas al interior de su estructura; lo que afecta negativamente a la actividad y a la selectividad de un buen número de catalizadores industriales que contienen zeolitas, como por ejemplo, los catalizadores de craqueo catalítico (FCC). Para solucionar esta limitación, hemos desarrollado una nueva estrategia que involucra el tratamiento de zeolitas en disoluciones de surfactantes catiónicos en la presencia de una base. Este proceso post-sintético permite introducir, de forma sencilla y controlable, la mesoporosidad en una gran variedad de zeolitas, a la vez que éstas mantienen sus propiedades más sobresalientes, tales como cristalinidad, estabilidad hidrotermal y actividad catalítica. Este método permite solventar las limitaciones de otros materiales mesoporosos preparados con surfactantes, tales como MCM-41 y otros similares, que carecen precisamente de las propiedades que acabamos de señalar. La presencia de mesoporosidad dentro de la estructura de la zeolita se ha confirmado mediante un amplio conjunto de técnicas de caracterización avanzadas.

Tras el estudio de la actividad y selectividad catalítica de estas zeolitas mesoporosas a escala laboratorio, se procedió a escalar la tecnología produciendo varios kilos de catalizador FCC en una planta piloto. Durante la presentación se mostrarán los resultados catalíticos del craqueo de gasóleo

de vacío obtenidos en una unidad de ACE (Advanced Catalytic Evaluation). Las zeolitas mesoporosas que aquí se presentan son una realidad comercial gracias al acuerdo entre la empresa Rive Technology y Grace. Hoy en día estos catalizadores se están utilizando con éxito en varias refinerías de EEUU. En la parte final de la presentación se mostrarán algunos resultados obtenidos en la refinería Alon Big Spring de Texas, EEUU, en el que se confirman los resultados obtenidos a escala piloto. Como era de esperar, la presencia de mesoporosidad en la zeolita incrementa el número de barriles procesados y la producción de gasolina y diésel, a la vez que se reduce notablemente la generación de CO2 y de gases.

2 de diciembre | **Applications of infrared microspectroscopy using the synchrotron radiation of Alba light source**
Dr. Ibraheem Yousef
MIRAS Beamline Responsible - Experiments Division in ALBA Synchrotron

Conferencia dentro del PROGRAMA DE DOCTORADO «CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES"

Ciclo de Conferencias / ICMS Invited Lectures

18 de marzo | **Flash Sintering of Ceramics**
Prof. Rishi Raj
Universidad de Colorado en Boulder

Flash sintering is a novel densification technology for ceramics, which allows a dramatic reduction of processing time and temperature. It represents a promising sintering route to reduce economic, energetic and environmental costs associated to firing. Moreover, it allows to develop peculiar and out-of-equilibrium microstructures. The flash process is complex and unusual, including different simultaneous physical and chemical phenomena and their understanding, explanation and implementation require an interdisciplinary approach from physics, to chemistry and engineering. In spite of the intensive work of several researchers, there is still a wide debate as for the predominant mechanisms responsible for flash sintering process. This talk will include an overview of the original research that led to the discovery of the technique by Prof. Raj, as well as an analysis of the most significant mechanisms proposed for explaining the "flash" event. It will also include future scientific activities and potential technological implementations.

4 de julio | **Colour Engineering: from nature to applications**
Dr. Silvia Vignolini
Department of Chemistry, University of Cambridge

The most brilliant colours in nature are obtained by structuring transparent materials on the scale of the wavelength of visible light. By controlling/designing the dimensions of such nanostructures, it is possible to achieve extremely intense colourations over the entire visible spectrum without using pigments or colorants. Colour obtained through structure, namely structural colour, is widespread in the animal and plant kingdom [1]. Such natural photonic nanostructures are generally synthesised in ambient conditions using a limited range of biopolymers. Given these limitations, an amazing range of

optical structures exists: from very ordered photonic structures [2], to partially disordered [3], to completely random ones [4].
In this seminar, I will introduce some striking example of natural photonic structures [2-4] and review our recent advances to fabricate bio-mimetic photonic structures using the same material as nature. Biomimetic with cellulose-based architectures enables us to fabricate novel photonic structures using low cost materials in ambient conditions [6-7]. Importantly, it also allows us to understand the biological processes at work during the growth of these structures in plants.

[1] Kinoshita, S. et al. (2008). Physics of structural colors. Rep. Prog. Phys. 71(7), 076401.
[2] Vignolini, S. et al. (2012). Pointillist structural color in Pollia fruit. PNAS 109, 15712-15716.
[3] Moyroud, E. et al. (2017). Disorder in convergent floral nanostructures enhances signalling to bees. Nature 550, 469.
[4] Burresi M. et al. (2014) Bright-White Beetle Scales Optimise Multiple Scattering of Light. Sci.Rep. 4, 727
[5] Parker R. et al. (2018) The Self-Assembly of Cellulose Nanocrystals: Hierarchical Design of Visual Appearance. Adv Mat 30, 1704477
[6] Parker R. et al. (2016). Hierarchical Self-Assembly of Cellulose Nanocrystals in a Confined Geometry. ACS Nano, 10 (9), 8443–8449
[7] Liang H-L. et al. (2018). Roll-to-roll fabrication of touch-responsive cellulose photonic laminates, Nat Com 9, 4632

24 de octubre | **Multi Photon Phosphors**
Dr. Andries Meijerink
Utrecht University

Lanthanides have transformed the world of lighting in the past 40 years. Presently, almost all artificial light sources rely on emission of light by lanthanide ions. In many luminescent materials, also known as phosphors, one-to-one photon conversion downshifts one high energy photon to one lower energy photon in the desired spectral region. However, recently, there is a significant increase of attention for multi-photon phosphors relying on multi-photon conversion processes, either upconversion or downconversion. Insight in the multi-photon processes is not trivial but is needed to understand the mechanism and improve the efficiency of spectral conversion processes in multi-photon phosphors which is crucial for applications, including solar cells to reduce spectral mismatch losses.

In this presentation a short historical introduction to single- and multi-photon conversion phosphors will be followed by an overview of recent developments of efficient up- and downconversion materials. Next it will be discussed how insight can be obtained in the mechanism and efficiency of up- and downconversion processes. An important aspect involves modelling of energy transfer and ligand quenching. For both up- and downconversion examples will be given on how modelling of

luminescence decay curves can provide quantitative insight. A new ligand-quenching model will be presented and applied to understand multi-phonon vibrational quenching in NaYF4:Er,Yb upconversion nanocrystals. Finally a new method will be presented that provides direct proof for downconversion. Correlated emission of photons in photon cutting materials can serve as a fingerprint for the occurrence of downconversion and can even be used to quantify the downconversion efficiency.

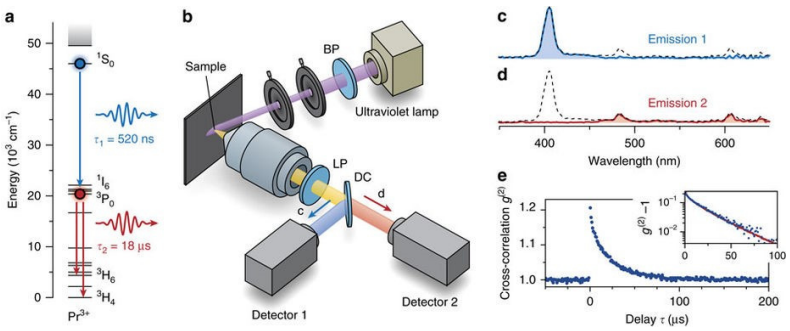

Figure 1 – Illustration correlated photon counting to demonstrate two-photon emission using NaLaF4:Pr3+ as model system. (a) Two-photon emission on Pr3+. (b) Schematic set-up for correlated photon counting (c, d) Emission of Pr3+ in blue and red spectral region detected by separate detectors. (e) Correlated photon-counting signal.

7 de noviembre | **Nanoscience and Surface Science for studying the Cosmos**

Dr. José Ángel Martín Gago
Instituto de Ciencia de Materiales de Madrid (ICMM-CSIC)

Evolved stars are a factory of chemical complexity, gas and dust, which contribute to the building blocks of planets and life. However, the dust formation process remains poorly understood. Different laboratory techniques are used to produce analogs of cosmic dust being based the majority of them on uncontrolled combustion or plasma decomposition of molecular precursors in conditions far removed from those in star photospheres.

We have designed and built an unprecedented ultra-high vacuum machine combining gas aggregation sources with in-situ advanced surface science characterization techniques (as STM, XPS or IRAS) and mass spectroscopy [2.] We show that astrochemical problems can be successfully addressed and understood using surface science methodologies. This combination opens the door to the investigation and modelling of processes related to dust particles and its interaction with the gas in different regions of the universe.

Different model systems will be presented. In the first analogues of cosmic dust are analysed to conclude that evolved stars do not form aromatics or fullerenes but aliphatic material made up by bottom-up coalescence[3]. In the second example we propose a new route for the formation of polycyclic aromatic hydrocarbons produced by etching of graphene on the SiC grains in a top-down process[1].

References

[1] Merino P., et al. Nature Communications 5, (2014), 3054
[2] L. Martinez et. Al. Sci Rep. 8 (2018) 7250
[3] L. Martinez, Nat. Astronomy, in press.

12 de diciembre | **Spectroscopic studies of dispersion and orientation of carbon nanotubes and graphene in aqueous inks and related nanomaterials**
Dr. Eric Anglaret
Laboratorie Charles Coulomb, CNRS, Univeristé de Montpellier, Francia

Processing single-walled carbon nanotubes (SWNT) and single-layer graphene (SLG) into thin films or composite nanomaterials are key issues to take advantage of their mechanical, electrical and optical properties. In this talk, we will review our recent results, and primarily optical spectroscopy studies (absorption, Raman and photoluminescence), of the dispersion and orientation of SWNT and SLG in aqueous inks [1-10] as well as the properties of liquid crystals [1-3], thin films [3-5] or composites [6-7] prepared from these inks.

[1] Dispersion and orientation of single walled carbon nanotubes in a chromonic liquid crystal, N. Ould-Moussa et al, Liq. Cryst. 40, 1 (2013)
[2] Liquid crystals of carbon nanotubes and graphene, C. Zakri et al, Phil. Trans. Royal Soc. (2013)
[3] Anisotropic thin films of single wall carbon nanotubes from aligned lyotropic nematic suspensions, C. Zamora-Ledezma et al, Nanoletters 8, 4103 (2008)
[4] Conductivity anisotropy of assembled and oriented carbon nanotubes, C. Zamora-Ledezma et al, Phys. Rev. E 85, 062701(5) (2012)
[5] Morphology and anisotropy of thin conductive inkjet printed lines of single-walled carbon nanotubes, F. Torres-Canas et al, Mat. Res. Exp. 4 (2017)
[6] Orientational order of carbon nanotubes in stretch-aligned photoluminescent composites, C. Zamora-Ledezma, et al, Phys. Rev. B 80, 113407 (2009)
[7] Dispersion and individualization of SWNT in surfactant-free suspensions and composites of hydrosoluble polymers, F. Torres-Canas et al, J. Phys. Chem. C 119, 703 (2015)
[8] Surfactant-free single-layer graphene in water, G. Bepete et al, Nat. Chem. (2016)
[9] Raman Signatures of Single Layer Graphene Dispersed in Degassed Water, "Eau de Graphene", G. Bepete et al, J. Phys. Chem. C 120, 28204 (2016)
[10] Hydroxide Ions Stabilize Open Carbon Nanotubes in Degassed Water, G. Bepete et al, ACS Nano 12, 8606 (2018)

ICMS - sci - talks

22 de enero | **Influence of Irrigation Conditions in the Germination of Plasma Treated Nasturtium Seeds**
Dr. Agustín R. González-Elipe

6 de febrero | **Unravelling the Role of Oxygen Vacancies in the Mechanism of the Reverse Water–Gas Shift Reaction by Operando DRIFTS and Ultraviolet–Visible Spectroscopy**
Dr. Luis F. Bobadilla Baladrón

12 de marzo | **Microstructure, Interfaces and Properties of 3YTZP Ceramic Composites with 10 and 20 vol% Different Graphene-based Nanostructures as Fillers**
Dra. Angela Gallardo López

2 de abril | **Laser-induced Coloration of Ceramic Tiles Covered with Magnetron Sputtered Precursor Layers**
Dr. Victor J. Rico Gavira

21 de mayo | **Selective CO Methanation with Structured RuO2/Al2O3 Catalysts**
Dr. Miguel Angel Centeno Gallego

18 de junio | **Self-Assembly of the Nonplanar Fe(III) Phthalocyanine Small-Molecule: Unraveling the Impact on the Magnetic Properties of Organic Nanowires**
Dr. Victor López Flores

17 de septiembre | **Antibacterial Nanostructured Ti Coatings by Magnetron Sputtering: From Laboratory Scales to Industrial Reactors**
Dr. Alberto Palmero Acebedo

8 de octubre | **Phase-pure $BiFeO_3$ produced by reaction flash-sintering of Bi_2O_3 and Fe_2O_3**
Dr. Pedro Enrique Sánchez Jiménez

22 de octubre | **Hydrophobicity, freezing delay, and morphology of laser-treated aluminium surfaces**
Dra. M. Carmen López Santos

5 de noviembre | **Understanding the role of the acid sites in 5 Hydroxumethylfurfural oxidation to 2,5-furandicarboxylic acid reaction over gold catalysts: surface investigation on CexZr1-xO2 compounds**
Dra. Anna Dimitrova Penkova

19 de noviembre | **Amber imitation? Two unusual cases of pinus resin-coated beads in iberian late prehistory (3rd and 2nd millenia BC)**
Dr. José María Martínez Blanes

10 de diciembre | **Bacterial behavior on coated porous titanium substrates for biomedical applications**
Dra. Ana M. Beltrán Custodio

DIVULGACIÓN / DISSEMINATION

FERIA DE LA CIENCIA / FAIR OF SCIENCE

La 17ª Feria de la Ciencia de Sevilla (del 16 al 18 de mayo de 2019, Palacio de Congreso y Exposiciones de Sevilla, FIBES) 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, el Instituto de Ciencia de Materiales de Sevilla presentó el stand con título: "Energías y Eficiencia Energética".

Coordinadora: Dra. T. Cristina Rojas Ruiz
Gerardo Colon, Nuria Núñez, Jose Manuel Obrero, María del Carmen Hidalgo, Rosalía Poyato, Paula de Navascués, Reyes Verdugo, Encarna Arrollo, Ana Isabel Becerro, Beatriz Sarrión, Carmen Muñoz, Cristina López, Andrea Rubino, Laura Calió, Victoria Esteso, Jose Maria Miranda, Mauricio Calvo, Carmen Gutierrez, Javier Castillo, Francisco Jesus Platero, Lucia Fernández, Jaume Carreras, Xabier García, Alma Garcia, Laura Blandon, Hernan Míguez, Elena Cabello

The Fair of Science (16 to 18 May 2019, in Seville) constituted 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. The Materials Science Institute of Seville presented the activity: "Energies and Energy Efficiency"

Coordinator: Dra. T. Cristina Rojas Ruiz
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SEMANA DE LA CIENCIA Y LA TECNOLOGÍA / SCIENCE AND TECHNOLOGY WEEK

La semana de la Ciencia (celebrado del 12-22 de noviembre de 2019) es unevento 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 compartirán un desayuno 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. Gerardo Colon Ibañez. Tema “Catálisis: soluciones frente al cambio climático”

Coordinadora: Dra. T. Cristina Rojas Ruiz

Cuenta la ciencia

Cuenta la ciencia es una actividad que pretende acercar y fomentar la cultura científica a los ciudadanos de distintas edades de pueblos y barrios de la provincia de Sevilla, que por su localización pueden presentar más dificultades de acceso a la oferta de actividades de divulgación científica. También se pretende despertar la vocación científica en los niños y jóvenes, así como transmitir la importancia y el papel del profesional científico en el desarrollo de la sociedad del bienestar. Para conseguir estos objetivos, investigadores del Instituto se desplazaron hacia los centros culturales para realizar las siguientes actividades:

Centro Cultural Biblioteca de Montequinto

Charla

El cambio de paradigma energético: Energia solar. Dirigido a estudiantes de bachillerato. Dr. Gabriel Lozano.

Talleres de ciencia
Ciencia divertida. Dirigido a niños de primaria.
Dr. Luis F. Bobadilla.

Fabricación de una celda solar de colorante. Dirigido a estudiantes de bachillerato. Mauricio Calvo, Andrea Rubino, Laura Calió y Carlos Romero Perez.

Desayunos–Científicos
Materiales fotonicos: Nanotecnologías para el control de la luz. Dirigido a estudiantes de bachillerato. Dr Gabriel Lozano.

Catálisis: Soluciones frente al Cambio climático. Dr. Gerardo Colon Ibañez

Biblioteca de San Jerónimo

Taller de ciencia
Para niños de segundo ciclo de primaria.
¿Qué es un científico?, ¿Qué es la Nanotecnología?. El plasma, cuarto estado de la materia. Paula Navascués, Javier Castillo, Xavier Garcia, Jose Manuel Obrero y Ester López.

Desayuno –Científico
Catálisis: Soluciones frente al Cambio climático. Dirigido a estudiantes de 4 ESO y bachillerato. Dr. Gerardo Colon Ibañez

Charla–Coloquio

Materiales fotónicos: Luz y Nanomateriales, un encuentro brillante. Dirigido a estudiantes de bachillerato. Dr. Mauricio Calvo

Organizadora y coordinadora: Dra. T. Cristina Rojas Ruiz

PARTICIPACION EN LA NOCHE EUROPEA / EUROPEAN RESEARCHERS' NIGHT

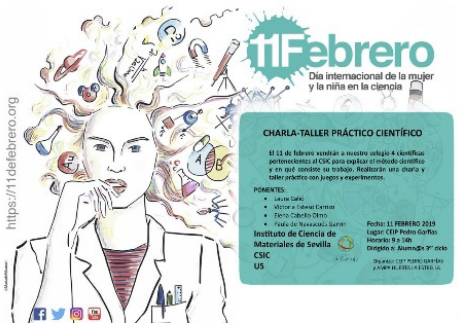
La Noche Europea de los Investigadores, celebrada en Sevilla 27 septiembre de 2019, es un proyecto de divulgación científica enmarcado en Horizonte 2020, bajo las acciones Marie Skłodowska-Curie. Su principal objetivo es acercar los investigadores a los ciudadanos para que conozcan su trabajo, los beneficios 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: Nanomateriales para controlar la luz. Elena Cabello

Charla-taller: Diamante, grafito y grafeno ¿Son familia?. Dra. Rosalía Poyato, Cristina Lopez y Carmen Muñoz

PARTICIPACION EN EL DÍA INTERNACIONAL DE LA MUJER Y LA NIÑA EN LA CIENCIA/ INTERNATIONAL WOMEN AND GIRL'S DAY IN SCIENCE



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 dia 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.

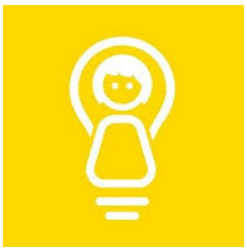
Charlas - taller: Mujer + científica + Grafeno: Dra. Rosalía Poyato, Carmen Muñoz y Cristina López.

Charlas: Viaje al interior de los Nanomateriales a través de la microscopia electrónica. Dra. T. Cristina Rojas.

Charla-taller práctico: Grupo de nanotecnología de superficies.

Video promocional: Ana Isabel Becerro Nieto (Investigadora invitada)

PARTICIPACION EN "LAS NIÑAS EN REDIRIS". REDIRIS Y LA FUNDACIÓN INSPIRING GIRLS



Este acto se enmarca dentro de la iniciativa "Mujeres en RedIRIS", que trata de visibilizar a las mujeres que forman parte de la red y que están además en contacto permanente con el mundo de la ciencia en sus respectivos centros.

El objetivo de la Fundación Inspiring Girls es mostrar a las niñas distintas salidas profesionales y trabajos en un entorno diferente, y por supuesto, que pasaran una mañana divertida, instructiva y dinámica, rodeadas de mujeres inspiradoras, cuyo ejemplo puede ayudarles a aumentar sus aspiraciones y a apuntar alto en su desarrollo profesional. En está actividad han participado científicas y becarias del ICMS.

Charla invitada: Las niñas y la ciencia. Laura Calió. Museo Casa de la Ciencia –CSIC, Sevilla.
20 febrero

Charla-taller para alumnas del CEIP Federico García Lorca. Laura Calió, Nuria Ofelia Núñez Alvarez. Rectorado de la Universidad de Sevilla.
28 mayo

Charla-Taller Práctico Científico. Laura Calió, Victoria Esteso Carrizo, Elena Cabello Olmo, Paula de Navascués Garvín.CEIP PEDRO GARFÍA, Sevilla.
11 febrero

PARTICIPACION EN FESTIVAL DE NANOCIENCÍA Y NANOTECNOLOGÍA 10 A LA MENOS 9 / NANOSCIENCE AND NANOTCHNOLOGY 10 A LA MENOS 9

El IV Festival de Nanociencia, celebrado del 8 al 12 abril del 2019 en el ICMS, 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. Para ello en el ICMS se realizaron las siguientes actividades, dirigidas a alumnos de 4 de la ESO y de 1 y 2 de bachillerato:

Nanoseminarios

¡Qué grande es lo NANO!. Dr. Juan Ramon Sanchez Valencia

Luz y Nanomateriales, un encuentro brillante. Dr. Mauricio Calvo Roggiani

Nanodiálogos

Revoluciones Tecnológicas en el Siglo XXI: Nano medicina e Inteligencia Artificial. Dr. Alberto Palmero

Nanociencia o el arte de "cocinar" materiales "a la carta ". Dr. Juan Carlos Sanchez López

Visitas guiadas

Como fabricamos y vemos los nanomateriales: Visita a los laboratorios de preparación de nanomateriales y a los microscopios electrónicos.

Exposicion

Exposición de fotografías: “Nanomateriales vistos a través del microscopio electrónico”

Organizadora y coordinadora: Dra. T. Cristina Rojas Ruiz

PARTICIPACION EN EL SEGUNDO SALÓN DE LA FORMACIÓN DE LA RINCONADA

6 de marzo. Charla a cargo del Dr. Mauricio Calvo

PARTICIPACION EN EL V ENCUENTRO NACIONAL DE NANODIVULGADORES

26 de abril (Madrid). T. Cristina Rojas

PARTICIPACION EN EL I ENCUENTRO DE COMUNICADORES/AS Y DIVULGADORES/AS DEL CSIC ANDALUCIA Y EXTREMADURA.

27 y 28 noviembre (Sevilla).

Participantes: T. Cristina Rojas Ruiz y Rosalía Poyato Galán

Charla: El ICMS divulga. T. Cristina Rojas Ruiz

PARTICIPACIÓN EN EL PROYECTO WWW.LAB.ELMUNDO.ES/LÍDERES DEL FUTURO. CATORCE PERSONAS CON MUCHO QUE DECIR. 2019 ORGANIZADO POR EL DIARIO EL MUNDO.

Enero 2019.
Participante: Gabriel Lozano Barbero
Video-reportaje: El físico que quiere controlar la luz

PARTICIPACIÓN EN EL ESPACIO DE DIVULGACIÓN CIENTÍFICA: LA CUADRATURA DEL CÍRCULO

www.eldiario.es. La cuadratura del círculo
12 septiembre

Participante: Elena Cabello.

Artículo de divulgación: **En busca de nuevas formas de luz.** La luz influye en la actividad fisiológica de animales y plantas, es por ello que resulta muy interesante encontrar alternativas que permitan controlar de manera precisa la tonalidad de la luz que generamos

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