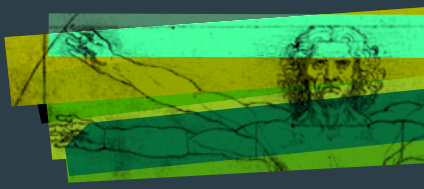
LE STUDIUM

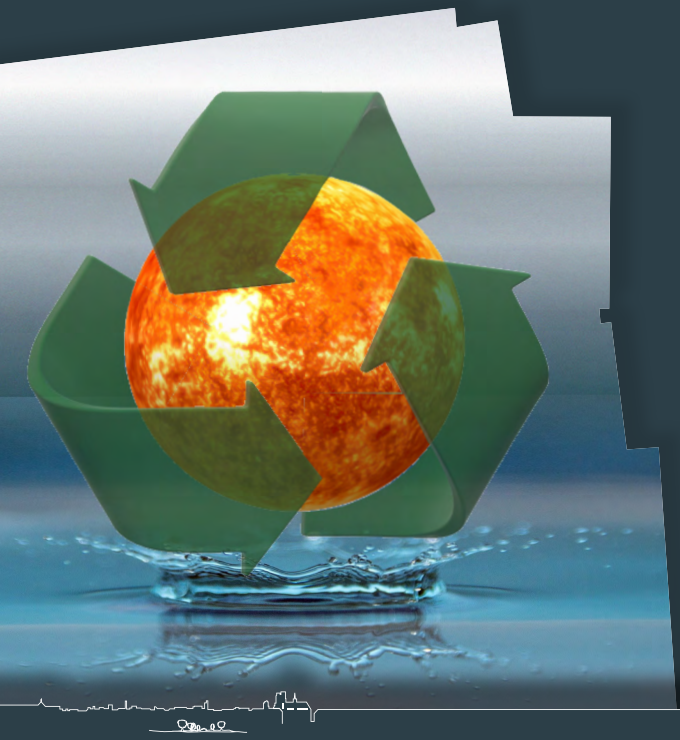
CONFERENCES

VIRTUAL MEETING | 2021



15 - 17 June 2021

Challenges and opportunities in materials for green energy production and conversion



LOCATION

VIRTUAL MEETING

CONVENORS

Dr Edurne Serrano-Larrea

FORMER LE STUDIUM RESEARCH FELLOW / ARD 2020 - LAVOISIER PROGRAMME

Mineralogy and Petrology Department, UPV/EHU - ES

Dr Conchi Ania & Dr Encarnacion Raymundo-Piñero

Extreme Conditions and Materials: High Temperature and Irradiation (CEMHTI) / CNRS - FR

LE STUDIUM

CONFERENCES

VIRTUAL MEETING | 15-17 JUNE 2021

ABSTRACTS

Challenges and opportunities in materials for green energy production and conversion

CONVENORS

Dr Edurne Serrano-Larrea
FORMER LE STUDIUM RESEARCH FELLOW / LAVOISIER ARD 2020 PROGRAMME
FROM: Mineralogy and Petrology Department, UPV/EHU - ES

Dr Conchi Ania
Extreme Conditions and Materials: High Temperature and Irradiation (CEMHTI) / CNRS - FR

Dr Encarnacion Raymundo-Piñero
Extreme Conditions and Materials: High Temperature and Irradiation (CEMHTI) / CNRS - FR

ORGANIZING COMMITTEE

Sophie Gabillet, General Secretary

Dr Aurélien Montagu, Scientific Relations Manager

Maurine Villiers, Communication & Events Manager

LE STUDIUM Loire Valley Institute for Advanced Studies • Région Centre-Val de Loire • FR

EDITO

Created in 1996 on the CNRS campus in Orleans La Source, LE STUDIUM has evolved to become the multidisciplinary Loire Valley Institute for Advanced Studies (IAS), operating in the Centre-Val de Loire region of France. LE STUDIUM has its headquarters in the city centre of Orleans in a newly renovated 17th century building. The amazing facilities are shared with the University of Orleans. In 2014 new developments and programmes linked to the smart specialisation of the Centre-Val de Loire region came to strengthen existing IAS collaborative relationships with the local and the international community of researchers, developers and innovators.

LE STUDIUM IAS offers to internationally competitive senior research scientists the opportunity to discover and work in one of the IAS's affiliate laboratories from the University of Tours, the University of Orleans, National Institute of Applied Sciences (INSA) Centre Val de Loire and ESAD Orléans, as well as of nationally accredited research institutions located in the region Centre-Val de Loire (BRGM, CEA, CNRS, INSERM, INRAE). Our goal is to develop and nurture trans-disciplinary approaches as innovative tools for addressing some of the key scientific, socio-economic and cultural questions of the 21st century. We also encourage researchers' interactions with industry via the IAS's links with Poles of Competitiveness, Clusters, Technopoles, and Chambers of Commerce etc.

LE STUDIUM has attracted two hundred and thirty experienced researchers coming from 47 countries for long-term residencies. In addition to their contribution in their host laboratories, researchers participate in the scientific life of the IAS through attendance at monthly interdisciplinary meetings called LE STUDIUM THURSDAYS. Their presentations and debates enrich the regional scientific community at large (researchers of public and private laboratories, PhD students, research stakeholders' representatives, etc...)

For the period 2015-2021, LE STUDIUM operates with an additional award from the European Commission in the framework of the Marie Skłodowska-Curie Actions (MSCA)-COFUND programme for the mobility of researchers. Since 2013, LE STUDIUM is also an official partner of the Ambition Research and Development 2020 programmes initiated by the Centre-Val de Loire Regional Council to support the smart specialisation strategy (S3) around 5 main axes: biopharmaceuticals, renewable energies, cosmetics, environmental metrology and natural and cultural heritage. New programmes are currently designed to include all major societal challenges.Researchers are also invited and supported by the IAS to organise, during their residency and in collaboration with their host laboratory, a two-day LE STUDIUM CONFERENCE. It provides them with the opportunity to invite internationally renowned researchers to a cross-disciplinary conference, on a topical issue, to examine progress, discuss future studies and strategies to stimulate advances and practical applications in the chosen field. The invited participants are expected to attend for the duration of the conference and contribute to the intellectual exchange. Past experience has shown that these conditions facilitate the development or extension of existing collaborations and enable the creation of productive new research networks.

The present LE STUDIUM CONFERENCE named is "*Challenges and opportunities in materials for green energy production and conversion*" the 110th in a series started at the end of 2010 listed at the end of this booklet.

We thank you for your participation and wish you an interesting and intellectually stimulating conference. Also, we hope that scientific exchanges and interactions taking place during this conference will bring opportunities to start a productive professional relationship with presenting research laboratories and LE STUDIUM Loire Valley Institute for Advanced Studies.

Yves-Michel GINOT

Chairman
LE STUDIUM

INTRODUCTION

The production of green and renewable energy plays a key role in all future energy scenarios towards the zero-emission transition that has to be tackled in the next 30 years. The rapid inter-conversion of electricity into chemical energy offers an important avenue for the use of renewable energy to replace fossil fuels. The generation of electricity in fuel cells from the electrochemical reaction of H_2 and O_2, coupled with the photoelectrochemical water splitting to produce oxygen and hydrogen gases and the valorization of CO_2 through its conversion into solar fuels are also strongly related with the concepts of circular economy. Hence, the study of materials able to efficiently harvest and transform solar energy is highly necessary. Moreover, due to the intermittent character of the solar energy, adequate energy storage and conversion strategies are also needed. This conference, organized by LE STUDIUM Loire Valley Institute for Advanced Studies, in collaboration with CEMHTI (UPR 3079 CNRS), Conditions Extrêmes et Matériaux: Haute Température et Irradiation, has the aim to gather international and national experts in the field of materials science whose works are contributing to electrochemical and photoelectrochemical approaches for clean energy production and conversion.

This international conference is organised in the framework of the LAVOISIER ARD 2020 Programme.

PROGRAMME

TUESDAY 15TH OF JUNE 2021 - 14:30 - 18:10 (GMT+2:00 - PARIS)

14:30 Welcome and introduction

14:40 Prof. Hermenegildo García - Getting closer to a large scale process. Photocatalysts for light-assisted CO_2 hydrogenation

15:20 Dr Jesus Iniesta - Electrochemical and structural characterization of mixed matrix membrane coated electrodes for CO_2 electroreduction

15:50 Geyla Dubed Bandomo - CO_2 conversion with $\{Mn(CO)_3Br\}$ molecular sites into Covalent-Organic Frameworks

16:20 Beatriz Avila - CO_2 electroreduction to formate on Sn, Bi and Sb nanostructured electrodes: from fundamental studies to practical devices

16:50 Break

17:00 Dr Joaquim Faría - Carbon geometries as the base concept for developing efficient photocatalysts

17:40 Dr Carlos Sanchez - Electrolyte engineering for electrochemical CO_2 reduction

WEDNESDAY 16TH OF JUNE 2021 - 14:30 - 18:10 (GMT+2:00 - PARIS)

14:30 Prof. Christel Laberty-Robert - Impact of Electrodes Nanostructuration on Photoelectrochemical Performances

15:10 Dr José Solla - Recent advances and remaining challenges on the use of shape-controlled-metal nanoparticles in Electrocatalysis

15:50 Dr Alejandro Anson - Photoelectrochemical characterization of C/TiO_2 and C/ZnO nanomaterials in aqueous electrolyte

16:20 Dr Conchi Ania - Metal-free nanoporous carbons: possibilities in photo(electrochemical) energy conversion

16:50 ***Break***

17:00 **Prof. Giuseppe Marci -** Photocatalytic reduction of CO_2 in gas-solid and in liquid-solid regimes

17:40 **Prof. Ally Aukaloo -** From molecular to nanostructured materials for artificial photosynthesis

THURSDAY 17TH OF JUNE 2021 - 14:30 - 18:40 (GMT+2:00 - PARIS)

14:30 **Prof. Olivier Joubert -** Brief Overview of Current French Hydrogen Research Activities, Focus on Materials

15:10 **Dr Pascal Brault -** Reactive molecular dynamics simulations of H_2 production and conversion

15:50 **Prof. Sixto Malato -** Solar photocatalytic hydrogen production at pilot scale

16:20 **Prof. Athanasios G. Konstandopoulos -** Material and Reactor Technologies for Solar Fuels

16:50 Break

17:00 **Prof. Juan Matos -** H_2 production on 1D and 2D Carbon-containing Fe-, Co, and Ni-based foamy catalysts.

17:40 **Prof. Bruno Pollet -** Ultrasound-Assisted Electrolytic Hydrogen Production unknowns

18:10 Round Table

TABLE OF CONTENTS

CONVENORS

SPEAKERS

CONVENORS

Dr Edurne Serrano-Larrea

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Edurne Serrano Larrea received her PhD in Chemistry in 2009 from the University of the Basque Country (UPV-EHU). Her thesis work was focused on the search of open inorganic-organic compounds based on the vanadate oxoanion, involving the crystal structure resolution and analysis, and the study of their thermal, spectroscopic, and magnetic properties. She was specialized on the study of the catalytic properties of inorganic-organic materials ana Metal-Organic Frameworks (MOFs). During her post-doctoral phase at the UPV/EHU (20010-2019) she focused to obtain porous crystal frameworks with open metal sites for enhanced catalytic activity. In 2009, received a Le Studium Fellowship to do an internship as invited researcher at CEMHTI-CNRS hosted by Conchi O. Ania and Encarnación Raymundo-Piñero in the project "Electrochemical energy conversion based on metal-free nanoporous electrocatalysts" and her research was centered in the electro and photo(electro) catalytic transformation of CO2 by nanoporous carbon materials. Currently she is Senior Innovation Scientist at microLIQUID S.L. and she is in charge of the materials surface functionalization area for the manufacturing of microfluidic point-of-care devices.

Dr Encarnacion Raymundo-Piñero

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Encarnación Raymundo-Piñero is Research Director at the CEMHTI-CNRS UPR3079 laboratory at Orléans (France). She got a PhD in Materials Science at the University of Alicante (Spain) in 2000 and after a post-doc in the USA she moved to France in 2002 with a Marie Curie fellowship and joined the CNRS as permanent staff in 2006. She received the Science Thesis Award of the University of Alicante in 2001 and in 2009 she was awarded with the CNRS bronze medal. Her research activity is devoted to porous materials for their use as electrodes in energy storage devices and as adsorbents for environmental applications. She has experience on synthesis and modification of nanotextured carbon materials and composites, their physico-chemical characterization and their electrochemical characterization. She is inventor of 13 patents in electrode materials for electrochemical applications, energy storage devices and depolluting materials. She has an H index of 40, with over 9200 citations.

Dr Conchi Ania

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She received her PhD degree in 2003 by Univ. Oviedo (Spain) and performed postdoctoral stays in USA and France. In 2009 she became Tenured Scientist at Agencia Estatal CSIC (in leave of absence), and in 2017 she joined CNRS as Directrice de Recherche. She received the national L'Oreal-Unesco Research Fellowship "For women in Science" (2008), and the Excellence Research Award by University of Granada (2008). She is a grantee of the European Research Council since 2016 and editor of Carbon journal (Elsevier) since 2020.
She has a long-standing interest on nanoporous materials for high-tech applications. Current research interests focus on environmental remediation, gas adsorption/separation, and solar energy conversion. She is co-inventor of two patents (one transferred to industry for 2 years). She has an H-index of 47 with over 6000 citations.

Metal-free nanoporous carbons: possibilities in photo(electrochemical) energy conversion

Co-authors: Edurne S. Larrea, Encarnacion Raymundo- Piñero

Evidences on the photochemical activity of semiconductor and metal-free nanoporous carbons upon irradiation in aqueous environments have opened new opportunities for these materials in photocatalytic applications, beyond their conventional use as inert supports of semiconductors. The aim of this work is to review the recent progress on the photocatalytic performance of nanoporous carbons under different illumination conditions, discussing the photochemical quantum yield of carbons of varied nature and physicochemical features towards energy conversion applications covering water splitting (ORR, OER) and CO_2 photoreduction. The tight confinement inside the nanopores is important for the stabilization of the charge carriers, to minimize surface recombination and promote transfer reactions involving chromophoric surface moieties. Some progress is yet to be done towards the understanding of the fundamentals of the light/carbon/molecule interactions to design photoactive nanoporous carbons with optimized features for an enhanced photocatalytic response under solar light.

SPEAKERS

Dr Alejandro Ansón Casaos

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Dr. Alejandro Ansón Casaos (Zaragoza, 24 June 1978) is a research scientist at ICB-CSIC since August 2012. He got a PhD in Physical Chemistry in 2005 from the University of Zaragoza. His PhD grant was financed by the CSIC Fuel Cell Network. He was a postdoctoral fellow in the Department of Chemical and Materials Engineering, University of Alberta, Edmonton (Canada), between 2005 and 2007. He is the co-author of 104 indexed scientific documents, with h-index of 33. He is the co-inventor of 2 international patents under the property of Nova Chemicals, and a recently disclosed Spanish patent. He has contributed to 110 conference communications.

Photoelectrochemical characterization of C/TiO_2 and C/ZnO nanomaterials in aqueous electrolyte

Carbon nanostructures, including single-walled carbon nanotubes, reduced graphene oxide, and carbon dots, are inserted in TiO_2 and ZnO photoanodes with the aim of improving their activity. For the electrode assembly, TiO_2 or ZnO nanoparticles, as well as carbon nanomaterials, are dispersed in a liquid and deposited on FTO by spray coating. Various mixed and layer-by-layer electrode configurations are considered. As-prepared electrodes are evaluated by photoelectrochemical (PEC) techniques, mostly cyclic voltammetry and transient photocurrent measurements.

The insertion of the carbon component improves the photocurrent response depending on the measurement conditions, specifically the electrolyte pH and the presence of methanol as a hole scavenger. Therefore, hybrid carbon/semiconductor metal oxide nanomaterials can be applied in PEC processes for the production of hydrogen and the degradation of organic pollutants in water.

Prof. Ally Aukaloo

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I am professor in Bioinorganic and Coordination Chemistry at the Université Paris-Saclay and scientific collaborator at the CEA-Saclay in the field of Artificial Photosynthesis. We are dedicated to the development of molecular metal catalysts for the activation of H_2O, CO_2 and O_2. Using light to convert these small molecules to energy rich compounds is a main thrust of our research. We are also developing nanostructured conjugated polymers as photocatalysts for the phot-splitting of water, a major theme in finding new ways to produce a solar fuel.

From molecular to nanostructured materials for artificial photosynthesis

Inspired by chemical subtleties at the active sites of enzymes dealing with the CO_2 management in our biosphere, we will discuss on how hydrogen bonding and electrostatic effects may thrive the catalytic power molecular catalysts. Photosystem II is the only enzyme that can capture sunlight to extract electrons and protons from water that are expelled in the form of reduced quinones. We have developed nanostructured conjugated polymers that can act as photocatalysts to replicate the functions of Photosystem II.

Conjugated nanostructured polymers for the complete photo drive water splitting

We have been interested in the development of conjugated nanostructured polymers (CNP) to manage the PSW. Our strategy was to keep the synthetic segment of the starting building block minimal and to control the degree of polymerization of commercially available monomers that usually lead to extended conjugated organic polymeric materials. We have for this target used surfactants' assemblies to provide a soft template matrix to confine a selected number of monomers within the inner space provided by the soft matrices. Our first incursion in this field was to take profit of the polymerization of diphenylbutadiyne (DPB:) in the confined oil domain of the mesophase to give conjugated poly diphenylbutadiyne (PDPB) nanowires. Upon polymerization in soft templates under radiolysis, we have been able to isolate short oligomers of DPB, we termed as nano-PDPB. We have demonstrated that these conjugated polymer nanostructures are very active photocatalysts under visible light. When dispersed in water without the addition of any co-catalyst nor any sacrificial agent and submitted to visible light irradiation, O_2 was detected reaching the saturation solubility in water within minutes. The electrons were recovered as reduced forms of quinones.

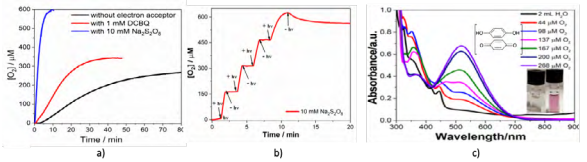

Figure : a) time courses of O_2 evolution in absence of an electron acceptor (EA), in presence of a quinone as a reversible EA and $S_2O_8^{2-}$ as an irreversible EA b) stepwise irradiation in presence of $S_2O_8^{2-}$ c) reduction of quinone to form quinhydrone after photocatalysis

Ref: Visible light-driven simultaneous water oxidation and quinone reduction by a nano-structured conjugated polymer without co-catalysts. J. Patel, X. Yuan, S. M. Marinho, W. Leibl, H. Remita, A. Aukauloo, Chem. Sci., 2020, 11, 7324.

Beatriz Ávila Bolívar

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PhD student in the Research Group in Electrocatalysis, ascribed to the Department of Physical Chemistry and to the Institute of Electrochemistry at the University of Alicante (Spain).

CO_2 electroreduction to formate on Sn, Bi and Sb nanostructured electrodes: from fundamental studies to practical devices

The electrochemical reduction of CO_2 into chemical products of interest has been considered as an interesting route not only to mitigate climate change but also to store renewable energy in the form of value-added chemicals. Among other possible chemicals, formic acid (HCOOH) or formate (HCOO–) (depending on pH value) is one of the most attractive carbon-based products due to its potential uses and its high world demand including its use as a fuel for low-temperature fuel cells and as a renewable hydrogen carrier molecule. In this contribution, we will summarize our most recent and relevant results on the use of Tin (Sn), Bismuth (Bi), and Antimony (Sb) nanostructured based electrocatalysts for the selective conversion of CO_2 into formic acid/ formate. This summary will cover from fundamental studies, including the synthesis, characterization and electrochemical behaviour of these nanostructured materials on conventional H-type electrochemical cells, to practical applications, focused on the development and testing of a process for the electrocatalytic reduction of CO_2 to formate (HCOO–) operating in a continuous way, with different electrochemical reactor configurations, such as Gas Diffusion Electrodes (GDE), or Catalyst Coated Membrane Electrodes (CCME), and under different operating conditions. The results obtained will demonstrate the potentialities of this electrochemical technology for the valorisation of CO_2 into formic acid/ formate.

Dr Pascal Brault

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Pascal Brault (60 years old) got is PhD in 1987 in theoretical atomic and molecular physics. He is now CNRS senior scientist at GREMI laboratory, University of Orléans. He is specialist of plasma deposition and treatments of materials, especially relevant for energy applications, nanocatalysts for fuel cells. For 10 years, he is focusing on reactive molecular dynamics simulations of plasma processing. He is co-author of more than 140 publications, 200 communications among them 47 invited. He is co-inventor of 7 patents. In 1992-1993, he was granted as Alexander von Humboldt fellow (Max-Planck-Institut für Strömungsforschung, Göttingen, Germany).

Reactive molecular dynamics simulations of H_2 production and conversion

Since H_2 production and conversion efficiencies can be improved and monitored at the molecular scale, reactive molecular dynamics simulations (rMDS) are expected to be of great promise for understanding associated basic mechanisms. Basically, rMDS is a technique aiming at solving full trajectories of a set of atoms, molecules, nanoparticles, with the only ingredient of force fields driving interactions between relevant species.

Due to the recent growth of improved and reactive force fields such as ReaxFF and COMB3 families, it become possible to address complicated reactive process such as H_2 production and conversion from various sources. Moreover, these force fields are including variable charges and thus electrochemical processes such as electron transfers become tractable by rMDS.

The present talk will review the works carried out on both production and conversion of H_2. This will cover a broad range of mechanisms such as H_2 production from water splitting, aluminium water interactions, PEM electrolysis as well nanocatalysts for PEM fuel cells, oxygen reduction reactions, etc. In all cases, the frame, performances and limitations of the simulations will be addressed.

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- PhD (postgraduate study) in Chemical Science and Technology (ICIQ/URV). Institut Català d'Investigació Química.
- MSc. in Electrochemistry. Synthesis, photochemical and photoelectrochemical characterization of TiO2 nanotubes membrane modified with electrocatalytic materials. Center of Research and Technological Development in Electrochemistry, Querétaro, México
- BSc. in Radiochemistry (Cum laude). Synthesis of neuromarkers as potential precursors for the radiodiagnosis of Alzheimer's disease. Higher Institute of Technologies and Applied Sciences, Havana, Cuba

CO_2 conversion with {$Mn(CO)_3Br$} molecular sites into Covalent-Organic Frameworks

Effective large-scale CO_2 conversion to fuels or value-added chemicals using renewable energies is critical to reduce our environmental impact [1]. To this end, better understanding of the CO_2 mechanism is needed to develop efficient and selective catalysts that operates in water controlling H_2 evolution. Covalent Organic Frameworks (COFs) are reticular materials, which can be used to combine the advantages of the well-defined molecular catalysts and the heterogeneous ones [2]. In this work, we present the first COF based on tricarbonyl Mn units, that by π-π stacking is attached to MWCNTs form electrocatalytic electrodes active for CO_2 reduction in neutral water. The activity of these catalysts was evaluated by electrochemical techniques with stability in aqueous solution. With these materials we have integrated the classical Mn(bpy)CO3Br catalyst into a heterogeneous material which clearly enhances its catalytic activity (FE~50%) at low overpotentials (~450 mV) in pure water. COF/MWCNTs/Nafion leds higher faradaic efficiency than molecular system. The encapsulation of tricarbonyl Mn active sites with a reticular covalent organic structure plays an important role by favouring the electrocatalytic CO_2 reduction over competitive H_2 evolution reaction. The spectroelectrochemical studies evidence the formation of five-coordinate species in the catalytic cycle for CO formation.

References :

[1] Alessandro Sinopoli, Nathan T. La Porte, *Coord. Chem. Rev.*, 365, **2018** pp 60–74.

[2] Lin S, O. M. Yaghi, *Science, 349*, **2015** pp 1208-1213.

[3] B. Reuillard, E. Reisner, *J. Am. Chem Soc.*, 169, **2017** pp 14425-14435.

[4] James J. Walsh, Alexander J. Cowan, ***Phys. Chem., 20, 2018*** pp 6811-6816.

Prof. Joaquim Faría

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Joaquim L. Faría (PhD Physical Organic Chemistry – 1993) is currently Associate Professor with habilitation at Faculty of Engineering of the University of Porto (FEUP), Board Member of the Department of Chemical Engineering, Scientific Committee member of the MSc in Chemical Engineering, Director of the PhD Program in Chemistry at FEUP and Vice President of the Portuguese Chemical Society. Head of the Materials research line of the Associated Laboratory in Chemical Engineering (FEUP). Published over 200 articles (ORCID: 0000-0002-6531-3978; h-index >50) on catalysis and catalysed reactions with thermal and photo-activation for environmental applications, energy, and fine chemical synthesis. He received the Scientific Prize of the Portuguese Association of PhD Studies in France (APDF).

Carbon geometries as the base concept for developing efficient photocatalysts

During the past several decades' new carbon allotropes have been accounted for and synthesised. Many notable breakthroughs include exciting works on synthesis, functionalisation, characterisation and applications of carbon nanotubes, graphene, and graphene-like structures, like graphitic carbon nitrides.

The exciting progress made since the initial use of carbons as modifiers of optical semiconductors has stimulated many types of research in different fields of the immediate and relevant application. Apart from the obvious applications in water treatment and remediation, there are compelling examples in photocatalytic synthesis, assisted conversion with the production of valuable chemicals or fuels, photocatalytic production of solar fuels, and photo-assisted purification of compounds of biological and health relevance.

Such materials also prompted the development of new integrated and technological solutions, including new photoreactors and combined systems for engineering processes, including microfluidics and additive fabrication.

The duality between fundamental and applied, as well as between theory and practice, will be used to present some of our latest progress in the field of conversion of hazardous chemicals in aqueous streams with the in-situ generation of added value compounds, selective photocatalytic synthesis, photo-assisted enzyme purification, and solar fuel production.

Prof. Hermenegildo Garcia

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Hermenegildo Garcia is Professor at the Instituto de Tecnologıa Quimica of the Technical University of Valencia, a joint centre of the Technical University of Valencia and the Spanish National Research Council. Prof. Garcia has been active in the field of heterogeneous catalysis and photocatalysis, has published over 800 papers with an H index 105. Prof. Garcia is Doctor Honoris Causa from the University of Bucharest, Honorary Professor of King Abdulaziz University and the recipient of the 2016 Rey D. Jaime I award in New Technologies.

Getting closer to a large scale process. Photocatalysts for light-assisted CO_2 hydrogenation

There is an urgent need to diminish atmospheric CO_2 emissions to meet with the International agreements on global warming and climate change. One of the possible strategies is the utilization of CO_2 as feedstock of fuels and chemicals. Among the possible reactions, CO_2 hydrogenation can give a wide variety of products from methane to light hydrocarbons and alcohols of high added value. However, due to its high chemical stability, CO_2 transformation requires a considerable amount of energy to run the reaction at temperatures above 450 oC and high pressure. In this context, the use of solar light as primary energy source and development of selective photocatalysts to promote the reaction becomes highly relevant. The presentation will summarize the contribution of the group to develop efficient solar photocatalysts for selective CO_2 hydrogenations, indicating the targets to be achieve to be competitive with conventional thermal catalytic processes and the possibility to achieve these goals in short term.

Dr Jesús Iniesta

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Dr. Jesus Iniesta is member of the research group of Applied Electrochemistry and Electrocatalysis. Dr. Iniesta completed his PhD at the University of Alicante in 1999. He received a Marie Curie Fellowship (2001-2003, Coventry University) and enjoyed several postdoc positions in Birmingham University and Manchester Metropolitan University. Full Professor at the Physical Chemistry Department since 2020.

Electrochemical and structural characterization of mixed matrix membrane coated electrodes for CO_2 electroreduction

Co-authors : Dr. Clara Casado-Coterillo[1], Dr Aitor Marcos-Madrazo[2]

[1]Department Chemical and Biomolecular Engineering, University of Cantabria, 39005 Santander, Spain

[2]University of Cantabria

The need for novel membrane coated electrodes (MCE) for the electrochemical reduction of carbon dioxide (CO_2RR) may offer advantages in terms of control of reaction, catalytic activity and stability of the electrode. The study of the possibilities of mixed matrix membrane (MMM) overlayers with tuneable ion-exchangeable, conductive and transport properties are essential targets for the performance of the MCEs for electrochemical applications. The knowledge on the effect of local pH and CO_2 concentration diffusion to catalyst site, among others, on the selectivity of the CO_2 electroreduction to high added-value products under alkaline conditions, especially when using Cu as catalyst, is still scarce. The communication aims at evaluating the relationship of the synthesis and morphological characterization of the MCEs and the electrochemical characterization of novel based copper-containing MMMCE with high CO_2 permeability, chemical and mechanical stability in alkaline solutions, on the performance, selectivity and stability of electrodes in CO_2RR. Cyclic voltammetry and electrochemical impedance spectroscopy will give insight about the electrochemical behaviour pf MCEs in comparison with the typically used gas diffusion electrodes (GDEs) prepared with commercial ionomers. Preliminary electrolysis for the CO_2RR will reveal the significant impact of the presence of the polymeric membrane overlayer on the efficiency and selectivity of CO_2RR.

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Olivier JOUBERT (1965) is presently full professor in Chemistry of Materials at University of Nantes and is Chair of electrochemical storage and conversion of energy group (ST2E) of "Institut des Matériaux Jean Rouxel (CNRS-IMN)". The major research interests of professor JOUBERT revolve around development of new materials for technological applications such as high and intermediate temperature ceramic Solid Oxide (SO) fuel or electrolyser cells (SOFC and SOEC). He is co-author of 120 publications, 25 invited talks and 5 patents. Olivier Joubert is chairing the French Research Network on Hydrogen Energy which assembles all French academic research groups in the field of electrolysis production and storage of hydrogen and also its conversion to electricity using fuel cell.

Brief Overview of Current French Hydrogen Research Activities, Focus on Materials Development

Twenty years ago, the French scientific community working in the field of hydrogen, started to federate under the leadership of the CNRS. It took the form of successive Research Grouping (GdR) bringing together experts in solid oxide fuel cell (SOFC), proton exchange polymer membrane (PEMFC) fuel cells, hydrogen storage and systems mainly from CNRS but also from CEA. These GdRs promoted and structured an interdisciplinary field of research with excellent results.

Since January 1st of 2020, the CNRS community has formed the Research network on Hydrogen energy (FRH2) based on the active nucleus of the former GDR laboratories (29 laboratories) with about 300 researchers and professors. A new organization, "from material to system", more visible from industries has been set up around four main technical axes, the production of hydrogen, its storage, and its conversion into electricity for mobile and stationary applications and around 2 transversal axes, education and technological platforms. The first part of the presentation will give a brief overview of this french CNRS research network on hydrogen energy including some highligts for each axes.

The second part will be dedicated to the development of materials used in solid oxide fuel cells (SOFC) and more precisely to the ceramic electrolyte: optimization of the microstructure of the material via accurate monitoring of the synthesis and temperature treatment steps.

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Dr. Athanasios G. Konstandopoulos, Descartes Laureate, Knight of the Legion of Honour of the French Republic, European Research Council Advanced Grant Holder, Fellow of the Society of Automotive Engineers, Professor of Chemical Engineering, Founder and Director of the Aerosol and Particle Technology Laboratory at CPERI/CERTH and Aristotle University, is a specialist in nanoparticle technologies, combustion aerosols, carbon-neutral solar fuels and structured reactors for catalytic emission control, sustainable mobility, clean energy, circular economy and biotech applications. He is the author of numerous widely cited publications and a former member of the Board of Governors of the European Commission's Joint Research Center.

Material and Reactor Technologies for Solar Fuels

The synthesis of carbon-neutral/zero-carbon footprint fuels via solar thermochemical processes ("Solar Fuels") represents a promising approach for the realization of a sustainable energy future. These high temperature processes (effected by concentrated solar technology) "regenerate" streams of "dead hydrogen" (H_2O) and/or "dead carbon", CO_2, through thermochemical Water (WS) and/or Carbon Dioxide Splitting (CDS) into H_2 and CO respectively, after contact with oxygen-deficient, "redox" materials that are thus re-oxidized. Subsequently thermal reduction of the material is applied, by raising the temperature at a higher level, thus establishing a two-step cycle. Solar CO and H_2 can be converted into solar fuels through gas-to-liquid catalytic processes. In this work, we summarize Material and Reactor Technologies for the development of solar fuels. The approach entails the identification of attractive candidate material families (exhibiting redox behavior) exploiting the potential of computational chemistry based on ab initio calculations. Subsequently the desired materials are synthesized employing advanced manufacturing methods to generate tailored compositions/architectures. These are shaped into structured reactor forms via various techniques and their performance is assessed under realistic conditions in the lab and at in-house and field-based, solar testing facilities.

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C. Laberty-Robert is a professor in Materials Sciences at Sorbonne University. She completed her PhD ('96) degrees in Materials Sciences at Toulouse University. Major research fields include nanostructured thin films and hybrid organic-inorganic materials for battery, low temperature fuel cell and water splitting. Noticeable milestones in C. Laberty-Robert's carrier are 2 years in Navrotsky's group at Princeton University, where she conducted research on the thermochemistry of iron and manganese polymorphs and 3 years at Naval Research Laboratory for a specialization in electrochemistry for fuel cell and battery. A special focus is dedicated to the investigation of chemistry and processing (dip-coating, electrospinning) to synthesize nanostructured materials with optimum performances.

Impact of Electrodes Nanostructuration on Photoelectrochemical Performances

Hydrogen is presented by some industrialists and managers as a potential pillar of the ecological transition, particularly in the context of transport (hydrogen-powered cars, hydrogen-powered trains, hydrogen-powered airplanes, etc.), the storage of energy from intermittent renewable sources or in industry (steel and chemical industries). Using hydrogen to decarbonize the economy only makes sense if it is produced in a decarbonized way. In this context, this talk will focus on hydrogen production from renewable sources, solar and water. The performance (absorption of light, creation of photo-charges, their migration and their reactivity with water) of the photoelectrodes depend strongly on the chemistry, structure and microstructure of the materials used. All these physical-chemical parameters can be trigger through a judicious control of processing parameters. This talk will focus primarily on three different processing approaches based on solution chemistry for synthesizing efficient and durable photo-electrodes (Mo-$BiVO_4$, TiO_2 and CuO/Cu_2O). The performance of the photoelectrodes will be discussed as function of structure, microstructure (porosity, particle size, thickness) and hetero-structure. More importantly, for comparing the performance of the three TiO_2 polymorphs, a judicious photoelectrodes' manufacturing is developed in order to solely measure the impact of the structure keeping all the other parameters (porosity, thickness and particle size) constant.

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Solar photocatalytic hydrogen production at pilot scale

Solar energy is well-recognized as a sustainable and clean energy source. Among the various approaches to solar energy conversion, solar-driven hydrogen production is one of the most promising ways to convert solar energy into hydrogen, a storable fuel. Hydrogen is undoubtedly one of the most attractive renewable alternatives to fossil fuels for a number of reasons: (i) hydrogen can be safely stored; (ii) it is an excellent energy carrier since it can store 3 times as much energy as conventional natural gas; (iii) its combustion is environmentally friendly (no release of greenhouse gases or hazardous by-products). However, the production of hydrogen currently relies in steam reforming of CH_4 and CO, and thus, involves the consumption of fossil fuel energy and the emission of greenhouse gases. There are efforts to find alternative renewable technologies to produce hydrogen, the so-called green hydrogen.

It is well-known that the efficiency of heterogeneous photocatalysis for hydrogen generation from water splitting is quite low. The efficiency of photocatalysis hydrogen generation can be improved by using aqueous sacrificial agents (organic electron donors) dissolved in water. Industrial wastewater containing high concentrations of methanol/glycerol/formic acid have been used as feedstock. Effluents from municipal wastewaters could also be used, as they would contain dozens of mg/L of organic load. This means that in a simultaneous process, hydrogen generation and contaminants removal can take place. It is important to notice that the application of this technology has a double purpose: first, to produce a clean fuel such as hydrogen and, second, to treat, reduce the organic load and decontaminate wastewater in certain extent.

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Giuseppe Marcì was born in 1967 in Palermo (Italy). He graduated in Chemical Engineering in 1992. PhD in Chemical Engineering (1997). He is currently Associate Professor of Chemistry (2015) at the University of Palermo and he has received in 2017 the full professor qualification in Chemistry. His scientific activity has been focused on the field of Heterogeneous Photocatalysis and recently also on catalytic processes for biomass valorisation. He is co-author of ca. 140 scientific papers and 230 abstracts in proceedings of congress. (H index 43, citation 7299 Scopus 2021).

Photocatalytic reduction of CO_2 in gas-solid and in liquid-solid regimes

The increasing CO_2 level in the atmosphere is a global environmental problem; therefore, the development of efficient catalytic processes for CO_2 reduction is a challenge. Heterogeneous photo-catalysis is an attractive technology for this purpose. Literature reports that formic acid, methanale, methanol and methane can be formed by photocatalytic reduction of CO_2. In this contribution, it will be showed that the CO_2 reduction products and their distribution depend on several factors like the kind of photocatalyst and the presence of water vapor [1]. Moreover, the coupling of different photo-catalyst (GaP/TiO2) and the use of nafion membranes as support of the catalyst can improve the photocatalytic performances [2,3]. In the case of photo-reduction performed in liquid-solid regime, the temperature and the pressure have a positive effect on the productivity. Finally, an investigation into the stability of C_3N_4 used as photo-catalyst for CO_2 reduction will be discussed [4].

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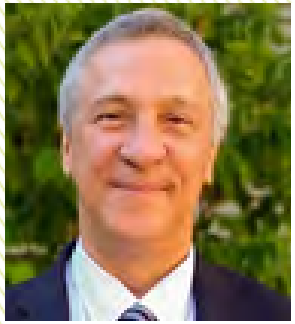

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H_2 production on 1D and 2D Carbon-containing Fe-, Co, and Ni-based foamy catalysts.

C-containing Fe-, Co-, and Ni-based catalysts have been synthesized from the controlled pyrolysis of saccharose. The topological properties of the 2D materials were significantly different from other 2D carbon-based cell structures. TEM and STEM showed that catalysts are composed by 1D and 2D morphologies, manly those with Co- and Ni, and these morphologies seems to be remaining from the pristine carbon structures in absence of metals. Carbon seems to play a template role for the growing of the metal-containing structures. Electron microscopy also showed that catalysts are composed by a high compaction on nanoparticles in the range 5-30 nm. XRD showed that graphite is the main crystalline phase; nevertheless, XPS showed important differences with respect to graphite. NEXAFS and RAMAN spectroscopy suggested the formation of graphene oxide. The textural analysis pointed out the formation of a hierarchically pore structure in the present materials. Fe-, Co- and Ni-doped carbon foams were studied in the thermocatalytic H_2 production from dry methane reforming under soft experimental conditions. The catalytic assays demonstrated that the metal-containing carbon foams were clearly more active and suffered less deactivation than bulk conventional catalysts prepared by wetness impregnation. It can be concluded then that the controlled pyrolysis of saccharides opens a new, low cost and eco-friendly method for the preparation of graphene-based foams as catalytic membrane reactors.

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Ultrasound-Assisted Electrolytic Hydrogen Production

In this study, we investigated the effects of power ultrasound (26 kHz, up to ~75 W/cm2, up to 100% acoustic amplitude, ultrasonic horn) on the hydrogen evolution reaction (HER) on a platinum (Pt) polycrystalline disc electrode in 0.5 M H_2SO_4 by cyclic and linear sweep voltammetry at 298 K. We also studied the formation of molecular hydrogen (H_2) bubbles on a Pt wire in the absence and presence of power ultrasound using ultra-fast camera imaging. It was found that ultrasound significantly increases currents towards the HER i.e., a ~250% increase in current density was achieved at maximum ultrasonic power. The potential at a current density of –10 mA/cm2 under silent conditions was found to be –46 mV and decreased to –27 mV at 100% acoustic amplitude i.e., a ΔE shift of ~+20 mV, indicating the influence of ultrasound on improving the HER activity. A nearly 100% increase in the exchange current density and a 30% decrease in the Tafel slope (b) at maximum ultrasonic power, was observed in the low overpotential region, although in the high overpotential region, the Tafel slopes were not significantly affected when compared to silent conditions. In our conditions, ultrasound did not greatly affect the "real" surface area and roughness factor i.e., the microscopic surface area available for electron transfer. Overall, it was found that ultrasound did not dramatically change the mechanism of HER but instead, increased currents at the Pt surface area through effective hydrogen bubble removal.

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Electrolyte engineering for electrochemical CO_2 reduction

Ionic liquids (ILs) in electrocatalysis have attracted a lot of attention from the seminal work of Rosen et al. [1] where they achieved a relevant overpotential decrease for CO_2 reduction reaction (CO_2RR) on a silver electrode. Moreover, ILs have been proposed as organic promoters for enhancing the catalytic activity by modifying the electrostatic properties at the electrochemical interface. Then, electrolyte engineering has become a powerful way to impact on the stability of reaction intermediates and control reaction selectivity.

The role of different ILs as simultaneous supporting electrolyte and co-catalyst in acetonitrile for CO_2 electroreduction in the presence of a model molecular catalyst [Re(bpy)$(CO)_3$Cl] is presented here. [2] In particular, the nature of the cation, anion and cation alkyl chain is varied by a choice of 5 different ILs, including imidazolium and pyrrolidinium cations and their results are compared to conventional benchmark supporting electrolyte. Under catalytic conditions an overpotential diminution of 0.33 V for CO_2 to CO conversion is achieved in the presence of ILs. Finally, a mechanistic explanation is provided to justify this behaviour.

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Recent advances and remaining challenges on the use of shape-controlled-metal nanoparticles in Electrocatalysis

The incorporation of shape-controlled metal nanoparticles in Electrocatalysis is contributing significantly to a better understanding of the correlations between surface structure and electrochemical reactivity at the nanoscale [1,2]. In this communication, some of the basic requirements to properly use these shaped metal nanoparticles in electrochemical reactions including surface cleanness, and correlations between particle shape and surface structure will be reported. Then, some recent advances in the application of these shaped nanomaterials for different electrochemical reactions of interest will be highlighted [3-5]. Finally, some of the most important remaining challenges on this topic will be discussed.

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