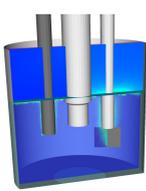




# CINTECX CHALLENGE 2024



## Optimization and scaling-up of electrochemical water splitting for H<sub>2</sub> generation



Bioingeniería y  
Procesos Sostenibles

Universidade de Vigo



Grupo Novos Materiais

### Introduction

#### Selection of OER\* catalyst\*\*

Potato peels hydrochar (PP-HC) [1,2]  
Laser oxidation [3]

\*OER: Oxygen evolution reaction

\*\*Results obtained out of project Challenge 2023



#### Scale-up validation

Catalytic working electrode { 1 cm<sup>2</sup>  
10 cm<sup>2</sup> }  
Electrochemical and hydrodynamic studies

#### Scale-up simulations



The reactor geometry was modelled in order to fit its thermodynamic response during water splitting processes.

### Materials and methods

#### Optimal electrode-laser modification



Application of 371.3 mJ/cm<sup>2</sup> in 10 repetitions to PP-HC electrode, obtaining PP-HC-laser.

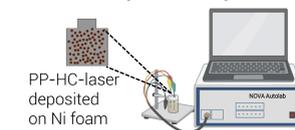
Industrial Deep UV-Excimer laser LEAP-60A

#### Surface analysis



PLu Neox, Sensofar, USA

#### Electrocatalytic activity

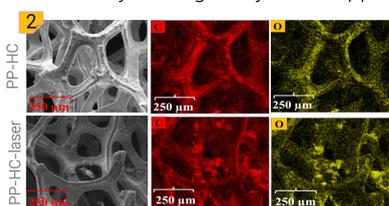


Reference electrode	Ag/AgCl
Counter electrode	Graphite stick
Working electrode	Catalytic electrode (Ni foam+0.25 mg/cm <sup>2</sup> catalyst)
Solution	NaOH 0.1 M
Potential window	0 - 0.74 V
Scan rate	0.01 V/s

### Results

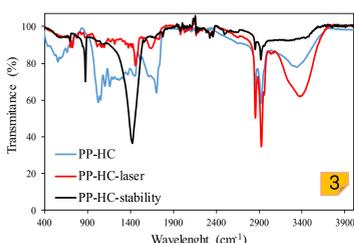
#### Evaluation of the laser homogeneity

The unreported preparation of the PP-HC-laser electrode resulted suitable, considering the reproducibility of the physicochemical properties and the intensity homogeneity of the applied laser (Fig.1).



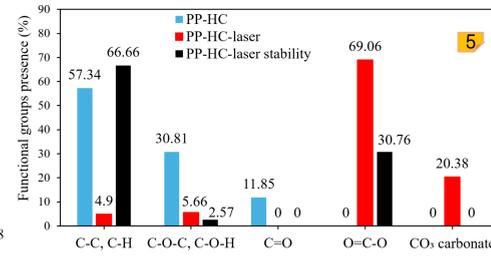
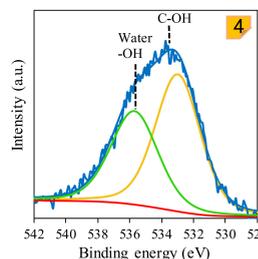
#### Functional groups analysis

PP-HC is homogeneously distributed on the Ni foam electrode (Fig. 2). Slight differences were found between PP-HC and PP-HC-laser, apart from the higher oxygen content which demonstrates the oxidation capacity of the laser beam.



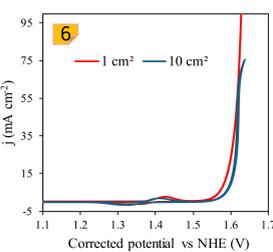
Moreover, FTIR spectra (Fig.3) verified more clearly the laser treatment enhanced oxygenated functional groups presence, reducing the alkenes content.

Moreover, the oxygenation of the surface (Fig. 4) after laser application is tangible, reducing C-C and C-H groups (Fig. 5).



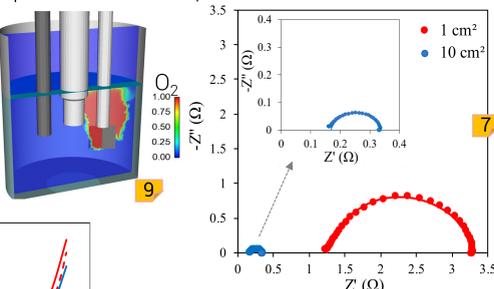
#### Electrochemical analysis

Increasing the catalytic working electrode area 10 times resulted in a practically constant electrochemical response (Fig. 6).

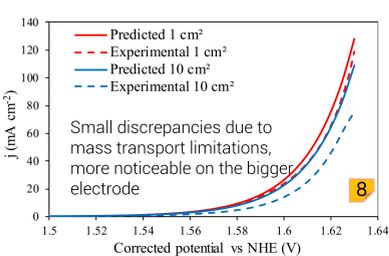


#### Electrochemical impedance spectroscopy

The electrochemical impedance spectroscopy (EIS), showed better results for the bigger electrode (Fig. 7), showing the scale-up of the process would be possible.



#### Scale-up simulations



Experimental and CFD predicted results were compared (Fig. 8), showing the power of CFD model to predict the electrocatalytic performance. Moreover, the gas released was simulated (Fig. 9).

#### Surface analysis

The catalytic electrode has higher roughness (Rz, Rq, Ra) than Ni foam, which has a smoother surface (Rh).

Laser treatment reduces slightly roughness (Rz, Ra) and peak height (Rh) due to the catalyst melting within Ni foam.

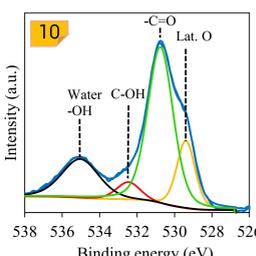
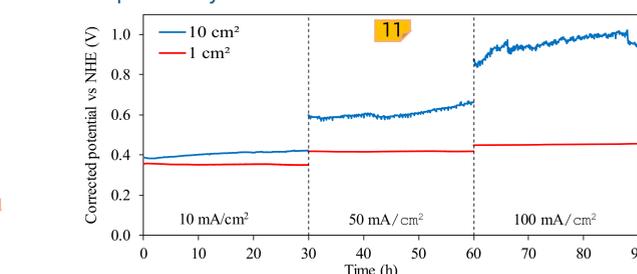
#### Anticipates/explains:

High stability of PP-HC-laser  
Better H<sub>2</sub> or O<sub>2</sub> liberation



Electrode	Rz (mm)	Rq (µm)	Ra (µm)	Rh (mm)
Ni foam	1.8	225	176	0.68
PP-HC	3.4	210	154	1.7
PP-HC-laser	3.2	245	127	1.5

#### Chronoamperometry studies



The long-term usage of the electrodes caused surface oxidation (Fig. 10). More stability is required at higher electrode sizes (Fig. 11), thus, as main conclusions, it is needed to:

- Explore new electrode preparation options
- Apply modifications to enhance stability
- Consider new cell geometries

#### References

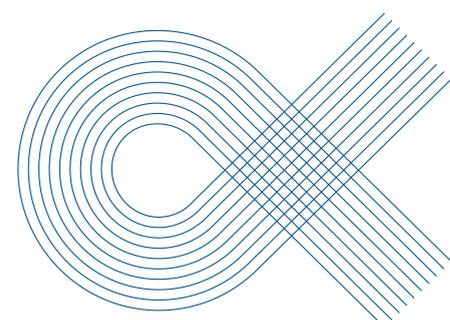
- [1] A. M. Díez, M. Bolaños-Vázquez, S. Chiussi, M. Pazos, M. A. Sanroman, Screening of agroindustry residues for their usage as oxygen evolution reaction catalysts. *J. Environ. Chem. Eng.* 12 (2024) 114527.
- [2] A.M. Díez, S. Escudero-Curiel, S. Chiussi, M. Pazos, M. A. Sanroman "From agroindustrial residues to catalysts for green hydrogen production" *10<sup>th</sup> carbocat International Symposium on Carbon for Catalysis*, Firenze, Italy, June 2024
- [3] Aida M. Díez, Stefano Chiussi, Marta Pazos, M. Ángeles Sanroman, "Laser oxidation of a carbon-based catalyst for upgrading overall water splitting performance", *44<sup>th</sup> annual meeting of the RSEQ Specialized Group in Electrochemistry +5<sup>th</sup> E3 Mediterranean Symposium*, Basque country, Spain, July 2024



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