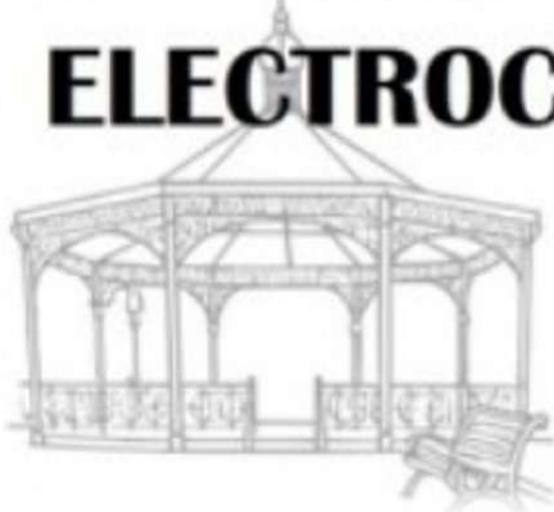


# 1<sup>ST</sup> MEDITERRANEAN ELECTROCHEMISTRY SCHOOL

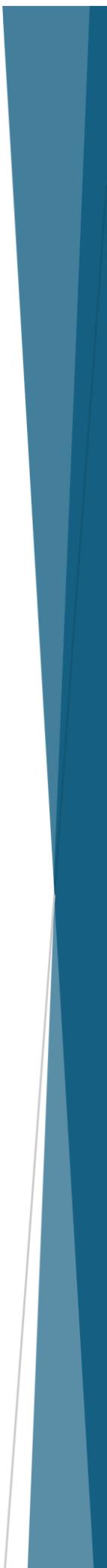


Puertollano (Spain)  
25-29 January 2026



## BOOK OF ABSTRACTS





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## ABSTRACTS

## Evaluation of the feasibility of scaling up CO<sub>2</sub> electroreduction to formate in a pre-industrial pilot plant

Jose Antonio Abarca\*, Guillermo Diaz-Sainz, Angel

*Departamento de Ingenierías Química y Biomolecular, Universidad de Cantabria, Avenida de los Castros s/n, 39005 Santander, Spain*  
\*Presenting author's email:

### Abstract

CO<sub>2</sub> electroreduction to value-added products is gaining increasing attention in the context of the energy transition, as it enables both CO<sub>2</sub> utilization and the storage of renewable energy in chemical bonds. Formate is particularly attractive due to its wide range of industrial applications and high hydrogen density, making it a promising liquid energy carrier [1,2]. Laboratory-scale studies have demonstrated Faradaic efficiencies above 90% and formate concentrations above 300 g·L<sup>-1</sup>, highlighting the feasibility of scaling this technology toward industrial implementation [3]. As a first step in this direction, the present work addresses the design, construction, and validation of a pilot plant capable of processing 2 L·min<sup>-1</sup> of CO<sub>2</sub> using an electrolyzer operating at 200 mA·cm<sup>-2</sup> (20 A total).

Scaling the active area from 10 to 100 cm<sup>2</sup> presents significant challenges, particularly in ensuring uniform CO<sub>2</sub> delivery through the gas diffusion electrode (GDE). CFD simulations reveal that conventional unstructured flow fields exhibit extensive dead zones, with up to 75% of the volume experiencing near-zero velocities, thereby limiting mass transport. In contrast, a serpentine flow field reduces dead zones to approximately 10%, doubles the effective volume-to-electrode area ratio, and increases both fluid velocity and Reynolds number, thus enhancing convective transport and improving CO<sub>2</sub> accessibility to catalytic sites [4]. After optimizing the flow field, a 3D-designed electrolyzer prototype was manufactured and integrated into a pilot plant designed to replicate industrial-like operating conditions. The setup enables automated data acquisition and real-time monitoring, laying the foundation for future digital twin development. The system includes gas feed controllers, electrolyte tanks and pumps, a 20 A power supply, gas analyzers, and sensors for flow rate, conductivity, and temperature. Experimental validation involved 5-hour continuous tests using a Bi<sub>2</sub>O<sub>3</sub>-based GDE cathode and a Nafion 117 membrane, with 1.5 M KOH as the anolyte and a mixture of 0.5 M KCl and 0.45 M KHCO<sub>3</sub> as the catholyte. Under a CO<sub>2</sub> flow of 2 L·min<sup>-1</sup> and a current density of 200 mA·cm<sup>-2</sup>, the system operated stably, achieving Faradaic efficiencies above 90%, formate concentrations up to 150 g·L<sup>-1</sup>, an overall energy efficiency of ~20%, and cell voltages below 7 V. Hydrogen evolution remained limited, with efficiencies below 8%.

Based on these results, the pilot plant could produce up to 350 g of formate per day. When powered by renewable electricity, the process enables storage of green energy in the chemical bonds of formate, which can subsequently be converted back to electricity via formate fuel cells. Future work will focus on further scale-up through stack-based configurations and extended operation times enabled by automated control, as well as improving energy efficiency via advanced anode materials or alternative anodic reactions, such as glycerol oxidation, that operate at lower potentials and yield additional value-added products.

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## Improvement in Catalytic Performance of Pt-Ru/C Electrocatalysts Prepared by Impregnation and Sulphite Complex Routes

Mairaj Ahmad<sup>a,\*</sup>, Carmelo Lo Vecchio<sup>a</sup>, Angelo Mondello<sup>a</sup>, Riccardo Dollenza, Vincenzo Baglio<sup>a</sup>

<sup>a</sup> CNR-ITAE, Via Salita S. Lucia sopra Contesse 5, 98126 Messina, Italy

\*Presenting author's email: [mairajahmad@cnr.it](mailto:mairajahmad@cnr.it)

### Abstract

Alcohol electroreforming is an emerging electrochemical method that enables the oxidation of alcohols such as methanol or ethanol at the anode, while simultaneously reducing protons to hydrogen at the cathode within an electrochemical cell [1]. The oxidation of short-chain renewable alcohols is much more favorable, with significantly lower oxidation potentials (e.g., 0.016 V for methanol and 0.084 V for ethanol) compared with the oxygen evolution reaction in the conventional water electrolysis process. As a result, alcohol electroreforming presents a more energy-efficient pathway for hydrogen generation, consuming approximately 18.5 kWh per kilogram of hydrogen produced [2]. To enhance the performance and scalability of these systems, ongoing advancements in catalyst design, a deeper understanding of reaction mechanisms, and optimization of operating conditions are essential. To investigate this approach, 50% Pt<sub>1</sub>Ru<sub>1</sub>/C anodic catalysts were synthesized in-house by combining a platinum sulfite complex with a carbon support (Ketjenblack, KB) and a ruthenium precursor by impregnation and sulfite complex routes. The catalysts' structural, compositional, and morphological properties were characterized using X-ray diffraction (XRD), and Scanning Transmission Electron Microscopy coupled with Energy Dispersive X-ray spectroscopy (S/TEM-EDX). The electrooxidation of alcohols was further evaluated using a rotating disc electrode (RDE) setup and full electroreforming cells in both acidic and alkaline environments to assess catalyst activity, hydrogen production, system performance, and long-term durability.

### Acknowledgements

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## Hydrogen production by thermo-electrochemical cycles based on ceria

Elena Alfonso-González<sup>a,\*</sup>, Raúl Peño<sup>a</sup>, Jonathan Perry<sup>b</sup>, Alberto de la Calle<sup>a</sup>, Timothy W. Jones<sup>c</sup>, Scott W. Donne<sup>b</sup>, Juan M. Coronado<sup>a</sup> and Alicia Bayón<sup>a</sup>

<sup>a</sup> Instituto de Catálisis y Petroleoquímica (CSIC), C/ Marie Curie, 2, Madrid, 28049, Spain

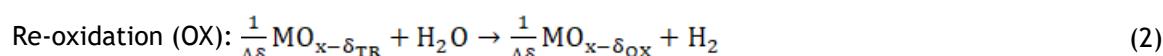
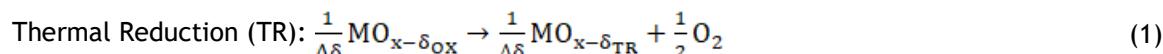
<sup>b</sup> Discipline of Chemistry, University of Newcastle, Callaghan, NSW, 2308, Australia

<sup>c</sup> CSIRO Energy, 10 Murray Dwyer Circuit, Mayfield West, 2304, New South Wales, Australia

\*Presenting author's email: e.alfonso.gonzalez@csic.es

### Abstract

The IEA has acknowledged green hydrogen as an essential energy technology in the short term.<sup>1</sup> Among the different green hydrogen production routes, solar thermochemical cycles (STCH) are an attractive alternative able to achieve low-cost hydrogen production. Specifically, two-step non-stoichiometric metal oxides are gathering most of the ongoing research efforts among the thermochemical water splitting.<sup>2</sup> In two-step STCH, first an endothermic reduction takes place at high temperatures and low oxygen partial pressures (Equation 1), followed by an exothermic re-oxidation of the  $\text{MO}_x$  with water in which the hydrogen is produced (Equation 2). The net outcome is the water splitting reaction, since the  $\text{MO}_x$  is not consumed in the cycle.



Here,  $\delta$  represents the degree of non-stoichiometry or the amount of oxygen vacancies in the crystalline structure. To date, the current state of the art material in non-stoichiometric STCH applications is ceria,  $\text{CeO}_2$ , which shows excellent stability with rapid re-oxidation reaction kinetics. However, in order to achieve oxygen vacancies in its structure, it requires a high reduction temperature. For example, a  $\delta$  of 0.038 needs 1400 °C and a  $\text{pO}_2$  of  $2 \cdot 10^{-6}$  bar.<sup>3</sup> For the re-oxidation of  $\text{CeO}_2$  in the presence of water, a temperature swing is required to be achieved (at least 200 °C lower than thermal reduction) to obtain a minimum of 10% hydrogen conversion yield.<sup>4</sup> However, a temperature swing implies the need of a heat recovery strategy in order to achieve high solar-to-hydrogen conversion efficiencies.<sup>5</sup>

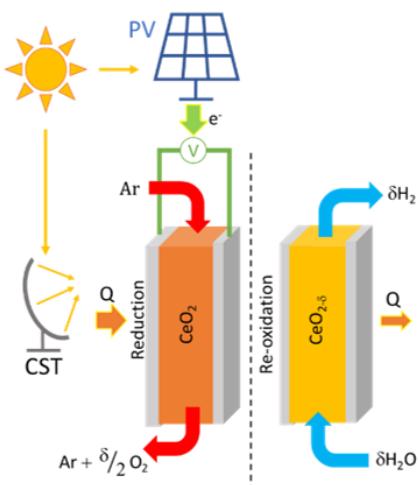


Figure 1. Scheme of the thermo-electrochemical cycle based on  $\text{CeO}_2$

The application of larger potentials results in the formation of a larger number of oxygen vacancies, which converts into a larger amount of hydrogen produced.

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## Development of Advanced Solid Oxide Fuel Cells and Electrolysis cells: Materials and electrochemical characterization

Irene Ayuso<sup>a\*</sup>, Angela Moratalla<sup>a</sup>, M. Rosario Serrano de la Cruz<sup>a</sup>, Adrián Pardo<sup>a</sup>, Noelia Díaz<sup>a</sup>, Jaime Broceño<sup>a</sup>, Cristina Montes<sup>a</sup>, Lidia Sanchez-Beato<sup>b</sup>, Roberto Campana<sup>a</sup>

<sup>a</sup>Hydrogen National Centre (CNH2), Puertollano, Spain

<sup>b</sup>Davinci, Madrid, Spain

\*Irene.ayuso@cnh2.es

### Abstract

Solid oxide cells (SOCs) technology offers a glimpse of the future: the transition toward sustainable energy. This technology is widely recognized for its high efficiency and low pollutant emissions [1]. Despite their advantages, several challenges including material degradation, carbon deposition on Ni-based electrodes, and the high operating temperatures required by conventional electrolytes, still limit large-scale deployment [2,3].

For this reason, the National Hydrogen Centre (CNH2) has developed and optimized novel planar structures for fuel and electrolysis cells (SOFC/SOEC), analyzing its electrochemical behavior. Yttria-stabilized zirconia (YSZ) has been selected due to its high chemical stability, robust mechanical properties, and competitive cost, making it the reference material in solid oxide cell technologies. Conversely, scandia-stabilized zirconia (ScSZ) has been chosen for its superior ionic conductivity in the 700-850 °C temperature range, which enables a reduction in operating temperature without compromising electrochemical performance. In contrast, anode-supported cells employ a structural support based on a Ni-YSZ cermet, which enables the deposition of thinner electrolyte layers and, consequently, a significant reduction in overall ohmic losses, allowing efficient operation at intermediate temperatures. Supports cells were elaborated by tape casting, while the electrodes were deposited by ultrasonic spraying or by screen printing, employing NiO-YSZ as the hydrogen electrode, GDC and LSM-YSZ as barrier interlayer between electrolyte and oxygen electrode, and LSM as the oxygen electrode.

Electrochemical characterization was performed changing both temperature and humidity conditions through polarization curves as well as electrochemical impedance spectroscopy (EIS). The results showed that YSZ-based electrolyte-supported cells reached ~600 mA/cm<sup>2</sup> under electrolysis at 850 °C with 75% steam, whereas ScSZ cells achieved 1.1 A/cm<sup>2</sup> under the same conditions, demonstrating superior ionic conductivity and enhanced intermediate-temperature performance. NiO-YSZ cathode-supported SOECs with thin electrolytes facilitated CO<sub>2</sub>/H<sub>2</sub>O co-electrolysis, achieving current densities up to 800 mA/cm<sup>2</sup> at 850 °C and reducing ohmic losses without compromising structural stability. Fuel cell operation with hydrogen and biogas yielded up to 600 mA/cm<sup>2</sup>.

### Acknowledgements

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## Impact of metal loading on the electrocatalytic behavior of carbon-supported Bi nanoparticles toward CO<sub>2</sub>-to-formate conversion

Elena Bujedo-Saiz<sup>a,\*</sup>, Jose Solla-Gullón<sup>a</sup>, Jesús Iniesta<sup>a</sup>

<sup>a</sup> Institute of Electrochemistry, University of Alicante, Apdo 99, 03080, Spain  
\* email: elena.bujedo@ua.es

### Abstract

The electroreduction of CO<sub>2</sub> has emerged as a promising strategy for obtaining value-added products, including formic acid and formate, methanol, ethylene, and carbon monoxide. Among these, formic acid has a wide range of applications in several industries (pharmaceutical, animal feed, leather tanning) and potential utilization as a hydrogen carrier in low-temperature fuel cells, which makes it a highly relevant electroreduction product<sup>(1)</sup>. Among the catalysts reported for CO<sub>2</sub> electroreduction to formate, Bismuth-based electrodes have gained great attention due to its low toxicity, low cost, high stability, and their electrocatalytic performance (near 100% faradaic efficiencies at relatively low overpotentials)<sup>(2)</sup>. In previous contributions, easily prepared carbon-supported bismuth nanoparticles<sup>(3)</sup> have been extensively tested as cathodes, reaching competitive faradaic efficiency and formate production values<sup>(4-6)</sup>. In this work, we systematically evaluate the effect of the metal (Bi) loading on the electrocatalytic properties for the CO<sub>2</sub> to formic acid/formate electrochemical valorization. Different Bi loadings between 10 and 80 wt% were prepared, physicochemically characterized, and their electrocatalytic activity in CO<sub>2</sub> electroreduction to formate was studied in an electrochemical H-cell at different cell potentials, aiming to identify the optimal Bi nanoparticle-to-carbon ratio that performs better faradaic efficiencies and formate production. The results obtained will be analyzed and discussed in terms of activity and selectivity towards formic acid/formate<sup>(7)</sup>.

### Acknowledgements

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## Fe-N-C electrocatalysts derived from *Posidonia oceanica* waste for electrochemical nitrate reduction to ammonia

Andrea Cárdenas-Arenas<sup>a,\*</sup>, Miguel A. Montiel<sup>a</sup>, Vicente Montiel<sup>a</sup>, Enrique Herrero<sup>a</sup>, José Solla-Gullón<sup>a</sup>

<sup>a</sup> Instituto Universitario de Electroquímica, Universidad de Alicante, Apt. 99, Alicante, E-03080, Spain

\*Presenting author's email: acardenas.arenas@ua.es

### Abstract

The global climate crisis and the depletion of natural resources have made the development of a sustainable energy economy imperative, requiring the use of environmentally friendly materials with minimal environmental impact [1]. In this context, the valorisation of biomass waste has emerged as a promising strategy. *Posidonia oceanica* is an abundant aquatic plant in the Mediterranean Sea that generates large amounts of dead leaves, which accumulate along coastlines and must be removed during the tourist season. This biomass represents a renewable, low-cost and highly available carbon precursor [2]. In this work, a simple route to produce porous carbons from *Posidonia oceanica* fibre waste and their subsequent conversion into Fe-N-C electrocatalysts is presented. These materials were prepared using a hydrochar obtained through the hydrothermal carbonisation of *Posidonia oceanica* waste as the carbon precursor. N-C materials were synthesised by pyrolysis of the hydrochar with melamine as the nitrogen precursor and a Mg<sup>2+</sup> salt as a templating agent to generate porosity and well-defined active sites. Fe-N-C materials were then obtained by low-temperature wet impregnation under reflux in methanol using FeCl<sub>2</sub> as the iron precursor. The resulting materials were characterised and evaluated as electrocatalysts for ammonia production via the electrochemical reduction of nitrate, a reaction that has attracted considerable interest in recent years [3]. The results demonstrate the strong potential of these Fe-N-C materials, derived from *Posidonia oceanica* waste, for the electrochemical production of ammonia through nitrate reduction.

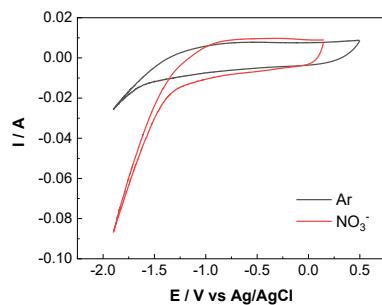


Figure 1. Cyclic voltammograms of the Fe-N-C electrocatalyst supported on carbon paper recorded in an H-type cell at 50 mV s<sup>-1</sup> in Ar-saturated 0.1 M K<sub>2</sub>SO<sub>4</sub> without NO<sub>3</sub><sup>-</sup> (black) and with NO<sub>3</sub><sup>-</sup> (0.5 M KNO<sub>3</sub>) (red).

### Acknowledgements

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## Platinum recycling as a pathway to more cost-competitive PEM technologies

Juan Carlos Carmona Pulido<sup>a,\*</sup>, Irene Peñas Fuertes<sup>a</sup>, Julia Isidro Elvira<sup>a</sup> and Jesús Rodríguez Ruiz<sup>a</sup>

<sup>a</sup> Scientific Development Unit, National Hydrogen Center, Prolongación Fernando el Santo s/n, Puertollano, Spain  
\* jc.carmona@cnh2.es

### Abstract

Analyses by organizations such as the International Renewable Energy Agency (IRENA) and the U.S. Department of Energy (DOE) indicate that achieving cost-effective and scalable green hydrogen production and end-use, through proton exchange membrane (PEM) technologies, requires a substantial reduction in platinum group metal (PGM) usage (1). Target PGM loadings must decrease dramatically in both PEM electrolyzers and fuel cells, motivating strategies that combine catalyst reduction with the recovery and recycling of critical materials.

Addressing these ambitious targets, our research group has initiated the formulation and optimization of low-loading platinum-based catalyst inks for PEM electrolysis, building upon prior experience in reducing platinum content in PEM fuel cells (2). Additionally, this work proposes the development of a methodology for platinum recovery from the porous transport layer (PTL), in contrast to many methods reported in the literature, which typically recover platinum from the nafion membrane.

Pt/C catalysts were recovered from exhausted titanium PTLs via mechanical and ultrasonic treatment, reformulated into inks, and airbrush-deposited onto various substrates. Electrochemical activity was assessed ex situ via cyclic voltammetry, including crucial parameters like the Specific Electrochemical Active Surface Area (SECSA) and in situ in PEM fuel cells, including polarization curves and impedance measurements. Further characterization was driven using SEM-EDS analysis.

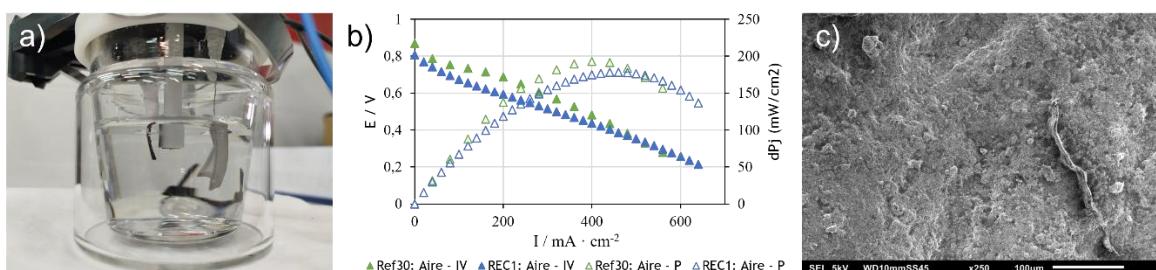


Figure 1. a) 3 Electrode Set-up; b) PEMFC results with recycled Pt; c) SEM image for a recycled Pt sample.

This study presents both a novel protocol for the recovery of Pt/C catalyst supported on PTLs and critical insights into the use of recycled inks and their impact on the electrochemical performance of PEM fuel cells.

Future work will focus on further optimizing the recovery protocol and determining the amount of recycled catalyst required to achieve a functional cell (both electrolyzer and fuel cell) comparable to a new one. Long-term studies will also be conducted to assess the degradation behavior of cells fabricated with recovered catalysts.

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## Photoelectroreduction of CO<sub>2</sub> using catalysts synthesised in a supercritical medium

I. Carrillo, M.A. Ortiz, F. Martínez, R. Camarillo, C. Jiménez, I. Asencio and J. Rincón

Universidad de Castilla-La Mancha, Faculty of Environmental Sciences and Biochemistry, Department of Chemical Engineering, Avda. Carlos III, s/n, 45071 Toledo, Spain  
e-mail: ivan.carrillo@uclm.es

In recent decades, the increase in anthropogenic CO<sub>2</sub> emissions into the atmosphere is having a major impact on global warming, to the point that it has become one of the main environmental problems today. Therefore, measures have been proposed to tackle this problem based on CO<sub>2</sub> capture, utilisation and conversion technologies. One of the conversion strategies is photoelectrocatalytic reduction (PEC) of CO<sub>2</sub>. It is generally based on the use of a semiconductor photoanode that is irradiated by light with an energy equal to or greater than that of its band gap, with the system simultaneously being subjected to a potential difference. Titanium dioxide (TiO<sub>2</sub>) is the most widely used photocatalyst in the literature, as it is stable, low in toxicity, low in cost and semiconductor in nature, with a band gap energy value in the UV and visible ranges. One of the most important challenges in a PEC process is to develop photocatalysts and electrocatalysts that allow maximum process performance to be achieved at both electrodes. An interesting alternative from the point of view of photocatalyst and electrocatalyst synthesis is the use of technologies based on supercritical fluids, whose properties can be easily adjusted with simple modifications in pressure and temperature<sup>[1]</sup>. Moreover, these catalysts exhibit better physico-chemical properties compared to those synthesised under non-supercritical conditions<sup>[2]</sup>.

Thus, in this study, TiO<sub>2</sub> with 6.5% Ni (TiO<sub>2</sub>/Ni 6.5%) was used as an anodic photocatalyst and CuCNT as a cathodic electrocatalyst synthesised in a supercritical medium for CO<sub>2</sub> reduction in a photoelectroreduction cell.

The cell used for the photoelectroreduction of CO<sub>2</sub> was designed by our research group. It is divided into two compartments (an anode and a cathode), separated by a proton exchange membrane (Nafion®). In both electrodes, the catalyst is deposited onto a piece of carbon paper to form the gas diffusion electrode (GDE). A 0.5 M KHCO<sub>3</sub> solution is circulated between the GDE acting as the cathode and the membrane as the catholyte. The gas (CO<sub>2</sub>) flows between the GDE and the external plate of the cell, using a series of gaskets that allow the independent circulation of gas and electrolyte in each compartment. The GDE serving as the anode is assembled onto the proton exchange membrane, and a 0.5 M KOH solution is circulated as anolyte. A quartz window is placed on the anodic compartment plate, allowing light from a Xe arc lamp (solar light simulator) to reach the photocatalyst. The gas phase leaving the cell is sent to a microGC to determine the different reaction products and their concentration, while the liquid phase (catholyte) is collected for further analysis by GC-FID, after a previous stage of preconcentration by solid phase microextraction (SPME), to determine the concentration of oxygenated hydrocarbons, and by HPLC to determine the concentration of formic acid.

On/off experiments were carried out at 20-minute intervals at voltages of 2, 3, and 4 V to observe photocurrent generation. In addition, to evaluate the stability of the catalysts, 6-hour experiments were conducted at 2, 2.5, and 3 V. The main reduction products obtained were formic acid and CO. An increase in the conversion rate of the products obtained was observed with increasing applied potential.

### Acknowledgements

The authors gratefully acknowledge grants PID2019-111416RB-I00, PID2023-147121OB-I00 funded by MCIN/AEI/10.13039/501100011033 (co-funded with European Union through FEDER) and SBPLY/23/180225/000204 (Regional Government of Castilla-La Mancha, co-funded by EU through FEDER).

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## A correlative microscopy strategy for investigating electrochemical processes at the nanoscale.

Marine Cheng<sup>a,\*</sup>, Alexandros Ploumistas<sup>b</sup>, Nikan Afsahi<sup>a</sup>, Jean-Marc Noël<sup>a</sup>, Nathaly Ortiz Peña<sup>b</sup>, Jean-François Lemineur<sup>a</sup>

<sup>a</sup> Université Paris Cité, ITODYS, 75013 Paris, France

<sup>b</sup> Université Paris Cité, Laboratoire Matériaux et Phénomènes Quantiques, 75013 Paris, France

\*marine.cheng@u-paris.fr

### Abstract

Improving the electrochemical activity of catalysts for the hydrogen evolution reaction (HER) is critical for using hydrogen in energy storage and conversion technologies. One popular strategy relies on nanostructured materials and nanoparticles (NPs). However, this approach raises new questions: are all NPs catalytically active and homogeneous? Are they stable under operating conditions?

To address these questions, we developed an electrochemical methodology combining local electrochemical measurement using the scanning electrochemical cell microscopy (SECCM) with high-resolution electron microscopy. Small pipets filled with the electrolyte are used to probe the local chemical activity of single/small groups of NPs while the physical and chemical changes induced by the electrochemical stress are then assessed by identical location imaging (before and after electrochemistry).<sup>1</sup>

This methodology is exemplified herein through the study of the local electrochemical behavior and the structural stability of multimetallic CoPtNi NPs towards HER in acidic conditions (0.1 M perchloric acid). The NPs are obtained by pulsed laser deposition followed by flash laser annealing.<sup>2</sup> The correlative microscopy data that are collected should then help to establish structure-activity and structure-stability relationships at the nanometer scale.

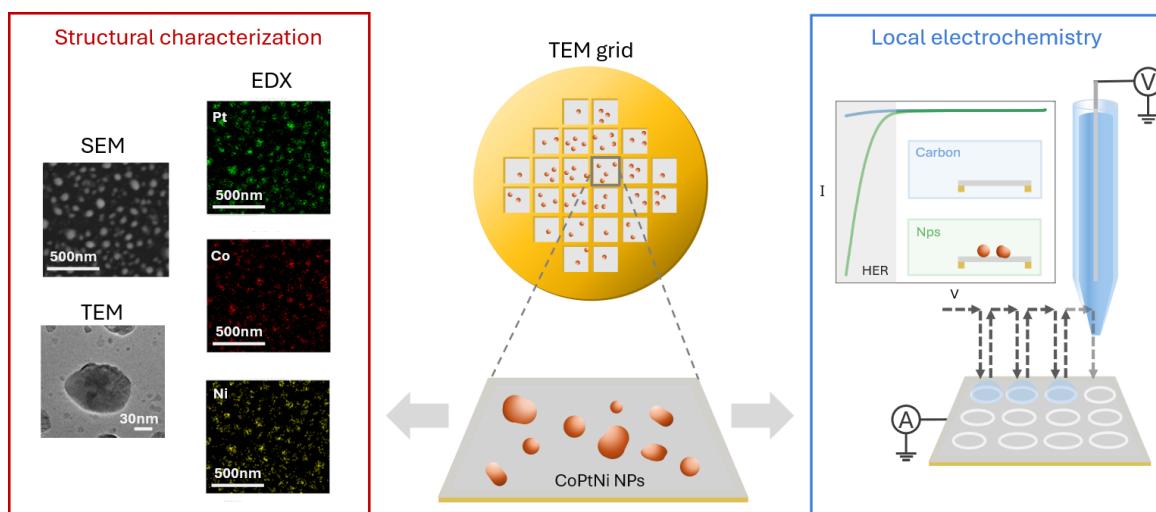


Figure 1. Scheme of the employed methodology. CoPtNi NPs are deposited on a TEM grid, which is then used both as a working electrode for SECCM and as a marked substrate for identical location imaging.

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## Electrochemical characterization of chloride and sulfate posolytes for All-Iron Redox Flow Batteries

Mireia Cifre Herrando<sup>a,\*</sup>, Carles Escoms Espert<sup>a</sup>, Fermín Sáez Pardo<sup>a</sup>, Juan José Giner Sanz<sup>a</sup>, Valentín Pérez Herranz<sup>a</sup>

<sup>a</sup>IEC group, Depto. Ingeniería Química y Nuclear, Universitat Politècnica de València, Camí de Vera s/n, Valencia 46022, Spain.

[\\*mcifher@upvnet.upv.es](mailto:mcifher@upvnet.upv.es)

### Abstract

As the global demand for clean and renewable energy grows, technologies capable of storing intermittent solar and wind power have become essential. Redox Flow Batteries (RFBs) are among the most promising large-scale storage solutions due to their long cycle life, high safety, flexible design, and the decoupling of power and energy. Among RFBs, All-Iron Redox Flow Batteries (AIRFBs) have attracted increasing attention since iron is abundant, inexpensive, and environmentally friendly. In AIRFBs, both half-cells rely on iron-based redox couples, minimizing cross-contamination effects and reducing electrolyte costs.

Despite these advantages, AIRFB performance is limited by several factors, including the low solubility of ferric species in aqueous electrolytes, and the narrow pH stability window of  $\text{Fe}^{3+}$ , as values above pH 3 promote hydrolysis and precipitation of iron hydroxides. In addition, the coulombic efficiency is reduced due to parasitic side reactions such as hydrogen evolution in the posolyte. A central challenge is the slow kinetics of the  $\text{Fe}^{3+}|\text{Fe}^{2+}$  redox couple, which increases activation losses. Therefore, optimizing the posolyte formulation is a key strategy for improving AIRFB performance. An ideal posolyte requires fast and reversible  $\text{Fe}^{3+}|\text{Fe}^{2+}$  kinetics, high selectivity toward the desired reaction and efficient mass transport.

Chloride- and sulfate-based electrolytes are the most widely investigated systems because they are inexpensive, highly soluble, and compatible with strongly acidic operating conditions. However, direct and controlled comparisons between both electrolytes (under identical concentrations and pH) remain limited, and significant variability in literature-reported kinetic parameters makes it difficult to establish consistent trends.

This work presents a systematic electrochemical characterization of chloride and sulfate posolytes under controlled and reproducible conditions, focusing on the kinetic and mass transport parameters of the  $\text{Fe}^{3+}|\text{Fe}^{2+}$  redox couple in these two electrolyte types. Cyclic voltammetry (CV) was used to obtain: the standard rate constant ( $k^0$ ), the charge-transfer coefficient ( $\alpha$ ), and the diffusion coefficient ( $D$ ). Significant differences were observed between electrolytes. Sulfate electrolytes yielded better charge-transfer coefficients, whereas chloride electrolytes enabled faster standard rate constants. In contrast, the electrolyte type exerted a more modest influence on mass transport, with diffusion coefficients remaining comparatively similar across media.

Overall, the study demonstrates that the electrolyte type plays a decisive role in the electrochemical behavior of the  $\text{Fe}^{3+}|\text{Fe}^{2+}$  redox couple. The insights obtained here contribute to the rational formulation of improved posolytes and support the development of higher-performance AIRFB systems.

### Acknowledgements

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## Electrocatalytic Performance of the Hybrid PEDOT@Ni Material toward Ammonia Oxidation in Alkaline Medium

Gonzalo Cima<sup>a,c\*</sup>, José R. Sosa<sup>a,c</sup>, Erik Castañeda<sup>a,c</sup>, Nicolás Veloso<sup>a,c</sup>, María J. Aguirre<sup>b,c</sup>, Mauricio Isaacs<sup>a,c</sup>, Juan F. Armijo<sup>a,c</sup>

<sup>a</sup> Escuela Química, Facultad de Química y Farmacia, Pontificia Universidad Católica de Chile, Av. Vicuña Mackenna 4860, Macul, Santiago, Chile

<sup>b</sup> Departamento de Química de los Materiales, Facultad de Química y Biología, Universidad de Santiago de Chile (USACH), Santiago, Chile

<sup>c</sup> Millennium Institute on Green Ammonia as Energy Vector - MIGA (ICN2021\_023), Pontificia Universidad Católica de Chile, Av. Vicuña Mackenna 4860, Macul 7820436, Santiago, Chile

\*Presenting author's email: [gcima@uc.cl](mailto:gcima@uc.cl)

### Abstract

The electrochemical oxidation of ammonia (AOR) is a promising route for sustainable energy conversion in direct ammonia fuel cells (DAFCs) (1). A hybrid electrocatalyst composed of poly(3,4-ethylenedioxothiophene) (PEDOT) and nickel species (PEDOT@Ni) was synthesized through electropolymerization of EDOT on carbon microfibers followed by pulsed electrodeposition of Ni from an acidic  $\text{Ni}(\text{NO}_3)_2$  solution. FE-SEM, XPS and FTIR-ATR analyses confirmed a homogeneous film and the coexistence of  $\text{Ni}(\text{OH})_2/\text{NiOOH}$  phases after activation in 1.0 mol·L<sup>-1</sup> KOH.

Electrochemical measurements revealed a stable voltammetric response and increased current density compared to pristine PEDOT (Figure 1a). The hybrid material exhibited an electrochemically active surface area of 113.25 cm<sup>2</sup>, surface coverage of 1.77 nmol·cm<sup>-2</sup>, and a charge transfer constant ( $k_s$ ) of 16.68 s<sup>-1</sup>, comparable to nickel hydroxide/oxyhydroxide systems. In the presence of ammonia, the oxidation onset appeared at 1.45 V vs. RHE, achieving 36.9% ammonia conversion after 2 h of electrolysis (Figure 1b).

The synergistic interaction between PEDOT and  $\text{Ni}(\text{OH})_2/\text{NiOOH}$  enhances charge transfer and catalytic activity, making PEDOT@Ni a promising, low-cost anode candidate for DAFCs and other alkaline energy applications.

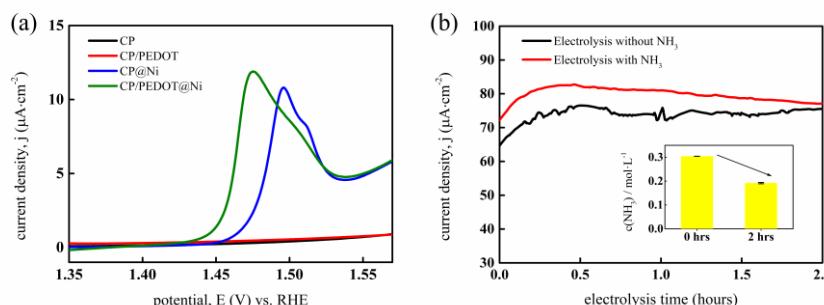


Figure 1. All in 1.0 mol·L<sup>-1</sup> KOH + 0.3 mol·L<sup>-1</sup>  $\text{NH}_3$ . (a) LSV curves at a scanning rate of 0.002 V s<sup>-1</sup>: CP (black), CP/PEDOT (red), CP@Ni (blue), and CP/PEDOT@Ni (green). (b) Electrolysis ammonia - Plot current density-time (inset bar graph of ammonia concentration and time)

### Acknowledgements

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## MIP-PDA platform for an impedimetric determination of Bentazon

Marina Córdoba-Aceituno<sup>a,\*</sup>, Carmen M. Cuesta-Chamorro<sup>a</sup>, Guadalupe Sánchez-Obrero<sup>a</sup>, Rafael Madueño<sup>a</sup>, Manuel Blázquez<sup>a</sup>, Teresa Pineda<sup>a</sup>, Rafael Del Caño<sup>a</sup>

<sup>a</sup>Departamento de Química Física y Termodinámica Aplicada, Instituto Químico para la Energía y el Medio Ambiente, Facultad de Ciencias, Universidad de Córdoba, 14014 Córdoba, España

\*e-mail: [q22coacm@uco.es](mailto:q22coacm@uco.es)

### Abstract

In this work, we present a simple, low-cost and non-destructive sensing methodology based on the electropolymerization of dopamine (DA) in the presence of Bentazon (BZ) to fabricate a molecularly imprinted polymer (MIP) film on a glassy carbon electrode (1). The controlled cyclic voltammetry of DA using 3 scans leads to the formation of a polydopamine (PDA) matrix containing cavities with size, shape and stereochemical complementarity to BZ. This enables precise molecular recognition, adequate probe permeation and efficient mass transport. After template extraction, the resulting PDA-MIP exhibits excellent permeability, structural stability and high affinity toward BZ, allowing its detection through electrochemical impedance spectroscopy (EIS) using  $[\text{Fe}(\text{CN})_6]^{3-}/[\text{Fe}(\text{CN})_6]^{4-}$  as a redox probe.

The resulting sensor exhibits LOD and LOQ of 0.12 and 1.07 pM, respectively, in ultrapure water and demonstrates high selectivity, with minimal interference from other pesticides. The feasibility of the platform is validated by detecting BZ in tap water, achieving a linear response ( $R^2 = 0.994$ ) from 1 to 30 pM, LOD and LOQ of 0.27 and 3.46 pM, respectively. Only slight matrix effects are observed, confirming the robustness of the PDA-MIP sensor. Furthermore, the sensor displays good reproducibility (RSD 4.2%) and can be reused up to 4 times without significant performance loss.

Overall, this study establishes a highly sensitive, selective, and easy-to-fabricate PDA-based MIP sensor for the impedimetric determination of BZ in environmental samples. In addition, the methodology's versatility and tunability highlight its broad potential for the detection of other pesticides and a wide range of molecular targets.

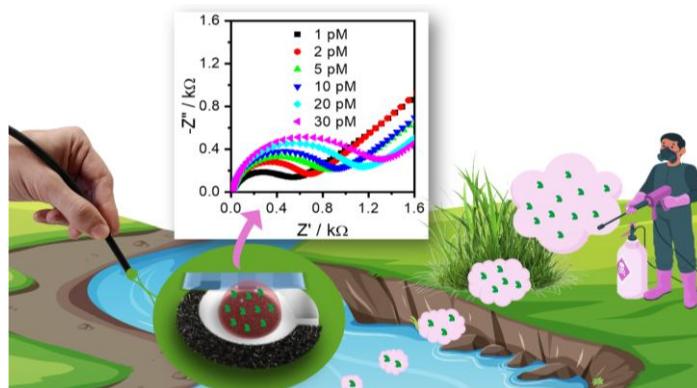


Figure 1. Scheme for BZ detection by EIS using the developed MIP.

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Ministerio de Ciencia e Innovación (Project RED2022-134120-T Red de Excelencia de Sensores y Biosensores electroquímicos), Programa FEDER Andalucía 2021-2027, Consejería de Universidad, Investigación e Innovación de la Junta de Andalucía (PP2F\_L1\_09) y Universidad de Córdoba. Proyecto PID2024-157222NA-I00 financiado por MICIU / AEI / 10.13039/501100011033 / FEDER, UE.

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## Advancing towards selective and stable solar-driven CO<sub>2</sub> and glycerol co-valorisation with multilayered photoanodes

Sara Crespo<sup>a,\*</sup>, Ivan Merino-Garcia<sup>a</sup>, Elena Bujedo-Saiz<sup>b</sup>, José Solla-Gullón<sup>b</sup>, Jonathan Albo<sup>a</sup>

<sup>a</sup> Departamento de Ingenierías Química y Biomolecular, Universidad de Cantabria, España

<sup>b</sup> Instituto Universitario de Electroquímica, Universidad de Alicante, España

\*E-Mail: [sara.crespo@unican.es](mailto:sara.crespo@unican.es)

### Abstract

This work reports the development of back-illuminated multilayered photoanodes engineered to enhance efficiency, charge transport, and stability for photoelectrochemical glycerol oxidation (PEC-GOR). These electrodes consist of a CaTiO<sub>3</sub>/WO<sub>3</sub>/BiVO<sub>4</sub> heterojunction, with the top layer decorated with Ni, Co, or Ni-Co nanoparticles synthesized via a chemical-reduction method. The design aims to improve surface reaction kinetics, reduce the anodic overpotential, and promote selective GOR, enabling energy-efficient CO<sub>2</sub>-to-formate conversion at the cathode within a co-valorisation framework [1,2].

PEC characterization (CP, LSV, EIS, IPCE) and performance tests are conducted under visible light in a filter-press reactor to evaluate the influence of Ni, Co, and Ni-Co (50:50) coatings on photoanodic activity, stability, full energy efficiency (FEE), and GOR product distribution, with particular attention to formate at the cathode and the simultaneous formation of dihydroxyacetone (DHA) at the anode.

The results show how Ni coating achieves a superior FEE (~35%), attributed to a high Faradaic Efficiency (FE) (>90%) for HCOO<sup>-</sup> production compared to Co coating. Meanwhile, the Ni-Co (50:50) decoration proves effective in lowering anodic overpotential. Consequently, pending a full quantification of GOR products, these findings suggest a compromise between maximizing global energy efficiency and optimizing the generation of target value-added chemicals. Ongoing studies on Ni/Co ratio adjustment and parameter optimization aim to clarify this balance.

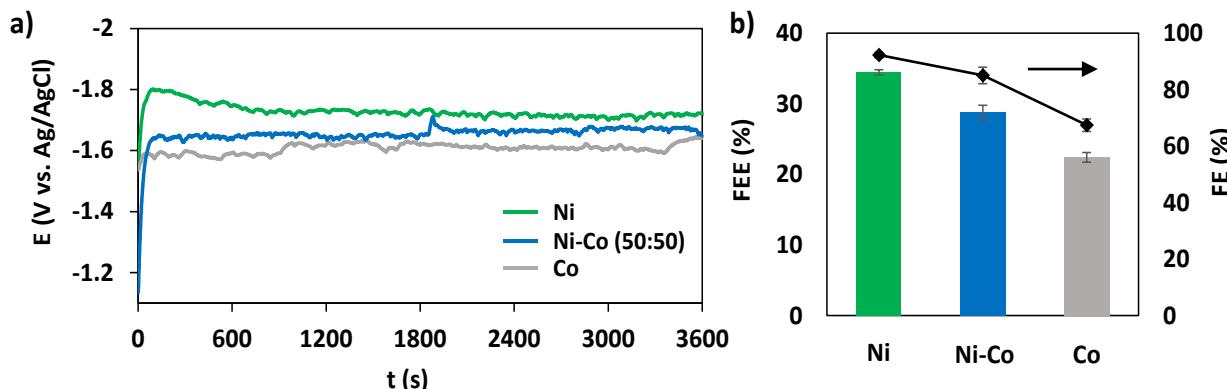


Figure 1. a) CP tests at -45 mA cm<sup>-2</sup> and, b) FEE and FE for CO<sub>2</sub>-to-formate production across the different coatings.

### Acknowledgements

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## The role of hydrogen peroxide in the electrochemical degradation of polystyrene nanoplastics from urban treated wastewater

A. Pérez-López, F. Cruz-Gómez\*, M. Navarro, C.M. Domínguez, A. Santos, S. Cotillas

Department of Chemical and Materials Engineering, Faculty of Chemical Sciences, Complutense University of Madrid, Avenida Complutense S/N, 28050 Madrid, Spain  
 \*fermincr@ucm.es

Nanoplastics are plastic particles under 1,000 nm in size, generated from chemical and physical degradation of plastic waste. Unlike microplastics, nanoplastics are more challenging to detect and remove due to their small size and greater surface area, which enhances their ability to interact with other pollutants. Due to their presence in aquatic environments, including urban-treated wastewater, they have become an emerging environmental concern (Gigault et al., 2018).

Wastewater treatment plants (WWTPs) are not designed to eliminate nanoplastics despite their efficiency in removing conventional pollutants. These nanoparticles can easily bypass filtration systems, posing risks to freshwater and marine environments. Their small size enables them to penetrate the cell membranes of aquatic organisms, potentially leading to cellular damage, oxidative stress, ... (Wu et al., 2022). Furthermore, these particles can be found in surface water intended for consumption, which can lead to their ingestion by humans through contaminated water sources of food. Although the long-term effects of nanoplastics on human health are still under investigation, early studies suggest that chronic exposure may contribute to inflammation, immune system disruption, and other adverse health effects (Bruno et al., 2024).

Given the growing concern about the persistence and impact of nanoplastics, novel technologies are urgently needed to remove or degrade these particles from wastewater. Since traditional physicochemical treatments lack effectiveness in addressing the challenges posed by the removal of nanoplastics; applying Electrochemical Advanced Oxidation Processes (EAOPs) could pose as a good alternative for the removal or degradation of nanoplastics from wastewater.

EAOPs are based on producing highly reactive radicals from water oxidation (hydroxyl radical) and the oxidation of the ions naturally contained in the effluents. These species significantly contribute to the degradation of organic pollutants without adding chemicals. Among EAOPs, technologies such as electrochemical oxidation (EO) or electro-Fenton (EF) have been proven efficient in removing organic pollutants such as pesticides or pharmaceuticals from industrial and urban wastewater. In particular, the EF process is based on producing large amounts of free hydroxyl radicals by the decomposition of in-situ electrogenerated hydrogen peroxide through the Fenton reaction (Eq. 1) (Martínez-Huitle et al., 2023).



With this background, this work compares the degradation of polystyrene nanoplastics in synthetic urban treated wastewater by electrochemical oxidation (EO), EO enhanced by in-situ hydrogen peroxide generation (EO-H<sub>2</sub>O<sub>2</sub>) and electro-Fenton (EF). The promising results of electro-Fenton led to a study to optimize operating conditions on nanoplastics degradation, such as influence of the anode and cathode material, current intensity and the catalyst dose. Results showed that nanoplastics were mineralized by both EO-H<sub>2</sub>O<sub>2</sub> and EF process under moderate conditions.

### Acknowledgements

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## Photoelectrochemical hydrogen generation via water splitting for the catalytic conversion of levulinic acid into $\gamma$ -valerolactone

Elianny Da Silva <sup>a,\*</sup>, Adrián García <sup>a</sup>, Ramón M. Fernández-Domene <sup>a</sup>, Benjamin Solsona <sup>a</sup>, Rita Sánchez-Tovar <sup>a</sup>

<sup>a</sup> IQCATL Group (Heterogeneous Catalysis), Chemical Engineering Department (ETSE), Universitat de València, Av. Universitat s/n, 46100 Burjassot-Valencia, Spain  
\*Presenting author's email: elianny.silva@uv.es

### Abstract

The photoelectrochemical generation of  $H_2$  offers a sustainable alternative to drive the transformation of compounds into higher value-added products. In this regard, this study proposes the generation of  $H_2$  via water splitting to perform the catalytic hydrogenation of levulinic acid (LA) into  $\gamma$ -valerolactone (GVL), a relevant platform molecule.

$WO_3$  nanostructures were synthesized by electrochemical anodization in an electrolyte containing 1.5 M  $CH_4SO_3$  and different concentrations of tartaric acid (0, 0.05, 0.1 and 0.3 M TA). Changes in the electrolyte composition modified the morphology of the nanostructures, producing nanoplates whose size decreased with increasing acid concentration (Figure 1A). Electrochemical impedance spectroscopy (Figure 1B) showed that the 0M TA sample exhibited higher charge-transfer resistance, a behavior associated with lower photoelectrochemical performance. Figure 1C shows that the 0.1 M TA sample had the best photoelectrochemical response.

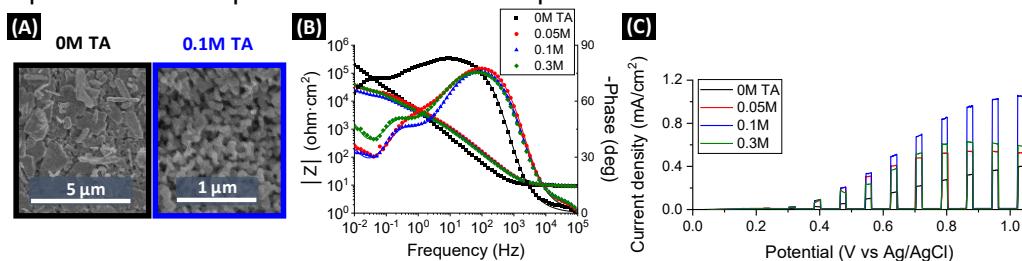


Figure 1. (A) FESEM images of the  $WO_3$  nanostructures, (B) Electrochemical impedance spectroscopy for  $WO_3$  nanostructures (Bode diagram), (C) Water splitting (AM 1.5 illumination).

The nanostructures were integrated into a photoelectrochemical system (Figure 2A) for the in situ generation of  $H_2$  and its use in the transformation of LA into GVL. A ruthenium catalyst supported on alumina (Ru/Alum) was used. With the optimal nanostructure, LA conversion above 75% at 30 °C after 8 h was achieved (Figure 2B), demonstrating that photoelectrochemical  $H_2$  generation is a promising alternative for carrying out the reaction under significantly milder reaction conditions than those typically reported in the literature <sup>1</sup>.

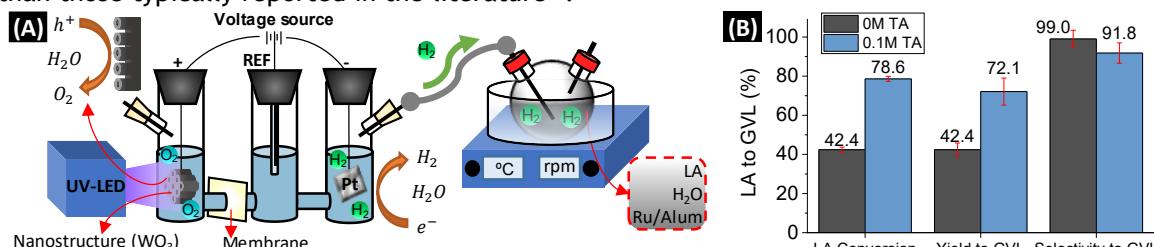


Figure 2. Transformation of LA into GVL: (A) Experimental setup and (B) Catalytic results (at 30 °C during 8 h).

### Acknowledgements

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## Sustainable Titanium Suboxide Anodes Derived from Recycled Additive Manufacturing Titanium for Electrochemical PFAS Remediation

Avik De<sup>a,\*</sup>, Johanne Mouzon<sup>b</sup>, Ivan Carabante<sup>a</sup>

<sup>a</sup> Waste Science and Technology, Luleå University of Technology, Luleå, Sweden

<sup>b</sup> Department of Engineering Sciences and Mathematics, Luleå University of Technology, Luleå

\*email: avik.de@associated.ltu.se

### Abstract

The electrochemical oxidation of per- and polyfluoroalkyl substances (PFAS) requires robust anode materials that combine high electrical conductivity, chemical stability, and resistance to anodic corrosion. Titanium suboxides (TSOs) belonging to the Magnéli phase family are promising candidates. However, their conventional synthesis relies on energy-intensive high-temperature ceramic processing.[1] Here, we present a sustainable route for fabricating conductive titanium suboxide anodes from recycled Ti-6Al-4V powders originating from additive manufacturing waste streams. Sintered titanium alloy pellets were surface-engineered to form substoichiometric titanium oxide layers while preserving a metallic core, resulting in mechanically stable and electrically conductive electrodes. Thermoanalytical and structural characterization confirmed the formation of reduced titanium suboxide phases ( $\text{TiO}_x$ ,  $x < 2$ ) at the electrode surface and the suppression of insulating  $\text{TiO}_2$ . The resulting electrodes exhibited a dark, metallic surface characteristic of Magnéli-type materials, indicating enhanced electronic conductivity. This recycling-based strategy enables the valorization of additive manufacturing titanium waste into functional anodes and provides a promising, resource-efficient pathway toward scalable electrochemical PFAS remediation technologies.

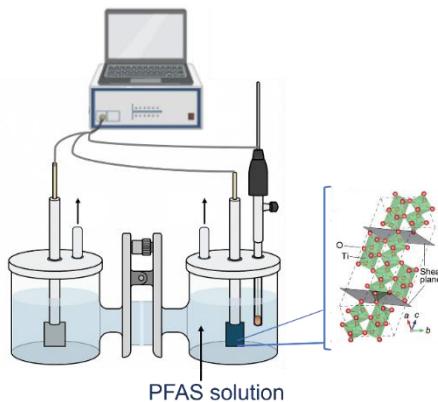


Figure 1. Schematic representation of TSO anode for electrochemical PFAS oxidation

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## Low electrolyte concentration effect on electrochemical reduction of CO<sub>2</sub> in microfluidic undivided reactor

Andrea Di Franco<sup>a\*</sup>, Paola Meli<sup>a</sup>, Federica Proietto<sup>a</sup>, Alessandro Galia<sup>a</sup>, Onofrio Scialdone<sup>a</sup>

<sup>a</sup>Dipartimento di Ingegneria, Università degli Studi di Palermo, Viale delle Scienze Ed. 6, Palermo, Italy

\*[andrea.difranco04@unipa.it](mailto:andrea.difranco04@unipa.it)

### Abstract

The electrochemical reduction of CO<sub>2</sub> (ERCO2) into formic acid (FA) using a tin electrode was widely studied as a sustainable and green route to convert a greenhouse gas into value-added products<sup>1</sup>. It was shown that ERCO2's performance in aqueous media depends sensitively on some key parameters, including both the nature and concentration of the supporting electrolyte (SE)<sup>2,3</sup>. However, despite extensive research on ERCO2 process, the specific influence of SE concentration ([SE]) has not yet been fully elucidated. Indeed, both beneficial<sup>4</sup> and adverse<sup>5</sup> effects of [SE] were reported. To determine the optimal [SE] for the ERCO2 into FA on Sn electrodes, a systematic approach was employed in this study using several SEs (NaCl, Na<sub>2</sub>SO<sub>4</sub>, NaHCO<sub>3</sub>, KCl, K<sub>2</sub>SO<sub>4</sub>, KHCO<sub>3</sub>, Cs<sub>2</sub>SO<sub>4</sub>, and Cs<sub>2</sub>CO<sub>3</sub>) at different concentrations (from 0.2 to 500 mM). The use of a microfluidic undivided device, with inter-electrode distance of 120  $\mu$ m, consented to evaluate the role of [SE] while minimizing the influence of ohmic drops and to compare the performance in presence and absence of SE. Experiments were carried out both in galvanostatic (8 mA/cm<sup>2</sup>) and potentiostatic (-1.7, -1.9, -2.1 V vs SCE) mode. It was shown that for all SEs tested, the production of FA depends strongly on [SE], with maximum values consistently observed at low electrolyte concentrations. At very low [SE] (5 mM), the addition of electrolyte to deionized water led, in most cases, to a substantial increase in FA production. Under these conditions, the cation size exerted a large positive effect, whereas the influence of the anion was comparatively modest. Conversely, at [SE] typical of conventional electrolyzers ( $\approx$  100 mM), the addition of electrolyte to deionized water resulted in a reduction in FA production for all SEs, demonstrating that high electrolyte concentrations negatively affect ERCO2 into FA. Moreover, under these conditions, the cation size exerted a small positive effect. Notably, the influence of SE nature was not uniform across concentrations; rather, it varied markedly with [SE], suggesting that the interplay between ionic species and concentration governs the overall electrochemical performance.

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## Perovskite-based symmetric solid oxide cells for co-electrolysis applications

Noelia Díaz<sup>a,\*</sup>, María del Rosario Serrano de la Cruz<sup>a</sup>, Ángela Moratalla<sup>a</sup>, Adrián Pardo<sup>a</sup>, Irene Ayuso<sup>a</sup>, Alfredo Iranzo<sup>b</sup>, Francisco José García-García<sup>b</sup>, Cristina Montes<sup>a</sup>, Roberto Campana<sup>a</sup>

<sup>a</sup> Hydrogen National Centre (CNH2), Puertollano, Spain

<sup>b</sup> Department of Materials and Transport Engineering and Science, University of Seville, Spain

[\\*noelia.diaz@cnh2.es](mailto:noelia.diaz@cnh2.es)

### Abstract

Solid oxide fuel cells (SOFCs) are highly efficient devices that directly convert the chemical energy of various fuels into electricity at high temperatures. Complementarily, solid oxide electrolysis cells (SOECs) are promising technologies for the electrochemical conversion of CO<sub>2</sub> and H<sub>2</sub>O through co-electrolysis, producing synthesis gas and fuels from renewable electricity.

Conventional electrodes, particularly Ni-YSZ anodes, suffer from degradation due to coking, sulfur poisoning, and loss of stability under variable operating conditions. Advanced mixed ionic-electronic conductors (MIECs) [1], exhibit robust structural stability and versatile functionality. Among them, double-layer perovskites, such as PBMO (PrBaMn<sub>2</sub>O<sub>5+δ</sub>), stand out due to their high electrical conductivity, fast oxygen exchange kinetics, excellent redox tolerance, and stability against carbon and sulfur. In this way, symmetric solid oxide cells have emerged as an alternative, employing a single ceramic material as both electrodes, which simplifies fabrication processes, reduces costs, and improves material compatibility, while also enabling their application as reversible electrodes in SOFC/SOEC systems [2].

In this work, different PBMO samples were synthesized via mechanosynthesis of binary oxides. Symmetric cells were prepared on YSZ electrolyte with a protective GDC layer, and the electrode powders, were deposited symmetrically (Figure 1).

The performance of PBMO electrodes was evaluated through electrochemical and microstructural characterization, considering their activity in electrolysis and H<sub>2</sub>O/CO<sub>2</sub> co-electrolysis (Figure 3). Electrochemical measurements were carried out using an open-flanges test set-up. These results demonstrate the electrochemical behavior and durability, providing key insights for the design of efficient and robust electrode materials.

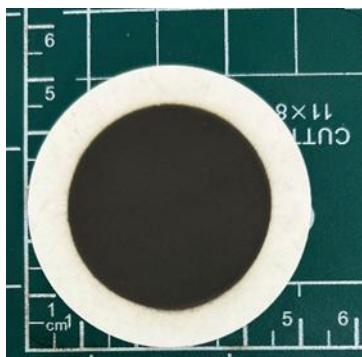


Figure 1. Symmetrical cell with a PBMO-GDC / GDC / YSZ / GDC / PBMO-GDC structure.

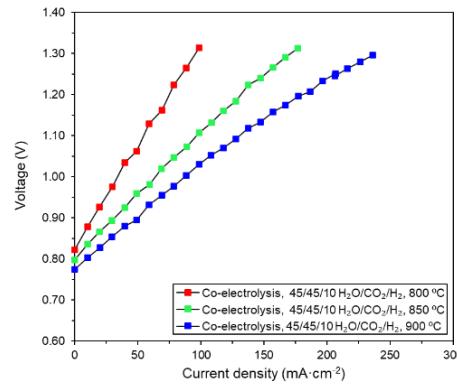


Figure 3. Influence of temperature on the I-V curves of the co-electrolysis process using a gas mixture of 45% H<sub>2</sub>O, 45% CO<sub>2</sub>, and 10% H<sub>2</sub>.

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## Pyrolysis of sludge for the synthesis of biochar used as support of photocatalysts and photoelectrocatalysts

Adrián Díaz-Díaz\*, Carlos Jiménez, Fabiola Martínez, Isaac Asencio, Jesusa Rincón and Rafael Camarillo

University of Castilla La-Mancha, Departament of Chemical Engineering, Faculty of Environmental Sciences and Biochemistry (Campus Fábrica de Armas, Toledo)

\*Presenting author's email: adrian.diaz@uclm.es

Implementing more sustainable processes is crucial to minimizing the environmental impact of human activities. Overexploitation of resources, pollution and climate change require innovative solutions to reduce greenhouse gas emissions and minimize waste. Adopting clean technologies, using energy efficiently, and managing waste responsibly are all essential to preserve ecosystems. This study focuses on the reuse of sewage sludge to produce biochar, which will then be used as a carbon support in the synthesis of catalytic materials. Concretely, the pyrolysis of sludge from the Toledo WWTP (Estiviel WWTP) has been used for this study. Pyrolysis is a thermochemical process carried out in an inert atmosphere, which causes the chemical bonds of the raw material to break down when subjected to temperatures that usually range between 450 °C and 1000 °C. This generates three distinct phases: gaseous, liquid, and solid. The gaseous phase consists of H<sub>2</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>O, etc.; the liquid phase consists of various organic compounds (oils); and, finally, the solid phase consists mainly of biochar, which is essentially carbon. This process can be classified as fast pyrolysis or slow pyrolysis, depending on the heating rate. Fast pyrolysis involves heating rates of around 1000 °C/min, which favors the yield of pyrolytic oils, significantly reducing the production of biochar. On the other hand, slow pyrolysis is usually carried out at heating rates of around 10 °C/min and favors biochar production, resulting in a considerable decrease in pyrolytic oil yield. For this reason, slow pyrolysis (1) has been chosen in this study to maximize biochar production.

The biochar obtained (with a 60 % yield) has been characterized to improve its properties as a catalytic support and has been compared with a commercial counterpart of plant origin (Vermiduero). To this end, the material has been analyzed using a variety of characterization techniques, including ICP, BET surface area, elemental analysis, and FTIR. The electrochemical and photochemical properties of the synthesized biochar were characterized through the utilization of electrochemical impedance spectroscopy, linear sweep voltammetry, and diffuse reflectance spectroscopy. In previous characterization analyses, the synthesized biochar has shown a carbon content of 25 %, as well as the presence of a wide variety of metals (K, Fe, P, Ca...) in addition to a considerable surface area (16-100 m<sup>2</sup>/g according to the pyrolysis temperature). The application of an acid activation process has also been proposed to improve the reproducibility of the process and the surface area. Furthermore, the degree of graphitization of the material will be the subject of study using RAMAN and XRD techniques.

Following the optimisation of the synthesis process, the biochar obtained will be utilised in the synthesis of photo- and electrocatalysts. The objective of this utilisation is to employ these catalysts in the photo-, electro-, and photoelectrocatalytic reduction of CO<sub>2</sub>, with the aim of transforming it into value-added compounds.

### Acknowledgements

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## Integrating ORR electrocatalysts in flow-through electro-Fenton treatment

Ivan Díaz-Redondo<sup>a,b,\*</sup>, Núria López-Vinent<sup>b</sup>, Alberto Cruz-Alcalde<sup>b</sup>, Pere L. Cabot<sup>a</sup>, Ignasi Sirés<sup>a</sup>

<sup>a</sup> Laboratori d'Electroquímica dels Materials i del Medi Ambient, Departament de Ciència de Materials i Química Física, Secció de Química Física, Facultat de Química, Universitat de Barcelona, Martí i Franquès 1-11, 08028, Barcelona, Spain

<sup>b</sup> Processos de Separació, Membranes i Polímers, Departament d'Enginyeria Química i Química Analítica, Secció d'Enginyeria Química, Facultat de Química, Universitat de Barcelona, Martí i Franquès 1-11, 08028, Barcelona, Spain

\*ivan.diaz@ub.edu

The increasing presence of contaminants of emerging concern (CECs) in wastewater effluents demands the development of advanced technologies capable of treating relatively low concentrations in complex matrices. Electro-Fenton (EF) process, based on in-situ electrogeneration of hydrogen peroxide ( $H_2O_2$ ) via the two-electron oxygen reduction reaction (2e<sup>-</sup> ORR) and its subsequent activation into hydroxyl radicals (·OH), has become a highly promising route towards reagent-efficient and decentralized water treatment. However, mass transport limitations, limited catalyst stability and the need for scalable reactors has hindered large-scale deployment so far (1). This comprehensive work explores hybrid systems that integrate C-based electrocatalysts with ceramic membranes (CMs) to develop flow-through electrochemical reactors that couple microfiltration with EF-based degradation (2). Commercial  $Al_2O_3$  membranes are functionalized with electrocatalysts from either main-group and/or transition-metal elements. Notably, Sn-doped carbons have shown increased 2e<sup>-</sup> ORR selectivity in neutral media with efficient EF activation of  $H_2O_2$  to reactive oxygen species (ROS) (3). CMs deposited via thermal spray onto porous Ti supports are engineered to serve simultaneously as filter and cathode, enabling reactive filtration and distributed ROS generation along the membrane cross-section. Structure-activity relationship and stability are assessed through advanced physicochemical characterization and electrochemical testing using both rotating ring-disk electrode (RRDE) and gas-diffusion electrodes (GDEs), providing realistic insights into durability under triple-phase boundary conditions (4). Preliminary flow-through tests showed that modified membranes outperform commercial ones, likely due to improved electrochemical oxidation efficiency arising from enhanced membrane-solution interaction and higher local availability of reactive species. Ongoing work is evaluating pollutant removal, mineralization, kinetics, by-product formation and effluent toxicity, while upcoming coupling with UVA and sunlight irradiation will allow assessing the scalability, long-term stability and energy efficiency for advanced electrochemical wastewater treatment applications.

### Acknowledgements

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## Harvesting thermal energy using activated carbon-based supercapacitors with aqueous electrolyte

Laia Figueres-Fernández<sup>a,\*</sup>, David Salinas-Torres<sup>a</sup>, Raúl Ligero-Peralta<sup>a</sup>, Cristian Daniel Jaimes-Paez<sup>b</sup>, Emilia Morallón<sup>b</sup>, Diego Cazorla-Amorós<sup>a</sup>

<sup>a</sup> Department of Inorganic Chemistry and University Institute of Materials, University of Alicante, Apartado 99, Alicante, E-03080, Spain.

<sup>b</sup> Department of Physical Chemistry and University Institute of Materials, University of Alicante, Apartado 99, Alicante, E-03080, Spain.

\* [laia.figueres@ua.es](mailto:laia.figueres@ua.es)

### Abstract

Improving energy efficiency is essential for the sustainable use of clean energy sources and, among them, the recovery of low-temperature waste heat (<100 °C) can be a relevant contribution [1]. One promising approach to recover that residual heat would be to convert this thermal energy into electricity using supercapacitors, which can significantly enhance overall efficiency and sustainability. Supercapacitors are electrochemical devices that store energy through the formation of an electric double layer in the electrode-electrolyte interface. They stand out for their rapid response to voltage changes, high power density, and long lifespan. Supercapacitors made with carbon-based materials, such as activated carbon, are notable for their high specific surface area and porosity, which enable greater charge storage and contribute to sustainability. In addition, the use of neutral aqueous electrolytes represents a safe and cost-effective alternative. Temperature affects capacitor parameters such as capacitance, energy, and power. An increase in the overall temperature of the capacitor can cause variations in the solvation degree of ions, changes in the viscosity and dielectric constant of the medium, and a redistribution of charges, which may influence the amount of stored energy [2].

This study evaluates the behavior of capacitors prepared with commercial activated carbons as electrodes and an aqueous electrolyte (Na<sub>2</sub>SO<sub>4</sub>). Cyclic voltammetry, chronopotentiometry, and electrochemical impedance spectroscopy tests were carried out at temperatures ranging from 0 to 80 °C. The voltages used in the charge-discharge cycles were below and above 1 V. To evaluate the feasibility of storing and recovering thermal energy, the capacitors were charged at low temperature, and after increasing the temperature, the open-circuit voltage (OCV) was monitored. The capacitors were then discharged to quantify the stored energy. The results reveal that heating the capacitor leads to an increase in the OCV, indicating a conversion of thermal energy into electrical energy that is recovered during the discharge. The maximum energy recovery (95%) occurred at 0.05 M Na<sub>2</sub>SO<sub>4</sub> and 0.6 V charge voltage.

Table 1. Values obtained during the energy recovery study for the capacitor with 0.5 M Na<sub>2</sub>SO<sub>4</sub> electrolyte.

V <sub>charge process</sub> (V)	ΔV OCV (mV)	E <sub>charge</sub> 0°C (Wh/Kg)	E <sub>discharge</sub> 80°C (Wh/Kg)	E <sub>thermal → electrical</sub> (%)
1.2	9	2.22	3.14	41
1.0	21	1.40	2.16	54
0.8	32	0.82	1.35	65
0.6	38	0.37	0.72	95

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## Thermogalvanic energy conversion improvement in ionic liquids: redox solvation and coordination chemistry

Ioannis Giannoutsos<sup>a,b,\*</sup>, Michel Beaughon<sup>a</sup>, Thomas Salez<sup>a</sup>, Tiago Fiúza<sup>a</sup>, Sawako Nakamae<sup>a</sup>, Vladimir Sladkov<sup>b</sup>, Veronika Zinov'yeva<sup>b</sup>

<sup>a</sup> Service de Physique de l'Etat Condensé, CEA, CNRS, Université Paris-Saclay, 91191 Gif-sur-Yvette Cedex, France

<sup>b</sup> IJCLab, CNRS-IN2P3, Université Paris-Saclay, 91406 Orsay Cedex, France

\*Presenting author's email: [ioannis.giannoutsos@ijclab.in2p3.fr](mailto:ioannis.giannoutsos@ijclab.in2p3.fr)

Thermogalvanic cells (or thermocells) utilize a temperature difference across the electrodes immersed in an electrolyte solution to drive the electrochemical (redox) reactions that generate an electric potential and current. Solid thermoelectric devices, on the other hand, function as thermodynamic engines to directly convert heat into electricity or vice versa. For both systems, the first indicator of the thermal-to-electric energy conversion capacity is the materials' Seebeck coefficient; i.e., the ratio between the applied thermal energy ( $\Delta T$ ) and the extracted electric energy ( $\Delta V$ ).

Among different types of thermocells, those made with room temperature ionic liquids not only have high Seebeck coefficient but also present technologically and environmentally desirable properties: e.g., a wide electrochemical range of stability, thermal stability ( $> 200$  °C), low toxicity and volatility, making this class of devices greener and more sustainable. It has been shown that the primary factor affecting the reaction potential in ionic liquids resides in the close vicinity of the redox pair near the electrode surfaces, more so than the liquid medium itself. In the present study we take a closer look at the effect of solvated water molecules in on a bare ion redox pair of europium ( $\text{Eu}^{3+/2+}$ ) and a strongly complexed cobalt,  $[\text{Co}(2,2'\text{-bipyridine})_3]^{3+/2+}$  in 1-ethyl-3-methylimidazolium bis(trifluoromethanesulfonyl)imide (EMI-TFSI) in the range of  $\text{H}_2\text{O}$  content between <500 and 16000 ppm. It is found that depending on the redox species, water molecules can coordinate with the metal ions, leading to marked changes in their structural and electrochemical behavior of the complexes. In the long term, exploring how to utilise the solvation and coordination chemistry of redox species will help advance our understanding of the fundamentals of the thermogalvanic effect in complex media, and simultaneously, enable the fine-tuning of the thermocell efficiency toward their industrial exploitations.

## Life Cycle Sustainability of Electrochemical Hydrogen Compression for High Pressure Green Hydrogen Storage

J.F. Gutierrez-Espinoza<sup>a,\*</sup>, N-E. Rahm<sup>b</sup>, B. Bensmann<sup>b</sup>, C.M. Fernández-Marchante<sup>a</sup>, J. Lobato<sup>a</sup>

a. Chemical Engineering Department, Faculty of Chemical Sciences and Technology, University of Castilla-La Mancha, Avda. Camilo José Cela 12, 13071, Ciudad Real, Spain.

b. Institute of Electric Power Systems (IfES), Faculty of Electrical Engineering and Computer Science, Leibniz University Hannover, Appelstraße 9A, 30167 Hannover, Germany.

\*javiera.gutierrez@uclm.es

### Abstract

Green hydrogen is essential for decarbonizing the energy system and achieving IPCC climate targets by offering a sustainable alternative to fossil fuels. Nonetheless, a major challenge in hydrogen production and deployment is storage, due to its low density and physical properties at atmospheric conditions pressure. Mechanical compression (MC) is the most common storage method but has drawbacks, such as wear from friction in moving parts, hydrogen embrittlement, noise, vibrations, and lubricant contamination. Electrochemical hydrogen compression (EHC) offers a promising alternative for compressing hydrogen to higher pressures. It uses a proton exchange membrane (PEM) with electrodes, where hydrogen at the anode splits into protons and electrons. The protons travel through the PEM to the pressurized cathode side, where molecular hydrogen is reformed. A key advantage of EHC is its near-isothermal compression, which is the most energy-efficient method. EHC operates silently, removes contaminants from hydrogen, and can compress hydrogen from very low to very high pressures in a single process step.[1]

This technology is part of a green hydrogen production system, under the framework of the EU Project MacGhyver, that contemplates a microfluidic electrolyzer, electrochemical hydrogen compressor and a separator. MacGhyver develops a compact, high energy efficiency hydrogen production system along with renewable energy.

This study conducts a Life Cycle Assessment (LCA) of an EHC system to evaluate environmental impacts across its entire value chain, from material extraction to operation and end-of-life disposal. Employing established LCA tools such as ReCiPe, UseTox, IPCC, and AWARE, the analysis measures the environmental footprint of EHC made with various materials tested within this technology. It also examines how different temperatures and pressures during hydrogen compression affect energy costs. This approach aims to identify the most critical components, supporting the development of effective decarbonization solutions. Ultimately, the research seeks to promote green, low-carbon hydrogen systems as a viable answer to global energy challenges.

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## Electrochemical oxidation: a sustainable approach for treating pharmaceutical contaminants in hospital effluents

Ana Hayat\*, Jose L.S. Duarte, Carmen M. Domínguez, Aurora Santos, Salvador Cotillas

*Department of Chemical and Materials Engineering, Faculty of Chemical Sciences, Complutense University of Madrid, Avenida Complutense S/N, 28040 Madrid, Spain*

[\\*anahayat@ucm.es](mailto:anahayat@ucm.es)

### Abstract

The contamination of the aquatic environment is one of the main challenges to be faced by scientists and industries since access to safe water is critical for human survival. More specifically, hospital wastewater is recognized as one of the most hazardous effluents due to its high concentrations of pharmaceuticals and pathogenic microorganisms. In fact, these places can generate a wastewater amount of 200 - 1200 L /bad/day, depending on if it is from a developing or high-income country (1). These drugs enter waterways via urine and/or faeces, either in their metabolized form or unchanged. The management of hospital wastewater is carried out together with urban wastewater, but conventional treatments have demonstrated low efficiencies to remove such pollutants. Additionally, the absence of specific regulations regarding the proper disposal of pharmaceuticals and the inappropriate disposal of expired medicines further exacerbates the prevalence of these pollutants in water, posing challenges to public health and aquatic ecosystems.

Carboplatin (CPt,  $C_6H_{12}N_2O_4Pt$ ) is a platinum-based cytotoxic chemotherapeutic agent widely used in the treatment of various solid tumors (2). Its clinical relevance has increased in recent decades due to the global rise in cancer incidence. In 2022, WHO and IARC reported nearly 20 million new cancer cases and approximately 10 million cancer-related deaths worldwide. Similarly, MRP (MRP,  $C_{17}H_{25}N_3O_5S$ ) is a widely consumed analgesic and antipyretic drug, commonly used to moderate pain and fever. Due to its extensive use and availability in many countries, MRP is frequently detected in wastewater effluents, raising concerns about its persistence and potential environmental impact. In this context, this work focuses on removing MRP and CPt from synthetic hospital effluents by electrochemical oxidation. This process is based on the production of highly reactive species from the electrolysis of the ions commonly found in the effluents that contribute to the degradation of organics. The nature and concentration of reactive species depend mainly on the electrode material used and the applied current density. Mixed Metal Oxides (MMO) was employed as the anode material. The effect of the current density on pollutants degradation was studied (0-50 mA  $cm^{-2}$ ), and the effectiveness of the proposed process on the removal of 5 mg  $L^{-1}$  MRP and CPt was evaluated using HPLC liquid chromatography to follow the decrease in its concentrations.

Other aspects related to the treated effluent have also been investigated through ion chromatography, total organic carbon analysis, toxicity, determination of degradation by-products and of the oxidizing and radical species involved in the degradation. The results obtained show that it is possible to completely remove MRP and CPt using low current densities. The ions contained in the effluent are oxidized during the process, promoting the formation of oxidants and radicals that significantly contribute to the degradation. Furthermore, the degradation byproducts generated during electrochemical oxidation with MMO anodes were identified, and different degradation pathways for the pollutants were proposed.

### Acknowledgements

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## Synthesis of porous carbon materials and their use as electrodes in zinc-ion capacitors

Óscar Jareño-Amorós<sup>a,\*</sup>, Jessica Chaparro-Garnica<sup>a</sup>, David Salinas-Torres<sup>a</sup>, Emilia Morallón<sup>b</sup>, Diego Cazorla-Amorós<sup>a</sup>

<sup>a</sup> Department of Inorganic Chemistry and University Institute of Materials, University of Alicante, Apartado 99, Alicante, E-03080, Spain.

<sup>b</sup> Department of Physical Chemistry and University Institute of Materials, University of Alicante, Apartado 99, Alicante, E-03080, Spain.

[\\*oscar.jareno@ua.es](mailto:oscar.jareno@ua.es)

### Abstract

The increasing demand for sustainable and efficient energy storage systems has promoted the development of electrochemical energy storage technologies capable of complementing intermittent renewable energy sources<sup>1</sup>. Among these, hybrid supercapacitors (HSCs) have emerged as promising devices that combine the advantages of both batteries and electrochemical double-layer capacitors, offering high energy density with long cycling stability.<sup>2</sup> Zinc-ion hybrid supercapacitors (ZHSCs) provide additional benefits such as high availability, low cost, and excellent safety, especially because they may use aqueous electrolytes.<sup>2</sup> In these systems, carbon-based materials play a key role as positive electrodes due to their high surface area, well-developed porosity, and versatile surface chemistry.<sup>3</sup> Developing low-cost and sustainable routes to obtain high-performance porous carbons is therefore essential for advancing these technologies.

In this study, activated carbons were synthesized from biomass waste (almond shells) through physical activation with carbon dioxide and chemical activation using a low-concentration phosphoric acid solution. In addition, the chemically activated carbon underwent a subsequent heat treatment to improve conductivity. Their electrochemical performance was evaluated in aqueous  $ZnSO_4$  electrolyte through cyclic voltammetry, galvanostatic charge-discharge and impedance spectroscopy. The voltage employed in these tests was selected to prevent undesirable side reactions, such as the formation of dihydrogen or dioxygen, particularly in aqueous electrolytes.

The results showed that the chemically activated carbon exhibits a superior electrochemical performance as a hybrid supercapacitor due to its excellent textural properties, such as high surface area, a balanced combination of microporosity and mesoporosity, and the formation of functional groups, which enhance ion accessibility and contribute to pseudocapacitive behavior. In contrast, the physically activated carbon exhibits poor performance, associated with its predominantly microporous texture that limits ionic transport and reduces the overall electrochemical efficiency. Finally, the results demonstrate that chemical activation represents a simple synthesis route for the development of electrochemical devices for energy storage.

### Acknowledgements

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## Carboxylic acids as substrate for microbial PHAs accumulation

Sabrina Valeria Ayala Bueno<sup>a,b</sup>, Juan Jerónimo Merino<sup>a\*</sup>, Raúl García-Cervilla<sup>a</sup>, Patricia Balderas Hernández<sup>b</sup>, Gabriela Roa Morales<sup>b</sup>, Manuel A. Rodrigo Rodrigo<sup>a</sup>

<sup>a</sup> Universidad de Castilla - La Mancha (Ciudad Real, Spain)

<sup>b</sup> Universidad Autónoma del Estado de México (Toluca de Lerdo, Mexico)

\*Presenting author's email: juan.jeronimo@uclm.es

### Abstract

The concept of the electro-refinery emerges from the success of modern biorefineries, yet it differs fundamentally by replacing biological conversion pathways with electrochemical processes. These processes enable the transformation of pollutants or waste materials into valuable products through controlled and selective reactions [1]. Moreover, electrochemical stages do not operate in isolation; they can be integrated with other types of processes, such as membrane-based separations, chemical catalysis, or even bioprocesses, that support the full valorization of the compounds generated. Taken together, this integration positions the electro-refinery as a versatile platform for resource recovery, advanced waste treatment, and the sustainable production of value-added chemicals, thereby expanding technological possibilities beyond those offered by traditional electrochemical approaches.

In this study was evaluated the production of polyhydroxyalkanoates (PHAs) using as substrates several compounds that can be generated during the electrochemical oxidation of organic waste, most of which correspond to carboxylic acids [2]. Specifically, four representative acids were examined: oxalic, maleic, acetic, and formic, selected due to their frequent occurrence in oxidized waste streams and their potential to be assimilated by PHA-accumulating microorganisms. To conduct these experiments, sewage sludge from the Wastewater Treatment Plant of Ciudad Real was employed, taking advantage of the indigenous microbial community as a natural biocatalyst responsible for PHA synthesis. The microorganisms were fed under anaerobic conditions with a mixture of mineral nutrients and the corresponding substrate, maintaining the system anaerobically for 6 hours to promote substrate uptake and intracellular storage. This was followed by an aeration phase, achieved through continuous bubbling for an additional 18 hours, enabling biomass growth and PHA accumulation. At the end of each anaerobic stage, a purge stream was withdrawn, which was expected to contain a fraction of microorganisms enriched in PHAs. This operational strategy allows the assessment of the real capacity of a mixed microbial consortium to convert waste-derived compounds into value-added biopolymers, bringing the process closer to conditions relevant for wastewater treatment and circular-economy applications. PHA are biodegradable, biocompatible plastics with properties similar to petrochemical ones. Research now targets cheaper substrates and extraction methods to lower production costs and improve market competitiveness.

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## Ferrite-loaded activated carbons derived from olive pits for arsenic removal

Luis Laime<sup>a,b,\*</sup>, Hugo Alarcon<sup>b</sup>, Ignasi Sirés<sup>a</sup>

<sup>a</sup> Laboratori d'Electroquímica dels Materials i del Medi Ambient, Departament de Ciència de Materials i Química Física, Secció de Química Física, Facultat de Química, Universitat de Barcelona, Martí i Franquès 1-11, 08028, Barcelona, Spain

<sup>b</sup> Center for Development of Advanced Materials and Nanotechnology, Universidad Nacional De Ingeniería, Av. Tupac Amaru 210, Rímac 15333, Lima, Peru

The present research is focused on the production of activated carbon, prepared from olive seeds as the raw material. Activated carbon was obtained through chemical activation and double carbonization under an inert N<sub>2</sub> atmosphere, at a temperature of 840 °C under a heating rate of 10 °C min<sup>-1</sup>, using potassium hydroxide (KOH) as the activating agent. The prepared activated carbons exhibited specific surface areas ranging from 2592 to 1186 m<sup>2</sup> g<sup>-1</sup>, and micropore volumes from 1.54 to 0.79 cm<sup>3</sup> g<sup>-1</sup>. Increasing the activation temperature to 840 °C, along with a carbon-to-activating agent impregnation ratio of 1:4, resulted in higher surface area and micropore volume. In a future step, this carbon, further impregnated with ferrite, is intended for use as an electrode for arsenic removal, primarily in its As(III) form. This will be performed via heterogeneous electro-Fenton process at the cathode, promoting the oxidation of As(III) to As(V) and subsequent arsenic elimination by coupling with a suitable anode. To assess the adsorption ability of the porous carbon, both particles and 3D-printed structures have been employed. For 3D printing, a mixture of activated carbon as precursor, an alkaline activator such as NaOH, and polyethylene glycol (PEG, 1.7 %), was homogenized in a planetary centrifugal mixer to generate the ink. Under optimized conditions, currently under investigation, As(III) could be efficiently removed, achieving a residual arsenic concentration below the threshold value (10 ppb) recommended by the World Health Organization (WHO) for drinking water consumption.

### Acknowledgements

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## Low-temperature heat-treated nickel oxide for electrooxidation of glycerol in alkaline media

Raul Ligero-Peralta<sup>a,\*</sup>, David Salinas-Torres<sup>a</sup>, Diego Cazorla-Amorós<sup>a</sup>, Emilia Morallón<sup>b</sup>

<sup>a</sup> Departamento de Química Inorgánica, Instituto Universitario de Materiales de Alicante (IUMA), University of Alicante, Ap, 99, 03080 Alicante, Spain.

<sup>b</sup> Departamento de Química Física, Instituto Universitario de Materiales de Alicante (IUMA), University of Alicante, Ap. 99, 03080 Alicante, Spain.  
raul.ligero@ua.es

Glycerol (GLY) is a by-product of biodiesel, and it comprises 10 wt% of biodiesel production<sup>1,2</sup>. OECD-FAO Agricultural Outlook for 2025-2034 points out that the global biomass-based diesel demand is projected to increase up to 80.9 billion liters by 2034. A huge production of GLY has fostered new emerging opportunities for glycerol valorisation, such as electrochemical reforming. This strategy emerges as a powerful tool for obtaining valuable chemical products in the anode under mild experimental conditions<sup>3</sup>, while hydrogen is being produced in the cathode in a safer and cheaper way than water electrolysis<sup>1</sup>. Ni-based catalysts are commonly used in GLY electrochemical oxidation as an alternative to noble metals<sup>3,4</sup>. We report the study of the synthesis of low-temperature heat-treated nickel oxide for the electrooxidation of glycerol in alkaline conditions. The XRD pattern of as-prepared NiO (Figure 1a) displays the characteristic diffraction peaks of NiO, from which a Scherrer crystal size of 5.4 nm was estimated. The TEM image (see inset) confirms the formation of small spherical nanoparticles. Figure 1b depicts steady state voltammograms of prepared NiO in the presence of GLY, which reveal the electrochemical activity of the obtained NiO towards GLY electrooxidation.

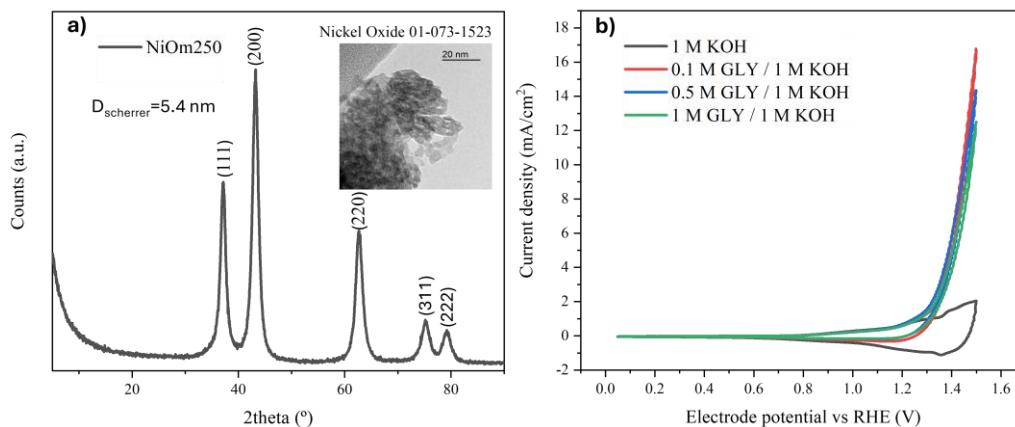


Figure 1. Low-temperature heat-treated NiO: a)XRD pattern (inset: TEM image); b) Steady state voltammograms of NiO electrode at different GLY concentrations, 1 M KOH, v=50 mV/s

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## Influence of cell structural features on the electrochemical behavior of Zero-Gap Alkaline Water Electrolysis

Nerea López-García<sup>a,\*</sup>, Rebeca.M Simion<sup>a</sup>, Alejandro Cánovas<sup>a</sup>, Jesús Rodríguez<sup>a</sup>

<sup>a</sup> National Hydrogen Center, Prolongación Fernando el Santo S/N, Puertollano, Spain

\*nerea.lopez@cnh2.es

### Abstract

One of the most significant advances in Alkaline Water Electrolysis (AWE) technology is the transition from conventional cell layouts to zero-gap architectures, which enable improved electrochemical performance (1). In this design, the cell assembly typically consists of a catalyst-coated porous electrode directly interfaced with the diaphragm, followed by a metallic porous layer acting as a gas diffusion layer and the bipolar plate. Although this arrangement has been broadly implemented by electrolyzer manufacturers, systematic optimization of the individual components remains limited, particularly regarding the influence of structural parameters—such as porosity and thickness—on gas evacuation and ohmic losses (2).

In this work, the impact of component structural characteristics on the performance of zero-gap AWE cells is investigated with the objective of identifying optimal design parameters. A range of commercially available porous nickel materials, including foams and mesh structures, were assessed as electrodes and gas diffusion layers. These materials were first subjected to morphological characterization and subsequently tested in alkaline electrolysis cells under multiple configuration schemes (Figure 1). Electrochemical performance was evaluated using polarization measurements, steady-state current analysis, and electrochemical impedance spectroscopy (EIS). The experimental findings were then used as input for CFD-based simulations to determine the structural parameter combinations that maximize zero-gap cell performance.

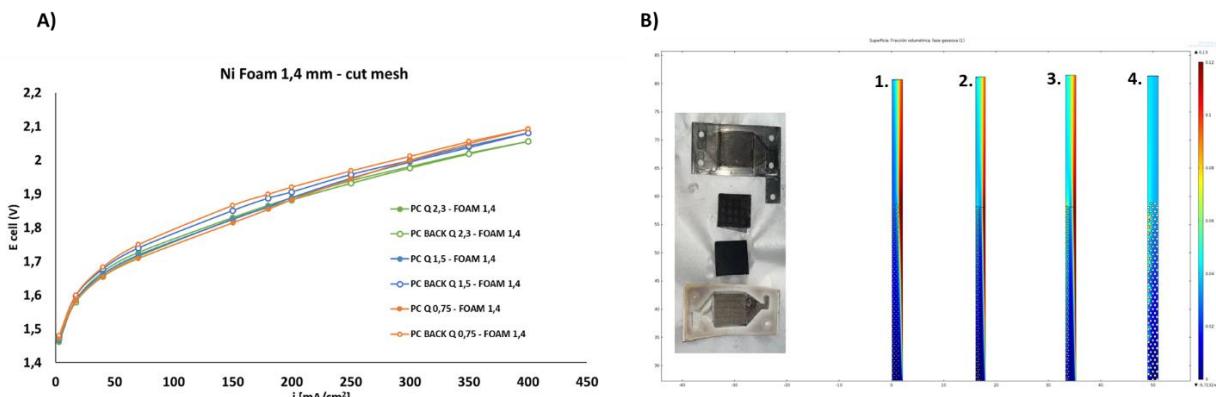


Figure 1. A) Polarization behavior at varying electrolyte flow rates for a 1.4 mm foam electrode; B) Half-cell gas volume fraction for different structural configurations: (1) 0.9 mm foam, (2) 1.4 mm foam, (3) 1.6 mm foam, (4) fully mesh-based configuration.

The optimal electrode configuration was 1.4 mm Ni foam with cut mesh, showing stable performance at high current densities. CFD indicates more homogeneous flow in the half-cell, while thicker or thinner foams negatively affect electrical contact, active area, and two-phase flow.

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## Molybdenum carbides as catalysts for the Hydrogen Evolution Reaction (HER)

Mas Marie<sup>a,\*</sup>, Dubois Guillaume<sup>a</sup>, Lagrost Corinne<sup>a</sup>, Tessier Franck<sup>a</sup>, Grasset Fabien<sup>a,b</sup>, Dumait Noée<sup>a</sup>, Cordier Stéphane<sup>a</sup>

<sup>a</sup> Univ. Rennes, CNRS, Institut des Sciences Chimiques de Rennes - UMR 6226, F-35000 Rennes, France

<sup>b</sup> CNRS-Saint-Gobain-NIMS, IRL 3629, Laboratory for Innovative Key Materials and Structures (LINK), NIMS, Tsukuba 305-0044, Japan

\*Presenting author's email: marie.mas@univ-rennes.fr

### Abstract

With the actual environmental context, developing new and greener energy sources appears as a necessity. Green H<sub>2</sub> can be produced through overall water splitting. Nevertheless, this process requires the use of catalysts, and the most commonly used so far are noble metals such as Pt, which are not only expensive but also showing low stability. Thus, replacing such catalysts with low cost, high efficiency and stable non noble metal is needed to develop promising systems, and among them, Mo-based catalysts have shown a great interest, due to their similar d-band electron structure to Pt. Their performances could even be increased by doping them with another transition metal such as Ni or Co<sup>(1,2)</sup>. In this work, Molybdenum carbides were used as catalyst for the hydrogen evolution reaction in alkaline media, occurring at the cathodic side of the electrochemical system, with the following reaction : 2 H<sub>2</sub>O<sub>(l)</sub> + 2 e<sup>-</sup> → H<sub>2(g)</sub> + OH<sup>-</sup>. Such materials were synthesized by an unusual route involving the use of molybdenum clusters as precursors<sup>(3)</sup>, which allowed to lower the synthesis temperature and obtain nanostructured catalysts, exhibiting an important specific surface area, small particle sizes and great surface porosity, all of this leading to high catalytic activity regarding the HER.

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## From CO<sub>2</sub> and Methanol to Formic Acid: A Paired Electrochemical Process on BDD/Sn

Paola Meli<sup>a,\*</sup>, Alessandro Galia<sup>a</sup>, Abdirisak Ahmed Isse<sup>b</sup>, Roberta Panepinto<sup>a</sup>, Federica Proietto<sup>a</sup>, Onofrio Scialdone<sup>a</sup>

<sup>a</sup> Dipartimento di Ingegneria, University of Palermo, Palermo, Italy

<sup>b</sup> Dipartimento di Scienze Chimiche, Università degli Studi di Padova, Padova, Italy

\*Presenting author's email: paola.meli01@you.unipa.it

### Abstract

Global decarbonization goals and the need to stabilize energy systems are accelerating research into scalable routes to convert waste carbon into chemicals and fuels. In this context, electrochemical CO<sub>2</sub> conversion is attracting strong interest because it can operate under mild conditions and be powered directly by renewable electricity<sup>1,2</sup>.

Paired electrochemical processes (PEPs) can improve the energy efficiency and economic viability of CO<sub>2</sub> conversion by valorizing both cathodic and anodic reactions<sup>3,4</sup>. Here, we investigate the paired synthesis of formic acid/formate (FA) by coupling CO<sub>2</sub> electroreduction at a tin cathode with methanol oxidation at a boron-doped diamond (BDD) anode in a divided cell.

BDD anodes offer a wide potential window, high current efficiency, excellent stability in aggressive media, and the ability to generate reactive species, making them a versatile platform for anodic electrochemical processes<sup>4,5</sup>.

Electrolyses were performed under galvanostatic conditions at 11 mA cm<sup>-2</sup> for 2 hours. A systematic study was carried out to elucidate the role of key operating parameters, including supporting electrolyte identity (cation and anion effects), electrolyte concentration, methanol concentration, and temperature. The supporting electrolyte composition was found to strongly impact both half-reactions, with distinct trends for anodic FA formation during methanol oxidation and cathodic FA formation during CO<sub>2</sub> reduction.

The results show that combining a BDD anode with a tin cathode enables very high Faradaic efficiencies for both the cathodic and anodic reactions under optimized conditions, especially when using 0.1 M Na<sub>2</sub>SO<sub>4</sub> as the catholyte supporting electrolyte and 0.5 M NaOH + 0.5 M CH<sub>3</sub>OH in the anolyte.

Acknowledgements Funded by the European Union - NextGenerationEU

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## Removal and Recovery of Carboxylic Acids from Wastewater Using Ion-Exchange Resins

Zeinabsadat Mirali Morteza-yirote\*, Raúl García-Cervilla, Manuel Andrés Rodrigo Rodrigo

*Department of Chemical and Environmental Engineering, Universidad de Castilla-La Mancha, Ciudad Real, Spain*

\*Presenting author's email: [zeinabsadat.mmiralim@uclm.es](mailto:zeinabsadat.mmiralim@uclm.es)

### Abstract

Carboxylic acids are frequently encountered in industrial effluents generated by chemical manufacturing, food processing, and pharmaceutical industries, where their persistence at low concentrations presents significant environmental and process-related challenges. Conventional treatment technologies often exhibit limited selectivity and efficiency for the simultaneous removal and recovery of these compounds. In this preliminary study, a set of commercially available ion-exchange resins was systematically evaluated and compared with respect to their removal performance and recovery capability over successive adsorption-regeneration cycles. emphasis was placed on assessing resin stability, reusability, and performance reproducibility under cyclic operating conditions. Regeneration experiments were conducted to examine the feasibility of repeated operation and the recovery of carboxylic acids within a cyclic treatment framework.



Figure 1. Schematic representation of the laboratory-scale ion-exchange column system

### Acknowledgements

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## Application of an Electrodialysis process to saline wastewaters from WWTP tertiary effluents

Vicent Ayala Andreu<sup>a</sup>, Miguel A. Montiel<sup>a,\*</sup>, Vicente García-García<sup>a</sup>, Flor Etchebarne<sup>b</sup>, Florence Lutin<sup>b</sup>, Vicente Montiel<sup>a</sup>

<sup>a</sup> Instituto de Electroquímica, Universidad de Alicante. PO 99, E-03080, Alicante, Spain

<sup>b</sup> Eurodia-ZAC Saint Martin-f84120 Pertuis, France

\*miguel.montiel@ua.es

### Abstract

Water is a scarce resource along Spain's eastern Mediterranean coast. Due to the proximity to the sea and the deficiencies in the local pipeline infrastructure, seawater intrusions occur into the wastewater system. Saline wastewater poses a major challenge for wastewater treatment plants (WWTPs), as it hinders water reuse and sustainable discharge. At Santa Pola WWTP (Latitude: 38.219109; Longitude: -0.562122) these intrusions result in conductivities between 4 to 5 mS/cm, primarily due to the presence of salts such as NaCl and Na<sub>2</sub>SO<sub>4</sub>. However, current regulations require conductivities below 3 mS/cm for water reuse in agriculture or safe discharge into natural channels. An electrodialysis (ED) system, is a membrane process of separation under the action of an electric field, where ions are selectively transported across specific membranes. Properties such as selectivity, ion-removal efficiency, and chemical-free treatment make ED process adequate for partial desalination of brackish wastewater with significant environmental benefits. For these reasons, an ED process is proposed to bring a solution to Santa Pola WWTP. The process is initially evaluated at laboratory scale. The ED reactor, provided by EURODIA Industries, has been characterized under ion concentrations and conductivities based on the real compositions of the treated wastewater from the Santa Pola WWTP (data obtained by analysis of the real waters from different years and seasons). Different stages have been done to conduct the study, initially synthetic water was used just with NaCl as a main salt, after that step, every ion that is present in the real water has been added, going from Na<sup>+</sup>, Cl<sup>-</sup> to Na<sup>+</sup>, Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, HCO<sub>3</sub><sup>-</sup>, Ca<sup>2+</sup> and Mg<sup>2+</sup>. Every ion added has been studied to observe the effects that produce over the water, from the changes of the pH and conductivity to the possible precipitations from the presence of Ca<sup>2+</sup> and Mg<sup>2+</sup>, and the possible solutions to study the real water. With synthetic water, Na<sub>2</sub>SO<sub>4</sub> has been used as electrolyte even with the presence of Ca<sup>2+</sup> and Mg<sup>2+</sup>, but in this case other electrolytes have been studied to keep the process efficient and to extend the useful life of the electrolyte due to the formation of CaSO<sub>4</sub> and MgSO<sub>4</sub> precipitate. Finally, real waters from the Santa Pola WWTP have been studied. Efficiency loss and initial conductivity and composition of the water samples have been measured since depending on the month, salinity of the water changes. Thus, this communication presents a complete study of the application of an electrodialysis process to a real problem, from the use of synthetic water to keep all parameters under control, to the use of real water where other variables are analyzed, such as the loss of system efficiency with successive experiments of the process or energy efficiency.

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## Evaluation of the electrochemical CO<sub>2</sub> reduction reaction on nanomaterials synthesized with supercritical fluids

Juan Carlos Morales-Gomero\*, Fabiola Martínez, Carlos Jiménez, Rafael Camarillo, Isaac Asencio and Jesusa Rincón

Faculty of Environmental Sciences and Biochemistry, Department of Chemical Engineering, Universidad de Castilla-La Mancha, Toledo, Spain

\*Presenting author's email: juanc.morales@uclm.es

### Abstract

The continuous increase in CO<sub>2</sub> emissions from fossil fuel-based industries, transportation, cement production, and hydrogen production from methane has intensified the greenhouse effect, motivating the development of effective CO<sub>2</sub> conversion strategies. Among these, the electrochemical reduction of CO<sub>2</sub> is a promising approach, although significant challenges remain in improving catalyst efficiency and product selectivity.

This work investigates the electrochemical reduction of CO<sub>2</sub>, building on previous studies involving catalysts synthesized under supercritical conditions and consisting of Cu- or Ni-based metallic nanoparticles supported on carbon nanotubes. These catalytic systems have demonstrated promising activity at low to moderate current densities. Current efforts are focused on strategies to enhance the achievable current density in the electrochemical cell, including electrochemical evaluation and physicochemical characterization. Furthermore, cell design optimization (Figure 1) and the replacement of carbon nanotubes with more sustainable carbon-based supports, such as biocarbons, are being explored to improve both electrochemical performance and process sustainability.

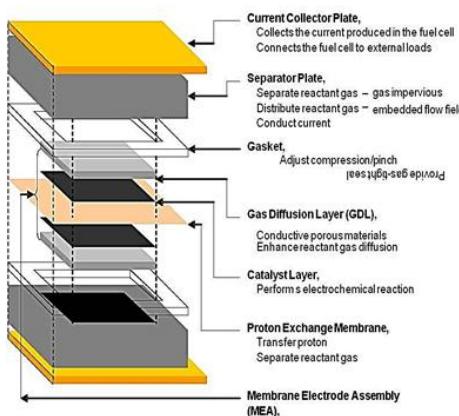


Figure 1. Configuration of Gas diffusion electrode (GDE)-based MEA for CO<sub>2</sub> reduction

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## From polluted soil to clean effluents: Surfactant-Assisted PAH emulsions and electrochemical treatment

Yaiza Moreno-De La Fuente<sup>a,\*</sup>, Pablo García<sup>a</sup>, Salvador Cotillas<sup>a</sup>, Aurora Santos<sup>a</sup>, Carmen M. Domínguez<sup>a</sup>.

<sup>a</sup> Department of Chemical and Materials Engineering, Faculty of Chemical Sciences, Complutense University of Madrid, Avenida Complutense S/N, 28040 Madrid, Spain \* [yaimor01@ucm.es](mailto:yaimor01@ucm.es)

In recent years, increasing levels of organic contaminants in soil and groundwater have attracted considerable scientific attention due to the environmental and public health risks they present. Among these pollutants, polycyclic aromatic hydrocarbons (PAHs) are of particular concern because of their high toxicity and carcinogenic properties. PAHs commonly contaminate soil and groundwater as a result of industrial waste discharges or the long-term degradation of fuel spills (1). These discharges percolate through the subsoil, eventually reaching groundwater sources. The remediation of PAHs from surface soils and subsurface environments is especially challenging due to their chemical stability and limited water solubility, which categorize them as Hydrophobic Organic Compounds (HOCs). These substances tend to strongly adsorb onto soil particles or associate with other hydrocarbons to form Non-Aqueous Phase Liquids (NAPLs). The use of surfactants can improve PAH solubility, thereby enhancing their removal through Soil Washing processes (2). However, these approaches generate contaminated aqueous emulsions that require subsequent on-site treatment.

In this context, the development of technologies capable of achieving complete degradation of organic contaminants in such polluted emulsions is essential to mitigate environmental risks. Over the past few decades, Electrochemical Advanced Oxidation Processes (EAOPs) have been extensively investigated for the degradation of a wide range of organic compounds in aqueous media. EAOPs rely on the *in situ* generation of highly reactive radical species, mainly hydroxyl radicals, through water oxidation and the oxidation of ions naturally present in the effluents, enabling the efficient degradation of organic pollutants without the addition of chemical reagents. Among these technologies, electrochemical oxidation (EO) has demonstrated high efficiency in the treatment of organic pollutants, including those present in contaminated aqueous emulsions.

Against this background, this study investigates the combined and optimized use of soil washing and subsequent electrochemical oxidation for the remediation of PAH-contaminated emulsions. Representative polycyclic aromatic hydrocarbons (phenanthrene, anthracene, pyrene, and benzo(a)pyrene) were targeted, using Emule 3® as a biodegradable non-ionic surfactant. The nature and concentration of the reactive species generated are primarily determined by the electrode material and the applied current density. In this study, a Boron-Doped Diamond (BDD) electrode was used as the anode. The influence of current density on pollutant degradation was investigated over a range of 30 to 125 mA cm<sup>-2</sup> and the effectiveness of the proposed process for the removal of 200 mg L<sup>-1</sup> of PAHs was assessed. High-performance liquid chromatography (HPLC) was employed to monitor the reduction in PAH concentrations over time. Additional aspects related to the treated effluent were also examined, including ion chromatography analyses, total organic carbon (TOC) measurements, identification of the oxidizing species and radicals involved in the degradation process, and evaluation of the evolution of the surfactant capacity of the remaining surfactant. The results obtained show that 98% of the PAHs were degraded within 14 hours under moderate conditions.

### Acknowledgements

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## Nature's first Fuel Cell: hydrothermal vents, the origin of life and CO<sub>2</sub> reduction

Francesco Panico <sup>a,\*</sup>, Hye-Eun Lee <sup>b,c</sup>, Alessandro Minguzzi<sup>a,d,e</sup>, Ryuhei Nakamura <sup>b,c</sup>,  
Michael Russell <sup>f</sup>, Alberto Vertova <sup>a,d</sup>

<sup>a</sup> Department of Chemistry, Università degli studi di Milano, via Golgi 19, 20133, Milan, Italy

<sup>b</sup> Earth-Life Science Institute, Tokyo Institute of Technology, Tokyo, Japan

<sup>c</sup> Center for Sustainable Resource Science, RIKEN, Wako, Saitama, Japan

<sup>d</sup> Consorzio Interuniversitario di Scienze e Tecnologia dei Materiali, Via San Giusti 9, 50121, Florence, Italy

<sup>e</sup> Department of energy, Politecnico di Milano, Via Lambruschini 4a, 20156 Milano, Italy

<sup>f</sup> Department of chemistry, Università di Torino, 10125 Torino, Italy

\*Presenting author's email: [francesco.panico@unimi.it](mailto:francesco.panico@unimi.it)

Oceanic Hydrothermal Vent are geological formations proposed as possible cradles for the emergence of life on our planet. In the Archean era, at the Alkaline Hydrothermal Vent (AHTV) environment, a mineral barrier, composed of iron oxide and hydroxide, iron sulphide, along with Ni, Zn, Co, and Mn ions precipitate creating the vent structure itself. This mineral barrier separates two different environments: the outer acidic with CO<sub>2</sub> and the inner alkaline with H<sub>2</sub>, in this situation an electrochemical potential difference is generated across the mineral barrier of the vent structure; this life-like thermodynamic disequilibrium can be dissipated through coupling of CO<sub>2</sub> reduction (CO<sub>2</sub>R) reaction with hydrogen oxidation (HOR), generating the very first organic molecules on our planet. (1)(2)

This fascinating lesson from nature - demonstrating how efficient, low-energy processes, based on abundant and non-toxic materials, can initiate complex chemical syntheses - provides a powerful paradigm for contemporary green chemistry and technological sustainability. Our research focuses on developing "emergence of life inspired materials" for CO<sub>2</sub> conversion, addressing the environmental challenges of the new millennium.

Recently, we proposed a new framework to describe hydrothermal vent behaviour using concepts from electrochemistry: treating the system as a short-circuited fuel cell, where CO<sub>2</sub>RR and HOR occur on distinct "mineral electrodes" that are short-circuited, the process is driven ultimately by the pH gradient between simulated vent fluids and ocean water. Using this model, we detected spontaneous current generation and measure the operating electrochemical potential (3).

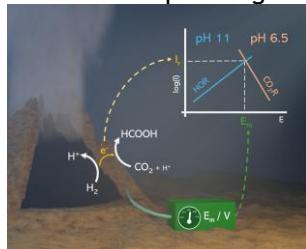


Figure 1. Measuring reaction current and mixed potential of CO<sub>2</sub> Reduction and H<sub>2</sub> oxidation

Furthermore, we demonstrated that mineral phases such as mackinawite (Fe-S) and violarite (Fe-Ni-S) can catalyse electrochemical CO<sub>2</sub> reduction, producing formic acid, methanol and carbon monoxide at -0.72 V vs RHE, and suggesting that a very low overpotential is needed to reduce CO<sub>2</sub> on these materials. (4)

Finally, microfluidic platforms enabled observation of mineral barrier formation and growth, and their integration with microelectrochemistry allowed direct measurement of the potential difference between the two environments as well as the potential of the mineral barrier itself.

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## Forecasting the performance of Nafion membranes using a novel empirical model approach

Irene Peñas Fuertes<sup>a,\*</sup>, Francesca Mennilli<sup>b</sup>, Juan Carlos Carmona Pulido<sup>a</sup>, Julia Isidro Elvira<sup>a</sup>, Mosè Rossi<sup>b</sup>, Flavio Caresana<sup>b</sup>, Gabriele Comodi<sup>b</sup>, and Jesús Rodríguez Ruiz<sup>a</sup>

<sup>a</sup> Scientific Development Unit, National Hydrogen Center, Prolongación Fernando el Santo s/n, Puertollano, Spain

<sup>b</sup> Department of industrial Engineering and Mathematical Sciences, Polytechnic University of Marche, Ancona, Italy  
\*irene.penas@cnh2.es

### Abstract

Currently, electrochemical cells (batteries, fuel cells, electrolyzers...) powered by surplus renewable energy are considered a key technology in the energy transition process. Among these, Proton Exchange Membrane (PEM) electrolyzers stand out due to their high current density, energy efficiency, and the high purity of the hydrogen produced. However, the substantial cost of their components remains one of the primary factors limiting their profitability and industrial scaling (1).

Nafion membranes (DuPont) are a critical element in system performance owing to their ability to selectively transport hydrogen protons ( $H^+$ ) from the anode to the cathode to facilitate the electrochemical reaction and, consequently, hydrogen production. Although the proton conductivity of these membranes has been extensively studied, systematic characterization of through-plane conductivity under continuous operating conditions remains scarce (2).

This work presents an experimental study of the through-plane proton conductivity of Nafion NR212, N115, and N117 membranes in a non-activated state. Measurements were conducted under controlled conditions of relative humidity (20-95%) and temperature (30-80 °C). Based on the experimental data obtained, a new empirical model has been developed to predict the behavior of Nafion membranes as a function of operating conditions. The results demonstrate strong predictive capability across the entire analyzed operating range, with Root Mean Square Error (RMSE) values ranging from 1.1  $mS \cdot cm^{-1}$  at 50 °C to 4.7  $mS \cdot cm^{-1}$  at 80 °C (2).

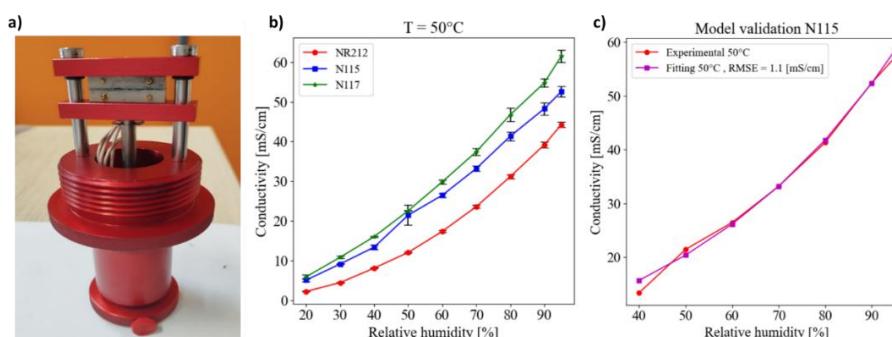


Figure 1. a) Cell head assembly for through-plane membranes conductivity measurements; b) Experimental conductivity of several non-activated Nafion membranes at 50°C; c) Model validation for N115 at 50°C.

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## Photoelectrocatalytic hydrogen generation and CO<sub>2</sub> reduction over optimized anodized WO<sub>3</sub> nanostructures

Alberto Pérez-Calvo\*, Gemma Roselló-Márquez, Ramón Fernández-Domene, Benjamín Solsona and Rita Sánchez-Tovar.

Dept. Chemical Engineering Universitat de València, Av. De la Universidad s/n, Burjassot, Spain.

\*alberto.perez-calvo@uv.es

### Abstract

The development of efficient, stable, and environmentally friendly photoanodes is essential for solar-powered hydrogen generation and CO<sub>2</sub> utilization for sustainable energy conversion. In this work, we present a nanostructured tungsten trioxide (WO<sub>3</sub>) photoanode synthesized by anodization of tungsten in the ionic liquid [EMIM][BF<sub>4</sub>] under hydrodynamic conditions. The synthesis strategy allows precise morphological control, yielding highly ordered nanoplates with large surface area and improved crystallinity. Optimal performance was achieved at 400 rpm, yielding well-defined nanoplate structures with high surface area and enhanced light absorption. Electrochemical impedance spectroscopy and Mott-Schottky analysis revealed increased donor density and reduced charge transfer resistance at this condition, favoring efficient electron-hole separation and leading to a better photoelectrocatalytic performance. Under solar illumination, the optimized WO<sub>3</sub> photoanode achieved a photocurrent density of 0.58 mA·cm<sup>-2</sup> at 1.0 V<sub>Ag/AgCl</sub>, doubling the activity of static samples (Figure 1A) and obtaining higher hydrogen production. On the other hand, the photoelectrochemical (PEC) reduction of CO<sub>2</sub> in a continuous H-type cell revealed the formation of formic acid, methanol and acetic acid (Figure 1B). The mechanistic study indicates that photogenerated e<sup>+</sup>/h<sup>+</sup> drive the transformation of adsorbed CO<sub>2</sub> through CO<sub>2</sub><sup>-</sup> and HCOO<sup>-</sup> intermediates to formic acid, which may further undergo proton-coupled electron transfer to yield methanol and acetic acid [1].

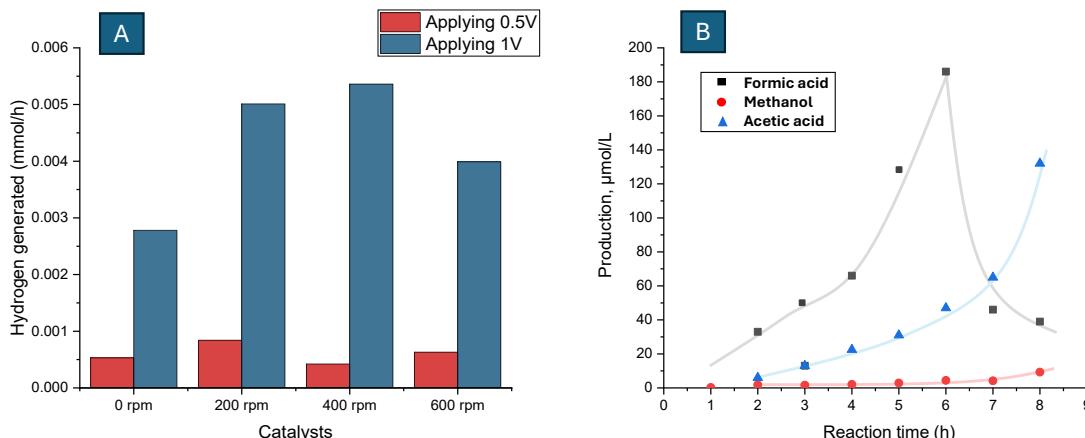


Figure 1. A) Generated hydrogen by PEC water splitting using the different WO<sub>3</sub> nanostructures and, B) Evolution of the products during PEC CO<sub>2</sub> for the 400 rpm sample.

Overall, the results show that the role of the photoanode is crucial, as OER-derived protons and charge carriers (e<sup>-</sup>/h<sup>+</sup>) influence on the selectivity and rate of the CO<sub>2</sub> conversion in the cathode.

**Acknowledgements:** We thank to the MICIU (FPU23/02557) and Generalitat Valencia for the CIAICO/2024/87.

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## Hydrochar dopped carbonaceous anodes for metal recovery in microbial fuel cells

María Teresa Pinés\*, Ester López, Martín Muñoz, Javier Llanos, Francisco Jesús Fernández-Morales

<sup>a</sup> Chemical Engineering Department, University of Castilla-La Mancha, Avenida Camilo José Cela, s/n, Ciudad Real, Spain

\*Presenting author's email: [mteresa.pines@uclm.es](mailto:mteresa.pines@uclm.es)

### Abstract

This research presents an innovative approach to metal recovery from acid mine drainage (AMD) through the use of carbon felt anodes enhanced with hydrothermal carbon (HTC) materials.

AMD, a hazardous byproduct of mining operations, contains high concentrations of dissolved metals and acidic compounds, posing serious threats to ecosystems and human health. Addressing this challenge is critical for advancing sustainable mining practices. Conventional remediation techniques—such as chemical precipitation, ion exchange, reverse osmosis, phytoremediation, and electrochemical treatments—are widely used but often suffer from limitations including high energy consumption, incomplete metal extraction, and the generation of secondary waste such as sludge. In recent years, Bioelectrochemical Systems (BES), particularly Microbial Fuel Cells (MFCs), have emerged as promising technologies for AMD treatment. These systems offer a dual benefit: they not only facilitate the removal and recovery of valuable metals but also generate electricity by converting the chemical energy of pollutants into electrical energy. This energy-positive aspect adds a compelling economic incentive to their environmental utility.

Within this framework, the present study explores the performance of MFCs equipped with carbon felt (CF) anodes modified with hydrochar—both in its raw form and thermally activated. The modified electrodes were evaluated for their ability to recover copper from AMD under controlled conditions. Remarkably, complete copper recovery was achieved across all electrode types. The best recovery rate was observed with CF anodes doped with activated hydrochar, recovering about 16 mg Cu/(L·h), followed by non-activated hydrochar at 11 mg Cu /(L·h), and raw CF at 7 mg Cu /(L·h).

Electrochemical analysis further confirmed the superior performance of hydrochar-doped electrodes, which achieved a peak current density about 25% higher than of the unmodified CF anodes. This enhancement is attributed to improved conductivity, surface area, and microbial interaction facilitated by the HTC materials. Overall, the findings underscore the potential of HTC-doped carbonaceous electrodes to revolutionize BES applications for metal recovery, offering a sustainable, efficient, and economically viable solution for the treatment of mining wastewater.

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## Effect of Iron Precursor Loading on the ORR Activity of ZIF-8-Derived Fe-N-C Catalysts in Acidic Media for PEM Fuel Cell Applications

Ettore Rosella<sup>a\*</sup>, Carmelo Lo Vecchio<sup>a</sup>, Gioacchino Davide Bucca<sup>a</sup>, Vincenzo Baglio<sup>a</sup>, Irene Gatto<sup>a</sup>

<sup>a</sup> CNR-ITAE, Salita Santa Lucia Sopra Contesse, 5, 98126 Messina ME, Italy.  
email: [ettorerosella@cnr.it](mailto:ettorerosella@cnr.it)

### Abstract

The development of active, durable, and low-cost non-precious metal catalysts for the oxygen reduction reaction (ORR) in acidic media remains a major challenge for fuel cell technologies. Fe-N-C catalysts derived from metal-organic frameworks (MOFs), particularly ZIF-8, have attracted significant interest due to their high surface area with uniform metal dispersion and nitrogen-rich structure with nanometric porosity, which make them ideal precursors for fundamental ORR studies in acidic environments.

In this work, a series of Fe-N-C catalysts were synthesized by impregnating ZIF-8 with different loadings of an iron precursor, followed by high-temperature pyrolysis. Ketjenblack carbon was added to improve electrical conductivity and promote a homogeneous dispersion of active sites. The influence of iron precursor loading on the electrochemical behavior and ORR performance was evaluated in acidic medium (0.1 M HClO<sub>4</sub>) using rotating disk electrode (RDE) measurements, including cyclic voltammetry (CV) and linear sweep voltammetry (LSV), the data of which are reported in the poster graphs.

The most promising catalysts were also tested in a fuel cell configuration, confirming their applicability under operating conditions. Preliminary results indicate that iron precursor loading significantly affects the electrochemical response and ORR performance of ZIF-8-derived Fe-N-C catalysts in acidic conditions. These findings contribute to a better understanding of structure-activity relationships and provide useful guidance for the rational optimization of Fe-N-C catalysts for fuel cell applications.

### Acknowledgements

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## Simulation of cathode dynamics of a PEM fuel cell using OpenFCST

Fermín Sáez-Pardo<sup>a,\*</sup>, Juan José Giner-Sanz<sup>a</sup>, Marc Secanell<sup>b</sup>, Aslan Kosakian<sup>b</sup>, Valentín Pérez-Herranz<sup>a</sup>

<sup>a</sup> IEC group, Depto. Ingeniería Química y Nuclear, Universitat Politècnica de València, Camí de Vera s/n, Valencia 46022, Spain

<sup>b</sup> Energy Systems Design Laboratory, Department of Mechanical Engineering, University of Alberta, 9211-116 Street NW, Edmonton T6G 1H9, Canada

\*Presenting author's email: fersaepa@etsii.upv.es

### Abstract

Polymer Electrolyte Membrane Fuel Cells (PEMFCs) are electrochemical devices that convert the chemical energy of the fuel, typically hydrogen, directly into electrical power. PEMFCs offer several advantages, including low operation temperatures (<100°C), absence of moving parts, quiet operation, and modularity, among others (1). These characteristics have promoted PEMFCs as a promising alternative for power generation devices for all sorts of vehicles and other applications (2). Nevertheless, PEMFCs face challenges such as high costs and limited durability (3).

To address these limitations, extensive research has been conducted to optimize cell performance and mitigate degradation in PEMFCs. In this regard, modelling has played a key role. Even though a significant number of steady-state models have been reported in literature, transient models remain scarce, limiting the analysis of impedance spectra and other transient processes taking place in PEMFCs (e.g. charge transfer, mass transfer, among others).

In this work, cathode dynamics of a PEMFC fuel cell were simulated using OpenFCST, an open-source software that provides a framework to develop new models of PEMFCs (4). The selected model for the simulations was developed by Kosakian et al. (5) and was used to study the effect of oxygen transport at macro- and pore-level scale, catalyst layer (CL) active area, and proton conductivity in the ionomer of the CL, on the impedance spectra of a PEMFC. The results were used to identify key changes in impedance and distribution of relaxation time spectra.

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## Nanocomposite Electrodes with CuFe Layered Double Hydroxides and Hydrochar for Alkaline HER and HOR: A Multifunctional Platform for Hydrogen Electrocatalysis

Michelle Saliba<sup>a,b</sup>, Philippe Knauth<sup>a</sup>, Emily Bloch<sup>a</sup>, Luca Pasquini<sup>a</sup>, Emanuela Sgreccia<sup>b</sup>, Riccardo Narducci<sup>b</sup>, Alessandra Varone<sup>b</sup>, and Maria Luisa Di Vona<sup>b</sup>

<sup>a</sup>Aix Marseille Univ, CNRS, MADIREL (UMR 7246) and International Laboratory: Ionomer Materials for Energy, Campus St Jérôme, 13013 Marseille, France

<sup>b</sup>University of Rome Tor Vergata, Dep. Industrial Engineering and International Laboratory: Ionomer Materials for Energy, 00133 Roma, Italy

e-mail: [michelle.saliba@univ-amu.fr](mailto:michelle.saliba@univ-amu.fr)

The transition toward carbon-neutral energy systems requires efficient, affordable electrocatalysts to drive both the hydrogen evolution (HER) and oxidation (HOR) reactions. Copper-iron layered double hydroxides (CuFe-LDHs) are promising due to their abundance, tunable redox properties, and Cu-Fe synergy, but their activity remains limited by poor conductivity and surface accessibility [1,2].

To address these challenges, we developed CuFe-LDH electrodes with functional additives and/or hydrochar (HC). HC, a porous,  $sp^2$ -rich carbon derived from pine needles, was introduced to systematically assess its effect on electrode performance, while additional additives were used to tune structural order and ionic mobility.

Electrochemical tests in 1 M KOH (LSV, PEIS, CV, CP) revealed composition-dependent activity. The best HER and HOR performances were achieved when highly crystalline CuFe-LDH was combined with a hydroxide-conducting ionomer binder. Nevertheless, HC-based electrodes displayed good durability and efficiency in HER and the lowest onset potential during HOR.

These findings confirm that compositional tuning and sustainable hydrochar integration improve CuFe-LDH electrodes for hydrogen technologies, though further optimization is needed to overcome kinetic and mass-transport limitations.

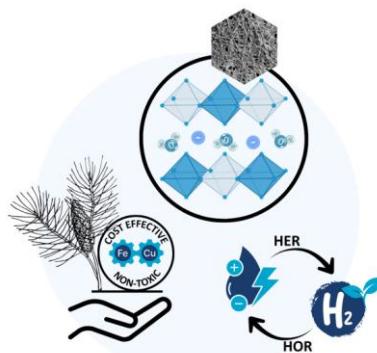


Figure 1. CuFe LDH-Hydrochar for Hydrogen Electrocatalysis

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## Relationship between electrochemical properties and catalytic results of M1-based catalysts used for ethylene production by ethane ODH.

Ginebra Sánchez-García<sup>a,\*</sup>, Agustín de Arriba<sup>b</sup>, Rita Sánchez-Tovar<sup>a</sup>, Ramón M. Fernández-Domene<sup>a</sup>, José Manuel López-Nieto<sup>b</sup>, Benjamín Solsona<sup>a</sup>.

<sup>a</sup> Departament d' Enginyeria Química, Universitat de València, Av. de la Universitat, s/n, 46100 Burjassot-Valencia, Spain.

<sup>b</sup> Instituto de Tecnología Química, UPV-CSIC, Avenida de los Naranjos s/n, 46022 Valencia, Spain.

\*ginebra.sanchez@uv.es

### Abstract

Ethylene, a key petrochemical feedstock, is mainly produced through steam cracking of naphtha or light alkanes—a highly energy-intensive process due to its endothermic nature and high operating temperatures. As an alternative, the oxidative dehydrogenation (ODH) of light alkanes offers higher conversions at lower temperatures through an exothermic catalytic reaction, though achieving high selectivity remains a major challenge due to competing side reactions.

Up to date, the most promising catalytic systems for the ethane ODH are multicomponent mixed metal oxides, i.e. Mo-V-Te-Nb-O catalysts presenting the M1 phase, which are known to follow a redox Mars-Van Krevelen mechanism during the reaction. These catalysts also exhibit semiconducting behavior, allowing their surface oxidation and reduction processes to be studied through electrochemical analysis. Few studies have explored the relationship between the electrochemical properties and catalytic performance of such materials, and none of these studies have employed Electrochemical Impedance Spectroscopy (EIS)<sup>1,2</sup>. Previous research has linked the semiconductor behavior of these catalysts to the semiconductor type of their constituent simple oxides, but this assumption has not been experimentally validated using capacitance measurements.

In this study, MoVO, MoVTeO, and MoVTeNbO mixed oxides were synthesized hydrothermally and subsequently heat-treated under N<sub>2</sub> at 400- 600 °C. The extensive electrochemical characterization revealed that the highest ethylene selectivity in ethane ODH was observed in materials exhibiting higher charge-transfer resistance at the active layers and n-type semiconductivity with fewer oxygen vacancies. Likewise, lower current densities in cyclic voltammetry measurements—indicating lower electrochemical activity—were correlated with enhanced selectivity in the ODH reaction.

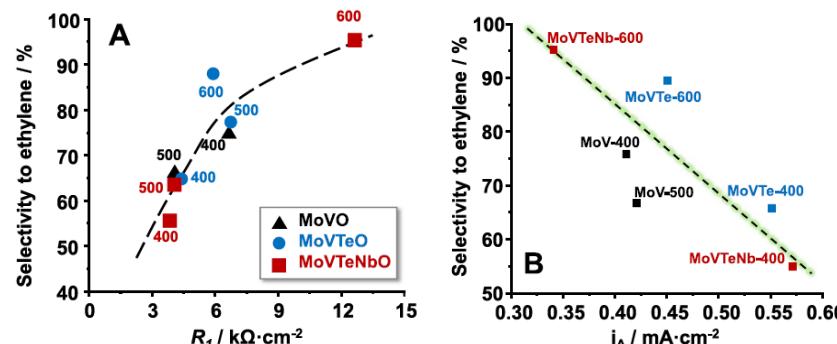


Figure 1. Relationship between the selectivity to ethylene and the  $R_1$  data from EIS data fitting (A) or the anodic current density,  $i_A$ , (B) for MoV-, MoVTe- and MoVTeNb-series calcined at 400, 500 and 600 °C.

### Acknowledgements

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## Optimized anode structures for high-temperature SOFCs with improved resistance to carbon deposition

M. Rosario Serrano de la Cruz<sup>1,\*</sup>, Ángela Moratalla<sup>1</sup>, Noelia Díaz<sup>1</sup>, Irene Ayuso<sup>1</sup>, Adrián Pardo<sup>1</sup>, Lidia Sánchez-Beato<sup>2</sup>, Cristina Montes<sup>1</sup>, Roberto Campana<sup>1</sup>

<sup>1</sup> Hydrogen National Centre (CNH2), Prolongación Fernando el Santo, Puertollano, Spain

<sup>2</sup> IDavinci, Las Rozas de Madrid, Spain

\*rosario.serrano@cnh2.es

Solid oxide fuel cells (SOFCs) are a highly efficient and versatile technology for clean energy conversion, operating at elevated temperatures (700-900 °C) that allow direct utilization of hydrogen, methane, biogas, and other hydrocarbon fuels. Conventional Ni-YSZ cermet anodes are widely employed due to their high electrocatalytic activity for H<sub>2</sub> and CO oxidation, excellent electronic conductivity, and good thermomechanical compatibility with the YSZ electrolyte. However, under hydrocarbon operation, these anodes are highly susceptible to carbon deposition, which can originate from hydrocarbon decomposition or CO reduction. Carbon accumulation on the Ni surface obstructs triple-phase boundaries (TPBs) and pores, hindering electrochemical reactions and progressively degrading performance. Additional factors such as Ni particle agglomeration at high temperatures and structural deterioration caused by redox cycling further compromise the long-term stability and durability of Ni-YSZ anodes, limiting their operational lifetime when using carbon-based fuels.

The aim of this study is development and optimization of a novel anode (Ni (Ce,Zr)O<sub>2</sub>) for solid oxide fuel cells, produced via electrospinning, to promote the controlled exsolution of nickel nanoparticles and enhance electrocatalytic activity and carbon deposition resistance. Electrospinning is an effective technique for producing ultrafine nanofibers with high porosity and surface-to-volume ratio, facilitating ion transport and increasing active-site density. Once the nanofibers are obtained, they are treated under reducing atmospheres to induce Ni nanoparticle exsolution and anchoring. Ni exsolution from the lattice results in uniformly distributed, stable nanoparticles on the perovskite surface, forming a nanoporous structure with high surface area and enhanced catalytic performance. Subsequent to nanofiber fabrication and optimization, they are applied as the anode functional layer onto a YSZ support. A GDC and LSM-YSZ interlayer is subsequently deposited to enhance chemical and mechanical compatibility, while LSM is employed as the cathode. All functional layers are deposited using ultrasonic spray coating, a technique that enables uniform and well-adhered films with controlled thickness.

The electrochemical performance of the SOFC anode was evaluated at 850-900 °C using polarization curves, EIS and durability tests with hydrogen, biogas and methane reformat. Nickel exsolution significantly improved anode efficiency, durability and resistance to carbon deposition. The fuel showing the best performance was selected for long-term tests at 100 mA·cm<sup>-2</sup>, demonstrating the potential of these nanofiber-based anodes for operation with complex fuels.

### Acknowledgements

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## ***In-situ* formation of nickel oxyhydroxides and electrochemical behavior of Ni200 cathodes under renewable-integrated alkaline electrolysis**

**Rebeca.M Simion** <sup>a, \*</sup>, **Nerea López-García** <sup>a</sup>, **Roberto Campana** <sup>a</sup>, **Jesús Rodríguez** <sup>a</sup>

<sup>a</sup> National Hydrogen Center, Prolongación Fernando el Santo S/N, Puertollano, Spain

\* rm.simion@cnh2.es

Alkaline water electrolysis is a well-established green hydrogen technology, but its integration with renewable energy faces challenges due to slow kinetics and dynamic operation. This work investigates *in-situ* formation of catalytic nickel oxyhydroxides on Ni200 electrodes, which reduce HER overpotential and enhance stability without additional catalysts (1). The electrochemical behavior and surface evolution of smooth Ni200 electrodes were studied under prolonged and intermittent cathodic polarization in 30 wt% KOH at 70 °C. Prolonged polarization was performed in a three-electrode cell at  $-150 \text{ mA cm}^{-2}$  for up to 100 h to evaluate long-term stability, while intermittent polarization under current profiles simulating a photovoltaic supply was tested in a two-electrode cell using JRC-defined Belgian grid data. Characterization was performed using CV and CP, complemented by XPS and XRD analyses to correlate electrochemical performance with surface phase evolution.

Results indicate progressive activation of Ni200, with a 20 mV decrease in HER overpotential after 100 h (Figure 1b), associated with the *in-situ* formation of  $\alpha\text{-Ni(OH)}_2$  and intermediate  $\text{NiO}_x\text{H}_y$  phases (2,3)(Figure 1a), whose presence becomes significant after 50 h of polarization. Furthermore, under intermittent polarization (Figure 1c), the electrodes exhibited no degradation and further improved electrocatalytic activity, with overpotential reductions of up to 50 mV (Figure 1d). Overall, these results demonstrate the suitability of Ni200 as a cathode for alkaline electrolysis under dynamic conditions.

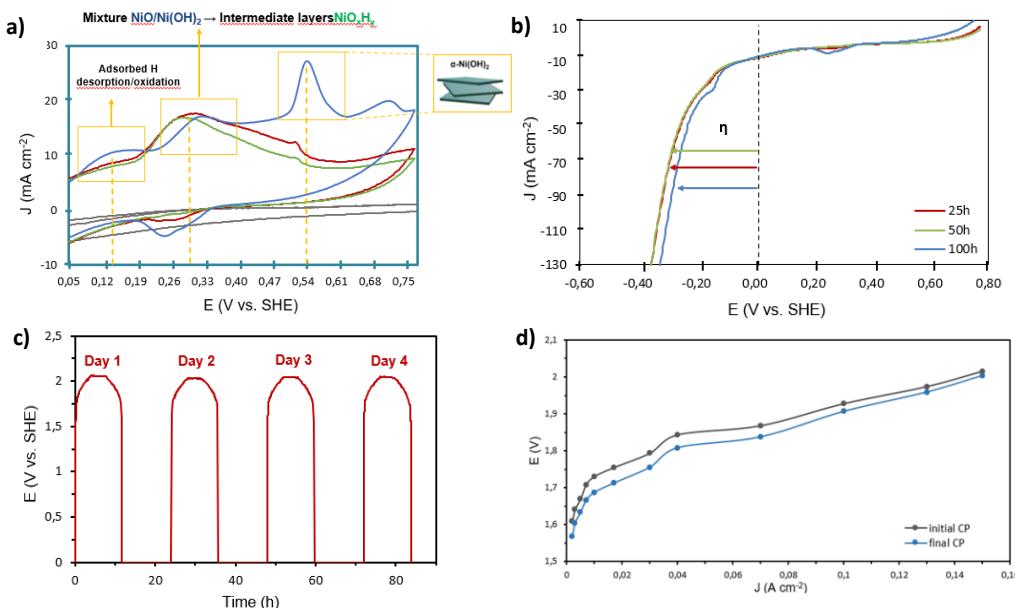


Figure 1. a) Anodic region of Ni200 CVs at 70 °C after CP ( $-150 \text{ mA cm}^{-2}$ ). b) CV comparison of Ni200 in the cathodic region at 70 °C after varying CP durations ( $-150 \text{ mA cm}^{-2}$ ). c) Simulated photovoltaic profile over four days. d) Initial vs. final polarization curve.

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## Electrochemical Strategy for the Sustainable Production of ICMs

Andrea Solida<sup>a\*</sup>, Letizia Sorti<sup>a</sup>, Carlo Morelli<sup>a</sup>, Alice Gargiulo<sup>b</sup>, Michele Sponchiado<sup>b</sup>, Luciano Lattuada<sup>c</sup>, Fulvio Uggeri<sup>c</sup>, Alberto Vertova<sup>a</sup>, Alessandro Minguzzi<sup>a</sup>

<sup>a</sup>Università degli Studi di Milano, Dipartimento di Chimica, via Golgi 19, Milano, Italy

<sup>b</sup>Industrie DeNora S.p.A., Via Leonardo Bistolfi, 35, Milano, Italy

<sup>c</sup>Bracco Imaging S.p.A., via Folli 50, Milano, Italy

\*Presenting author's email: andrea.solida@unimi.it

### Abstract

Iodinated contrast media (ICM) are among the most used medical imaging agents to enhance the visibility of blood vessels and organs on medical X-ray-based imaging techniques.<sup>[1]</sup> A notable commonality across all ICM is the iodination reaction, which remains a subject of ongoing improvement in their synthesis. Iodination reactions are currently carried out using toxic and hazardous reactants such as ICl, which add steps and costs to the final product. We have focused on the development of an easily scalable and eco-friendly method for the electrochemical iodination of some important ICM intermediates. The electrochemical iodination reaction has been previously investigated by other research groups, but there are still several problems to be solved, for example the generation of the iodinating agent and the iodination itself are usually carried out separately and in organic solvents.<sup>[2]</sup> It would be highly desirable to find synthetic methods that are greener and more capable of undergoing an industrial scale-up than those investigated so far.<sup>[3]</sup> We have demonstrated that the electrochemical in situ generation of highly iodinating species (e.g. IO<sup>-</sup>) can lead to the iodination of aromatic molecules and almost no by-products. This methodology increases the atom economy of the reaction respect to a classical iodination, as both the atoms of the molecular iodine are exploited to iodinate the molecule (see Figure 1). The oxidation potentials of iodine, iodide and different possible ICMs precursors were investigated through the screening of various electrode materials to identify the most suitable conditions for selective electrochemical iodination. The experimental conditions have been optimized and the potentiostatic electrolysis in a recirculating divided cell with cation exchange membrane has been successfully scaled for the iodination of 3 L of 0.7 M reagent solution. The process exhibits current density of 100 mA·cm<sup>-2</sup>, near unity faradaic efficiencies and yields exceeding 90%. Furthermore, water is employed as the reaction medium, exploiting the polar organic reactants acting also as co-solvents to dissolve iodine. This choice makes the process greener and more attractive for industrial scale-up due to its improved safety and sustainability. In conclusion this process can provide a concrete, as well as green, alternative to the current use of highly toxic, corrosive and expensive iodinating agents in the synthesis of ICMs and other important pharmaceuticals.

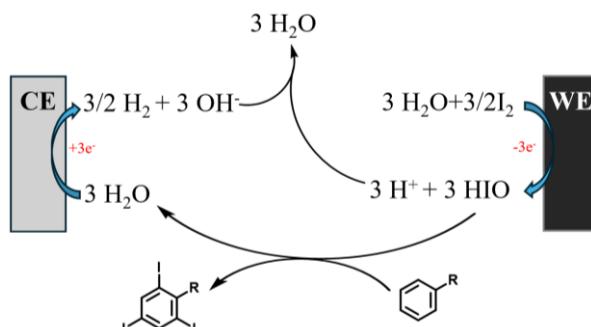


Figure 1: Electrochemical iodination process

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## New water treatment for hydrogen production

Francisco Manuel Soria López<sup>a</sup>, Manuel Andrés Rodrigo Rodrigo<sup>a</sup>, Jesús Rodríguez Ruiz<sup>b</sup>, Carmen María Fernández Marchante<sup>a</sup>

<sup>a</sup> Universidad de Castilla la Mancha, Avenida Camilo José Cela, s/n.- 13071 Ciudad Real

<sup>b</sup> Centro Nacional del Hidrógeno, Prolongación Fernando el Santo, s/n 13500 Puertollano (Ciudad Real)

[\\*Francisco.Soria@uclm.es](mailto:Francisco.Soria@uclm.es)

### Abstract

In the last two decades, the risk and consequences of climate change have been more prominent than ever. To remediate this situation, promising technologies are rising; some of these are related to green hydrogen, an energy vector. It is produced by electrolysis using only water and electricity making it a renewable and ecofriendly process. However, the water necessary for the electrolysis process must have a certain quality (type I) for its continuous operation. A treatment process involving demineralization is required to obtain this water, resulting in pure water for the electrolyzer and a solution concentrated in salt, which is generally returned to the source (lake, river, sea, underground water...). This water concentrated in salt can become a problem in the future when hydrogen industries are operational, resulting in the salinization of lakes, rivers, aquifers and small seas. That is why the aim of this work is to create a process that produces pure water without salinizing our water resources, because we believe that for hydrogen to be truly green, it has to be zero environmental impact on every aspect.

In order to achieve this a process with different stages is proposed. In the first part of the process the well water is softened adding a basic solution, after which a white solid salt is obtained. In the second part of this process the water is introduced in a electrodialysis unit where we obtained a pure water current and a brine. The last part of the process is divided in two, the brine will be introduced into a EDEN electrolyzer<sup>1,2</sup> while the pure water will be introduced in the commercial electrolyzer PEM and Alkaline to produce Hydrogen. Using the EDEN electrolyzer Hydrogen, Chlorine and different subproduct like NaOH and an acid current will be obtained using the Brine. As it has been described during this process no residue is produced during the water treatment.

### Acknowledgements:

This work has been developed as part of Energy and Renewable Hydrogen Program of the Complementary R&D&I Plans of the Ministry of Science and Innovation, funded by the European Union NextGenerationEU within the Framework of Component 17 of the Recovery, Transformation and Resilience Plan. (ref C17.I01.P01).0.

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## Operando characterization of bi-functional Co catalyst for Zn-H<sub>2</sub> energy storage

Martha Stando<sup>a, b, \*</sup>, Merel Rittel<sup>b</sup>, Peter Strasser<sup>b</sup>, Jakub Drnec<sup>a</sup>

<sup>a</sup> European Synchrotron Radiation Facility, 71 Avenue des Martyrs, 38000 Grenoble, France

<sup>b</sup> The Electrochemical Energy, Catalysis, and Materials Science Laboratory, Department of Chemistry, Technical University Berlin, Strasse des 17. Juni 124, 10623 Berlin, Germany

\*Presenting author's email: martha.stando@esrf.fr

Recently, a novel Zn-H<sub>2</sub> system has been proposed, where energy is stored in the form of zinc, and released as hydrogen and electricity, reaching high overall efficiencies up to 50%. Implementation of this technology requires development of a bi-functional catalyst stable in the 6.9M KOH and saturated ZnO electrolyte, with high activity for both the oxygen and hydrogen evolution reactions (HER and OER) during charge and discharge respectively.

Out of several Co and Ni based candidates (Figure. 1), a microwave synthesized Co catalyst (Co<sub>sol</sub>) shows promise, displaying a large decrease in reaction overpotentials over prolonged cycling in 6.9M KOH. Moreover, in the presence of ZnO, it was the sole material that retained satisfactory performance over time. Presently, the reasons for the superiority of Co<sub>sol</sub> remain unclear, where the catalyst surface is subject to significant changes as the applied potential is adjusted to induce the OER and HER in an alternating manner. Moreover, the interaction between ZnO in the electrolyte and the catalyst is not understood.

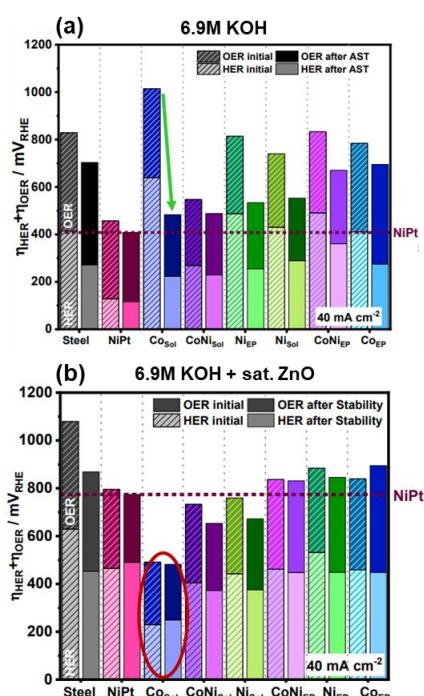


Figure 1. Recorded overpotentials for catalysts before and after ASTs (a) in 6.9M KOH, (b) in 6.9M KOH + sat. ZnO.

In order to resolve these unknowns, the Co<sub>sol</sub> catalyst underwent full operando characterization using wide-angle X-ray scattering (WAXS) at the ID31 beamline. Use of grazing incidence geometry allowed for acquisition of high-quality diffraction patterns throughout operation, including accelerated stress testing (AST). The catalyst was studied in pure 6.9M KOH, as well as in the presence of ZnO, decoupling the structural changes of the catalyst itself throughout operation from the impact of the latter. Additionally, a Co catalyst synthesized using electrodeposition (Co<sub>EP</sub>) was investigated in the same conditions in order to elucidate what aspect of the Co<sub>sol</sub> catalyst is key to its improved performance.

For both materials, in both electrolytes, Co(OH)<sub>2</sub> phase was found to increase with cycling. In addition, a significant amount of Co<sub>3</sub>O<sub>4</sub> was also present in the case of Co<sub>sol</sub>. In the ZnO containing electrolyte, ZnFe<sub>2</sub>O<sub>4</sub> build up under oxidative conditions was evident, yet it was more gradual for Co<sub>EP</sub>, commencing only after several hours of cycling, and almost instantaneous for Co<sub>sol</sub>, present already after initial catalyst activation. Furthermore, in the case of the latter at approximately 1.5V vs RHE an abrupt phase shift was noted, potentially corresponding to substantial lattice strain or transition to either CoFe<sub>2</sub>O<sub>4</sub> or Fe<sub>3</sub>O<sub>4</sub> spinels. This unique behavior of ZnFe<sub>2</sub>O<sub>4</sub> is most definitely a crucial aspect towards the improved Co<sub>sol</sub> stability and low overpotentials.

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## Removal of antibiotics by photoelectro-Fenton process at neutral pH using a filter-press cell with a sustainable gas-diffusion cathode

Paola Tirira<sup>a,\*</sup>, Panagiota Petsi<sup>b</sup>, Konstantinos V. Plakas<sup>b</sup>, Ignasi Sirés<sup>a</sup>

<sup>a</sup> Laboratori d'Electroquímica dels Materials i del Medi Ambient, Departament de Ciència de Materials i Química Física, Secció de Química Física, Facultat de Química, Universitat de Barcelona, Martí i Franquès 1-11, 08028, Barcelona, Spain

<sup>b</sup> Chemical Process and Energy Resources Institute, Centre for Research and Technology-Hellas, 6th km of Charilaou-Thermis, GR57001 Thessaloniki, Greece  
\*tirirapaola@ub.edu

In recent years, antibiotics like sulfadoxine (SDX) have been routinely detected in aquatic environments, which is demonstrated to affect microorganisms and disrupt ecosystems by promoting the growth of antibiotic-resistant bacteria (1). Considering the limitations of conventional wastewater treatment methods to remove these drugs, advanced oxidation processes (AOPs), particularly photoelectro-Fenton (PEF), have emerged as a promising alternative for the complete elimination of antibiotics from water and wastewater. Although the traditional Fenton reaction operates optimally at acidic conditions, recent studies have addressed the operation under a more extended pH range, reaching near-neutral conditions, by using iron chelating agents (2). As a matter of fact, this strategy also aims at preventing the production of iron sludges. On the other hand, the use of waste biomass as a resource for the development of carbonaceous cathodes in electrochemical water and wastewater treatments offers an innovative solution to waste management challenges, contributes to the circular economy (3), and promotes the advancement of eco-friendly treatments. In this study, a more sustainable gas-diffusion electrode prepared from wood scrap-derived biochar was employed in filter-press reactor to treat solutions of 2.5 L of 5 mg L<sup>-1</sup> SDX + 0.050 M Na<sub>2</sub>SO<sub>4</sub> at neutral pH (= 7). After 60 min of electrolysis, the biochar-based cathode enabled the accumulation of ~60 mg L<sup>-1</sup> H<sub>2</sub>O<sub>2</sub> under a recirculation flow rate of 180 L h<sup>-1</sup>, at a current density of 20 mA cm<sup>-2</sup> and 25 °C. Likewise, SDX degradation tests were carried out using different concentrations of Fe(III)-DTPA, a commercial fertilizer, as homogeneous catalyst. A comparison with other AOPs confirmed that PEF process was more efficient for the SDX degradation. Therefore, complete removal of SDX at pH 7 was attained with 0.1 mM Fe(III)-DTPA fertilizer after 45 min; additionally, a plausible degradation pathway for SDX was finally proposed based on the identified transformation byproducts.

### Acknowledgements

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## Performance of different hydrogel separators in microbial fuel cells

Isabel Torrejón<sup>a,\*</sup>, Carlos Martín<sup>b</sup>, Rafael Granados<sup>a</sup>, Alberto Rodríguez<sup>a</sup>, Manuel A. Rodrigo<sup>a</sup>, Ester Vázquez<sup>b</sup>, Carmen M. Fernández<sup>a</sup>

<sup>a</sup> Dpto. de Ingeniería Química, Universidad de Castilla-La Mancha, Avda. Camilo José Cela, 12, 13071, Ciudad Real

<sup>b</sup> Instituto Regional de Investigación Científica Aplicada (IRICA), Universidad de Castilla-La Mancha, Avda. Camilo José Cela, 1, 13005, Ciudad Real

\*Presenting author's email: isabel.torrejon@uclm.es

### Abstract

Hydrogels are soft materials formed by a three-dimensional polymeric network that preserves its integrity through physical or chemical crosslinking. These materials also have unique properties such as biocompatibility, porous structure, and electrical conductivity, which make hydrogels highly suitable for electrochemical biosensing applications and development (1). In contrast, microbial fuel cell (MFC) technology enables simultaneous wastewater treatment and sustainable energy production. Several factors affect the performance of MFCs, including operating conditions, reactor design, electrode materials, and membrane types. This study aims to investigate the influence of different hydrogels on the power density of MFCs. To this end, eight types of hydrogels made from synthetic monomers and natural polymers were tested in double-compartment cells, designed using 3D printing. Carbon cloths were used as anionic and cationic electrodes, setting an electrode-hydrogel-electrode assembly (HEA). The results highlight hydrogels as a promising sustainable alternative to proton exchange membranes (PEMs) in MFCs (2).

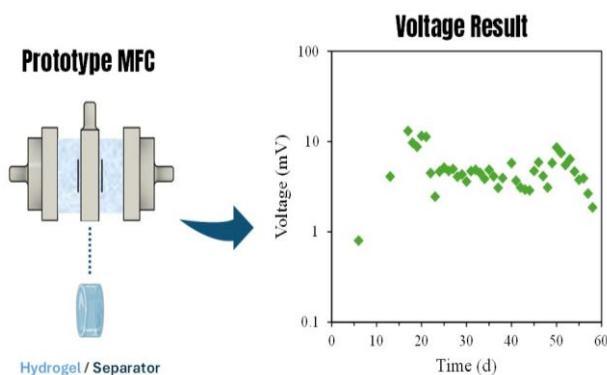


Figure 1. Prototype MFC incorporating a hydrogel and voltage output generated by the MFC over time.

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## Closing the loop: electrochemical degradation of PFOA in green solvents matrices from anion exchange resin regeneration

Laura Villar\*, Fernando Pardo, Gabriel Zarca, Ane Urtiaga

Department of Chemical and Biomolecular Engineering, Universidad de Cantabria, Av. Los Castros 46, 39005 Santander, Spain.

\*Presenting author's email: villarl@unican.es

### Abstract

The presence of poly- and perfluoroalkyl substances (PFAS) in drinking water has given rise to global concern due to its high persistence and accumulation in the environment and living beings. The European Drinking Water Directive (2020/2184) and its subsequent transposition into Spanish regulation (RD 3/2023) have already included PFAS within the group of chemical parameters used to assess the quality of water intended for human consumption.

In this regard, the focus of our work is on the use of new regenerable ion exchange (IX) resins capable of retaining PFAS, particularly, perfluorooctanoic acid (PFOA) in their polymeric structure. As the operation progresses, the IX resin gradually becomes saturated and the exchange is no longer efficient. The main objective of this work is to identify a novel green solvent with enhanced affinity towards PFOA for the successful regeneration of the PFOA-loaded IX resin.

This approach is designed to generate a concentrated PFOA-rich stream that will be subsequently introduced into an electrooxidation (ELOX) cell to perform the electrochemical degradation of the PFOA. In conclusion, the integration of ion exchange and electrooxidation technologies offers a promising dual approach, allowing both efficient PFOA capture and their subsequent degradation, while enhancing the sustainability and reusability of the IX resins over multiple operational cycles.

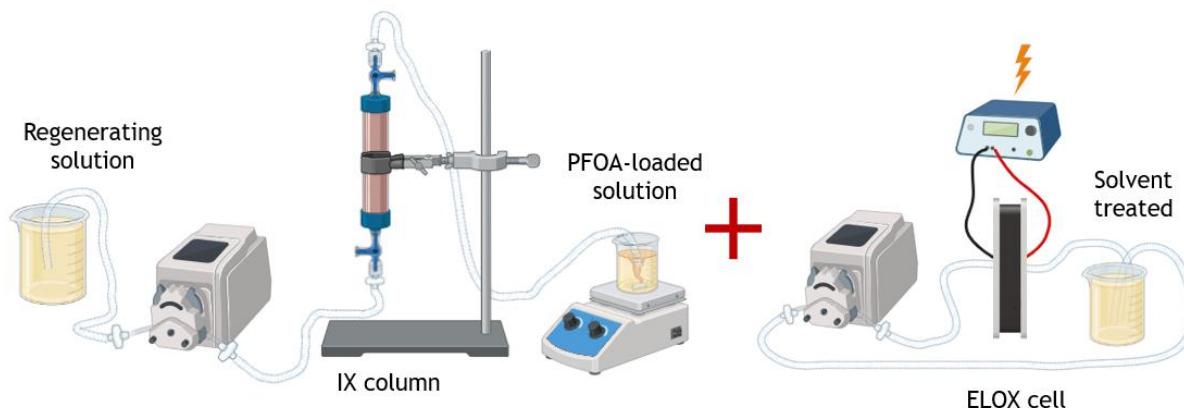


Figure 1. Experimental setup of a coupled regeneration-electrooxidation process for PFOA degradation

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## Magnetically-induced spin modulation to boost hydrogen peroxide production

Zhuowen Wang<sup>a,\*</sup>, Fengxia Deng<sup>b</sup>, Ignasi Sirés<sup>a</sup>

<sup>a</sup> Laboratori d'Electroquímica dels Materials i del Medi Ambient, Departament de Ciència de Materials i Química Física, Secció de Química Física, Facultat de Química, Universitat de Barcelona, Martí i Franquès 1-11, 08028, Barcelona, Spain

<sup>b</sup> State Key Laboratory of Urban-rural Water Resource and Environment, School of Environment, Harbin Institute of Technology, 150090 Harbin, China  
\*zhuowenwang@ub.edu

The insufficient selectivity of the oxygen reduction reaction (ORR) is a critical fundamental challenge, especially in the context of scalable electrochemical technology competing with well-established conventional methods for H<sub>2</sub>O<sub>2</sub> production. The core issue lies in the inherent spin-mismatch barrier between triplet oxygen (O<sub>2</sub>) and singlet product (H<sub>2</sub>O<sub>2</sub>), thus adding to well-known limitations in macroscopic O<sub>2</sub> mass transport to the reaction site (1). Inspired by recent findings on chirality-induced spin selectivity (CISS), which have demonstrated that can significantly boost the ORR activity (2,3), we are developing a groundbreaking magnetoelectrochemical strategy to simultaneously address both quantum and macroscopic constraints. This design aims to leverage a built-in gradient magnetic field to achieve efficient O<sub>2</sub> enrichment and directional migration within the micro-region, coupling it with the spin interfacial effect to realize spin-selective adsorption and activation of O<sub>2</sub>. To elucidate this scientific problem, this project will innovatively explore two critical areas: (i) The mechanism and structure-property relationship governing the O<sub>2</sub> micro-region mass transport driven by the gradient magnetic field; and (ii) the synergistic enhancement rules of spin-selective adsorption on the ORR pathway. This research is expected to reveal a novel coupling mechanism between micro-region mass transport and reaction dynamics, thereby pioneering a new direction for the future of electrocatalytic interface regulation and providing theoretical guidance for highly efficient electrochemical conversion.



Figure 1. Magnetically-induced ORR: A quantum-macroscopic strategy for ultra-selective oxygen reduction

### Acknowledgements

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## Imaging nanometric layer growth: in-situ and real time optical monitoring of the diazonium electrografting

Binbin Xu<sup>a,\*</sup>, Sara Helis<sup>b</sup>, Enrico Daviddi, Jean-Francois Lemineur<sup>a</sup>, Breton Tony<sup>b</sup>, Jean-Marc Noël<sup>a</sup>

<sup>a</sup> Université Paris Cité, Department/Laboratory of Electrochemistry, Paris, France

<sup>b</sup> Université d'Angers, Department/Laboratory of Chemistry, Angers, France

\*Presenting author's email: binbin.xu@etu.u-paris.fr

### Abstract

Surface functionalization with nanometric organic layers is widely used to tune interfacial physicochemical properties and fabricate active and stable materials. Among available approaches, electrochemical reduction of diazonium salts enables the formation of robustly anchored films.<sup>1</sup> Understanding the dynamics of this process requires real-time characterization techniques. While spectroscopic methods such as UV-vis or Raman spectroscopy<sup>2,3</sup> provide valuable insights, their temporal resolution remains limited. Optical microscopy, by contrast, offers high sensitivity and has been successfully applied to monitor diazonium electroreduction in real time.<sup>4</sup> Herein, we employ an accessible reflection-enhanced optical microscopy configuration to investigate in real time the electrochemical grafting and redox behavior of a bis(terpyridine) ruthenium diazonium complex on transparent electrodes. As illustrated in Figure 1, This approach, inspired from recent studies,<sup>5</sup> relies on positioning a 25  $\mu$ m platinum mirror above an ITO electrode to enhance interfacial optical contrast. Combined with RGB imaging, it enables visualization and quantitative analysis of electrochemical processes in nanometric ultrathin organic films on semitransparent electrodes.

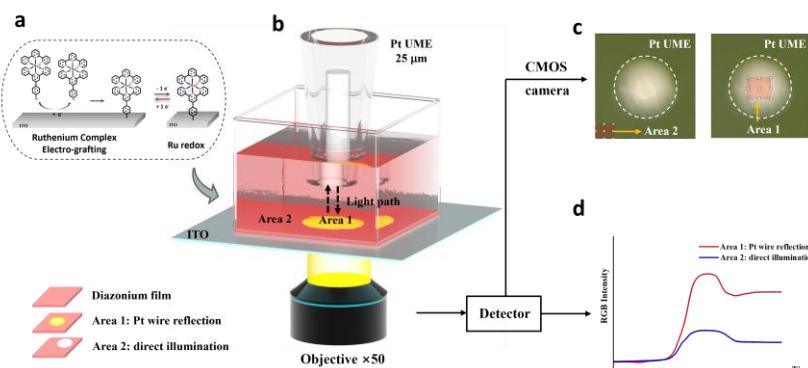


Figure 1. Reflection-enhanced setup (Pt UME above ITO) defining Area 1 (reflection) and Area 2 (direct illumination); representative RGB traces during grafting show a larger red-channel increase in Area 1.

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## Advancing electrochemical oxidation technique for complete PFAS degradation

Xueyao Zhang<sup>a,\*</sup>, Jurate Kumpiene<sup>a</sup>, Ivan Carabante<sup>a</sup>

<sup>a</sup> Waste Science and Technology, Luleå University of Technology, 97187 Luleå, Sweden

\*Email: xueyao.zhang@ltu.se

### Abstract

Per- and polyfluoroalkyl substances (PFASs), also known as “forever chemicals”, are increasingly recognized as major toxic pollutants worldwide. Electrochemical oxidation (EO) has emerged as a promising method for PFAS degradation,<sup>1</sup> involving the oxidation of PFAS compounds by continuous shortening through functional-group cleavage.<sup>1</sup> Complete mineralization of PFASs into fluoride and carbon dioxide is the ultimate goal (Figure 1). However, tracking, identifying, and quantifying the relevant degradation products during the EO remains challenging.<sup>2</sup> In addition, the degradation mechanisms of PFASs are not yet fully understood. Therefore, completing the fluorine mass balance for the degradation of different PFASs is essential.

In this project, we aim to close the fluorine mass balance during the EO process and establish a comprehensive fluorine profile for each degradation product formed from various PFASs. Achieving this requires combining multiple analytical detection methods and advancing our understanding of electrochemical principles. The results will help better understand the full degradation process pathway and support the continued development of electrochemical oxidation towards an established PFAS treatment method.

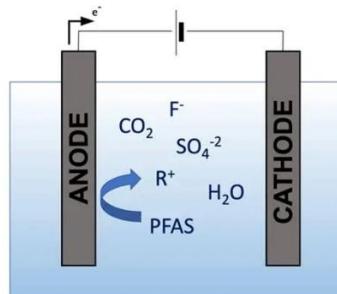


Figure 1. Mechanism of Electrochemical Oxidation of PFAS.<sup>3</sup>

### Acknowledgements

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