2nd LATAM MEETING ON GREEN AMMONIA AND POWER-to-X

Materials for the electrochemical production of hydrogen

Elena Pastor

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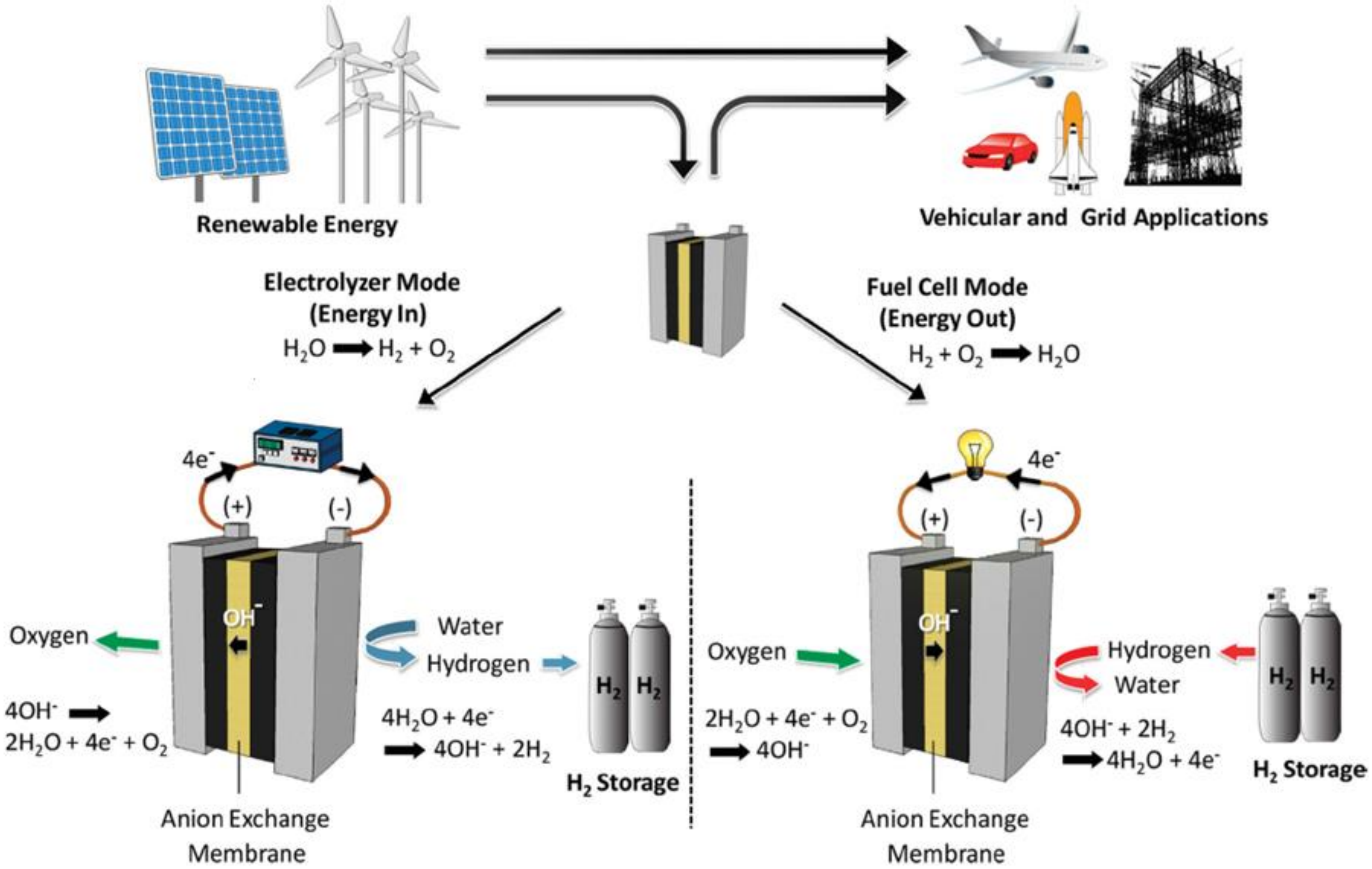
Instituto de Materiales y Nanotecnología
Universidad de La Laguna

Grupo de Ciencia de Superficies y Electrocatálisis

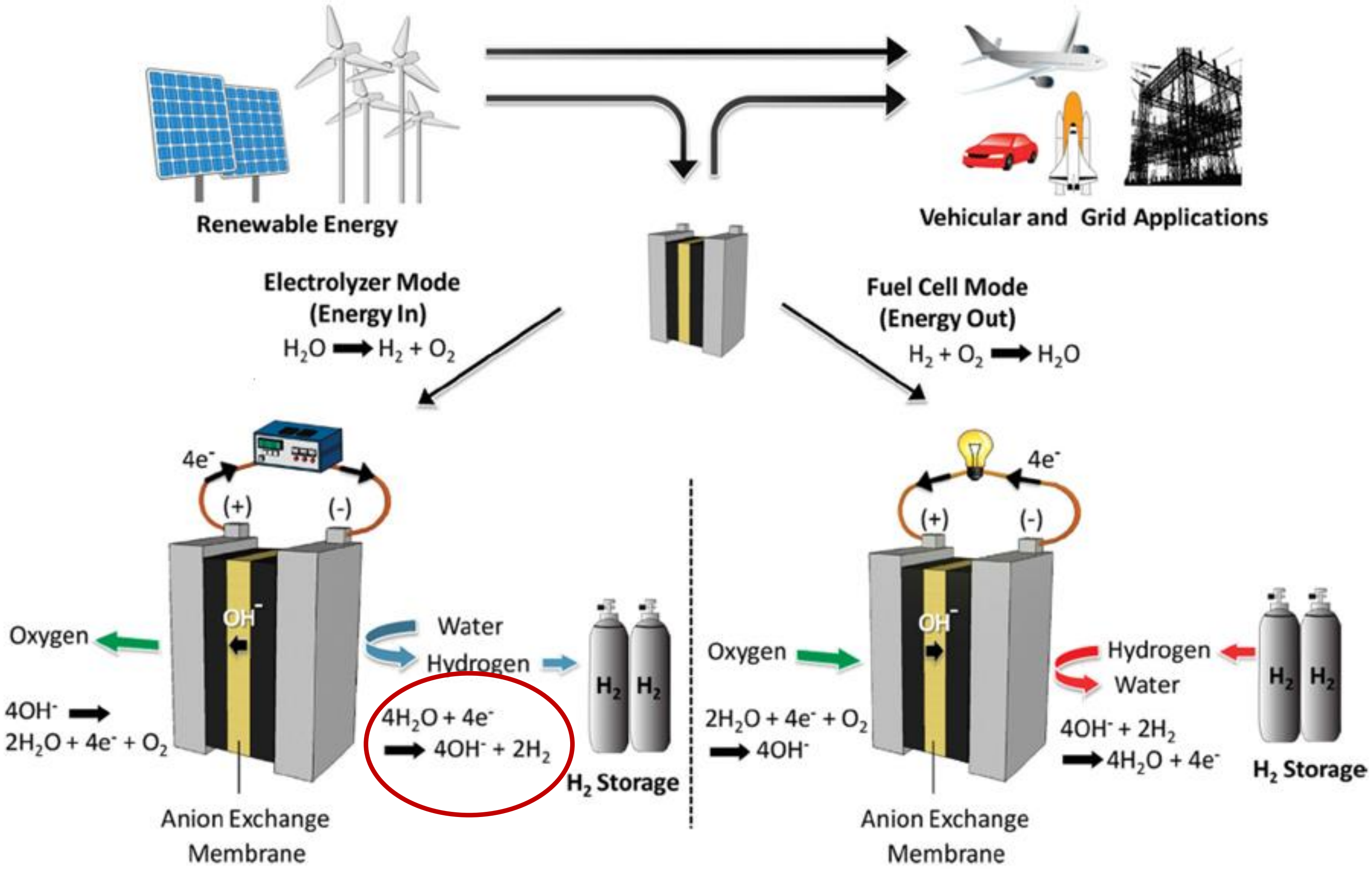
LAB H2

PEM devices



Renewable Energy
Vehicular and Grid Applications
Electrolyzer Mode
(Energy In)
H2O → H2 + O2
Fuel Cell Mode
(Energy Out)
H2 + O2 → H2O
4e-
(+)
(-)
OH-
Oxygen
Water
Hydrogen
4OH- → 2H2O + 4e- + O2
4H2O + 4e- → 4OH- + 2H2
H2
H2
H2 Storage
Anion Exchange Membrane
Oxygen
Hydrogen
Water
2H2O + 4e- + O2 → 4OH-
4OH- + 2H2 → 4H2O + 4e-
H2 Storage
Anion Exchange Membrane

PEM devices

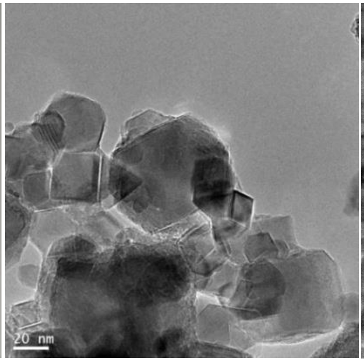


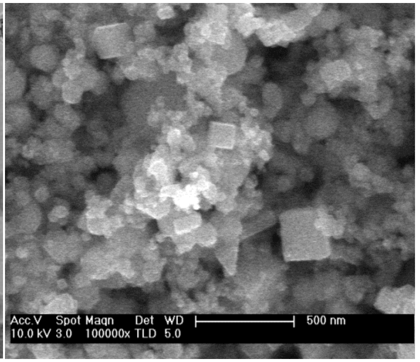
Renewable Energy
Vehicular and Grid Applications
Electrolyzer Mode
(Energy In)
$H_2O \rightarrow H_2 + O_2$
Fuel Cell Mode
(Energy Out)
$H_2 + O_2 \rightarrow H_2O$
$4e^-$
(+)
(-)
OH^-
Oxygen
Water
Hydrogen
$4OH^- \rightarrow 2H_2O + 4e^- + O_2$
$4H_2O + 4e^- \rightarrow 4OH^- + 2H_2$
H_2
H_2 Storage
Anion Exchange Membrane
$4e^-$
(+)
(-)
OH^-
Oxygen
Hydrogen
Water
$2H_2O + 4e^- + O_2 \rightarrow 4OH^-$
$4OH^- + 2H_2 \rightarrow 4H_2O + 4e^-$
H_2
H_2 Storage
Anion Exchange Membrane

Alternative: metal carbides

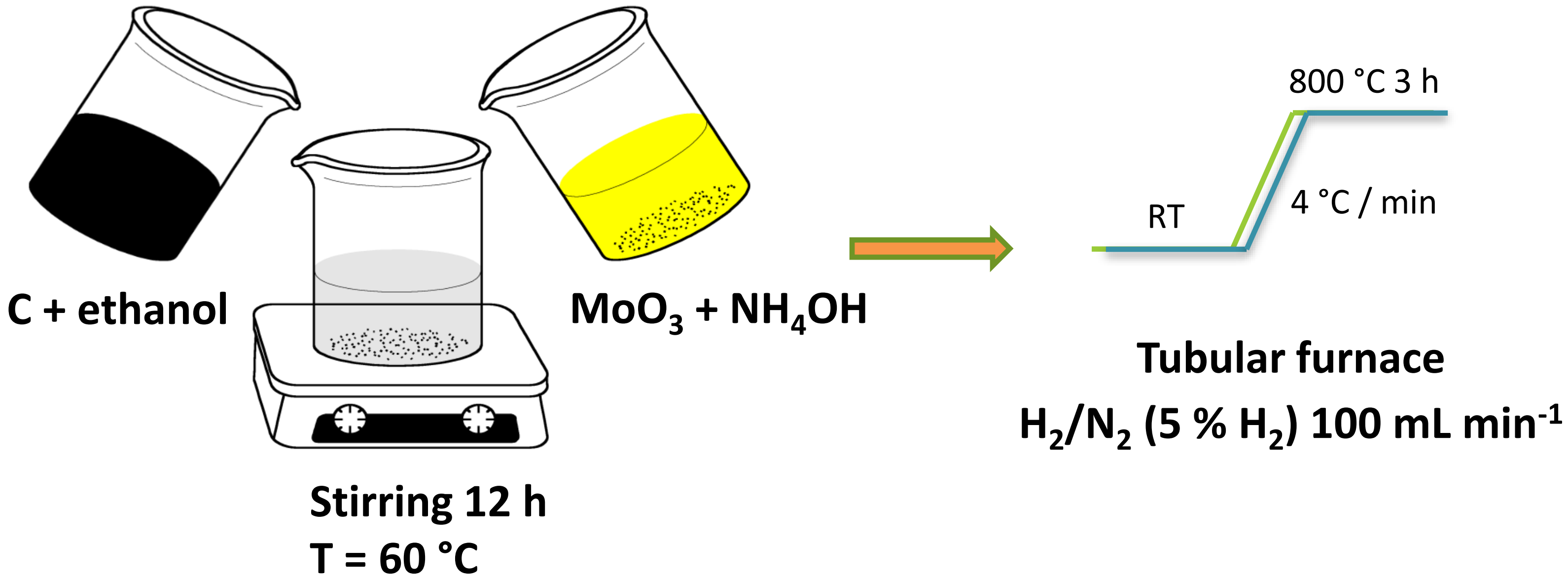
- **Could be used as catalysts, catalyst supports and/or promoters**
- DFT: electronic structure near the Fermi level similar to noble metals (e.g., Pt and WC).
- High electrical conductivity
- Corrosion and wear resistant
- Low-cost







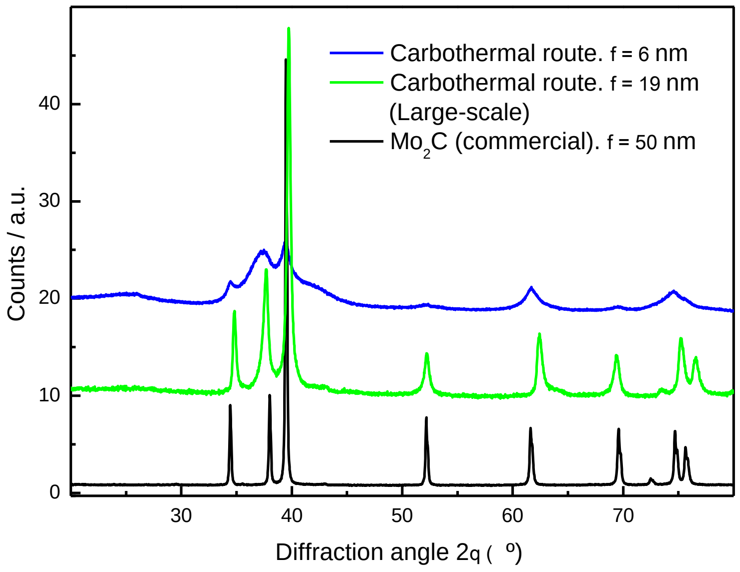
Synthesis of Mo_2C by the carbothermal route

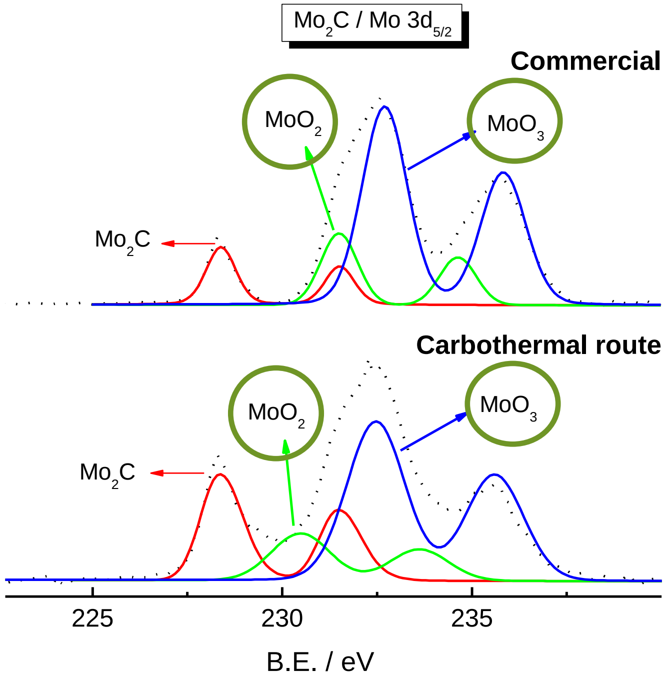


Low-cost and easy method

G. García et al. *International Journal of Hydrogen Energy 41* (2016) 19664-19673

Synthesis of Mo_2C by the carbothermal route



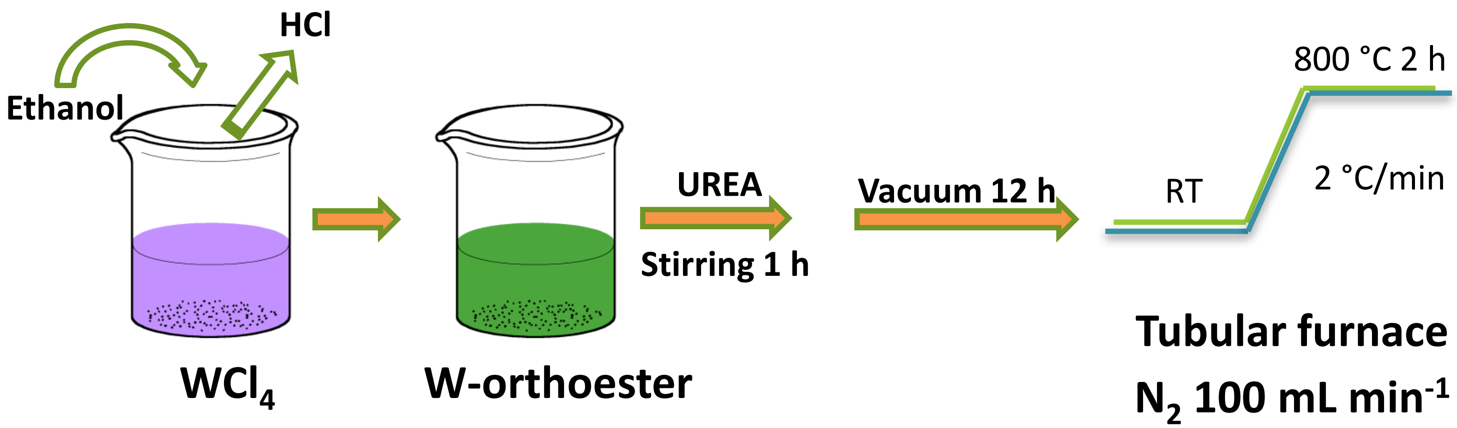


Catalyst	Main phase/s	Secondary phase/s	Crystallite Size (nm)
Mo_2C (commercial)	Mo_2C	---	> 50
Mo_2C (carbothermal route)-0.1 gram	Mo_2C	---	6
Mo_2C (carbothermal route) Large-Scale	Mo_2C	---	19

Surface: metal carbide + transition metal oxides

G. García et al. *International Journal of Hydrogen Energy 41* (2016) 19664-19673

Synthesis of W_2C by the urea-glass route



WCl_4 (1.21 M)
in CH_3CH_2OH

Urea:W molar ratio = R

R < 2 → W_2N

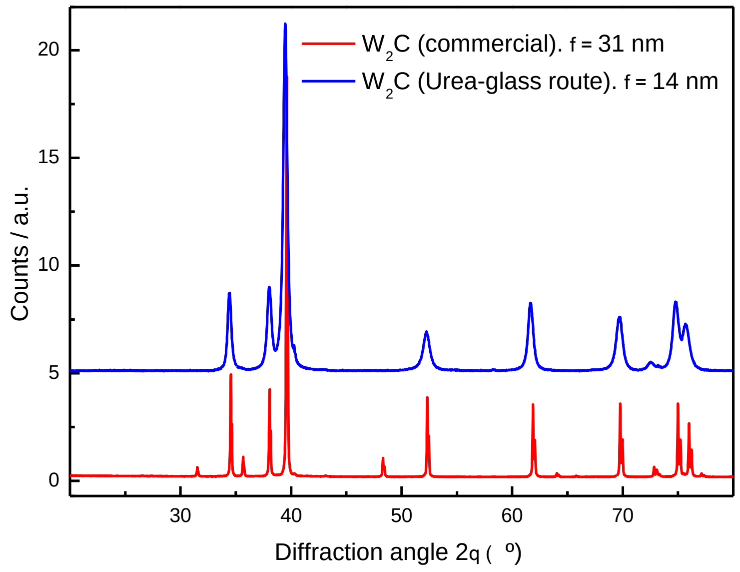
2 < R < 4 → W°

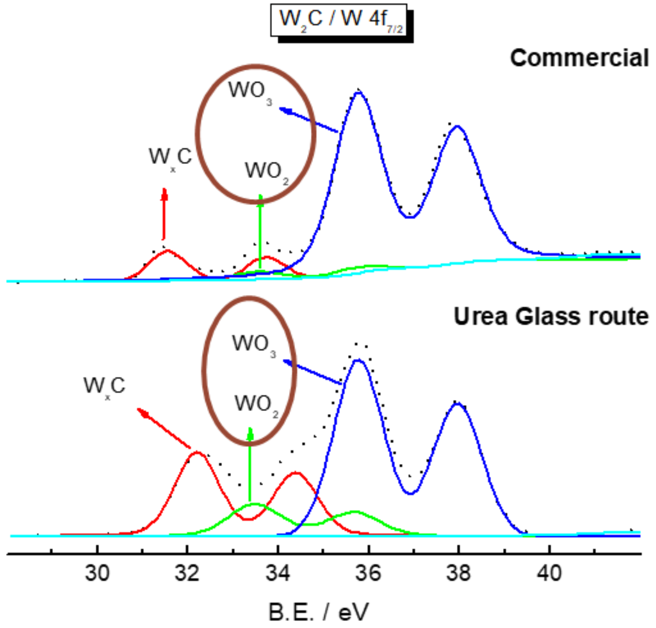
R = 7 → W_2C

Modification of
Nano Lett., Vol. 8, No. 12, **2008**

Low-cost

Synthesis of W_2C by the urea-glass route





Catalyst	Main phase/s	Secondary phase/s	Crystallite Size (nm)
W_2C (Commercial)	W_2C	WC	> 50
W_2C (urea glass route)	W_2C	----	14

Surface: metal carbide + transition metal oxides

What are REALLY metal carbides?

TRANSITION METAL CARBIDE

→

TRANSITION METAL OXIDES

+

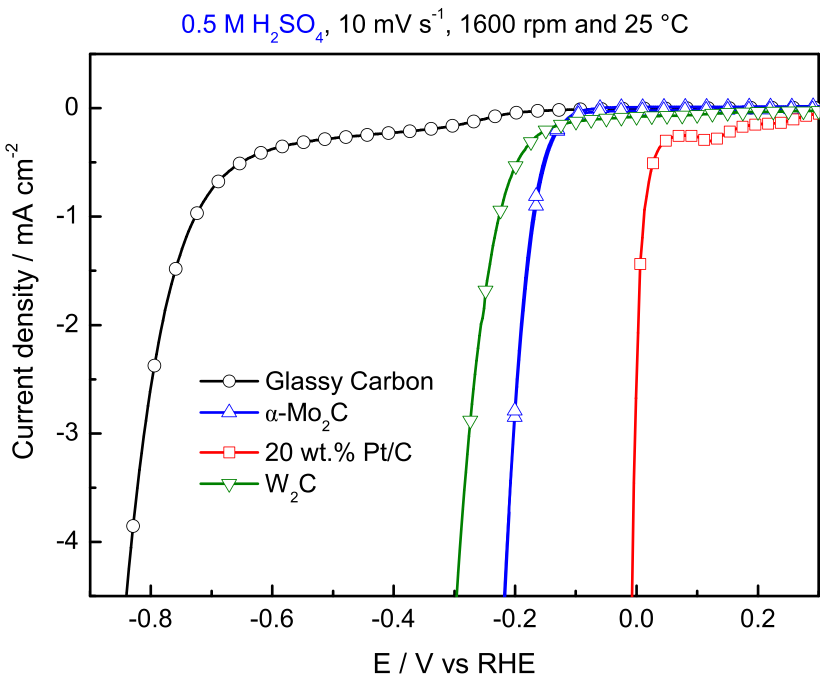
TRANSITION METAL CARBIDES

Good conductivity – Small particle size – Low-cost production ….

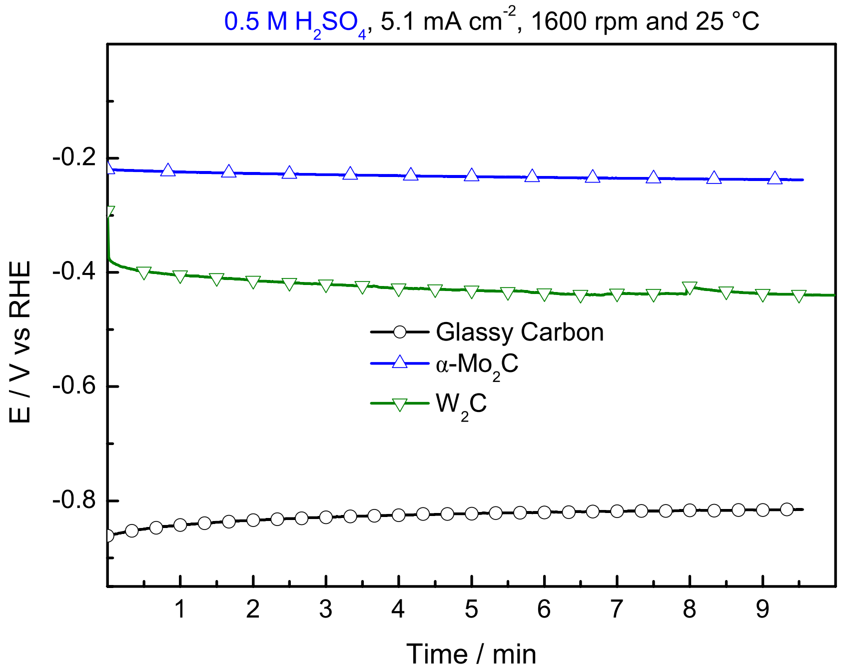
G. García et al. *International Journal of Hydrogen Energy 41* (2016) 19664-19673

Noble metal free catalysts for the HER: metal carbides

Linear Sweep Voltammetry



Chronopotentiometry

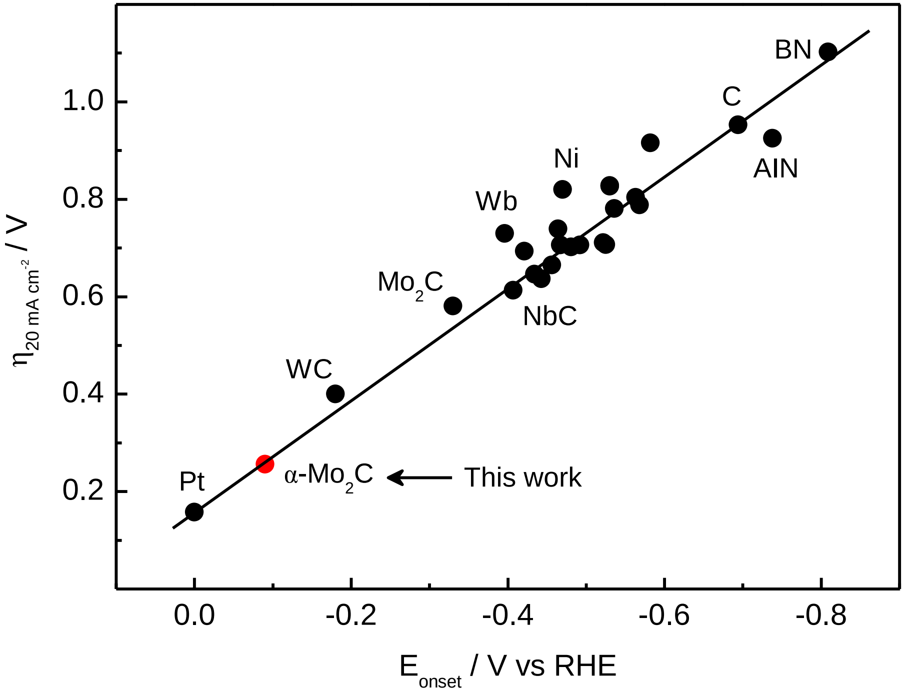


Onset potential strongly depends on the nature of the ***metal***

G. García et al. *Journal of Electroanalytical Chemistry* 793 (2017) 235

Hydrogen evolution reaction

η @ 20 mA cm^{-2} *vs* onset potential

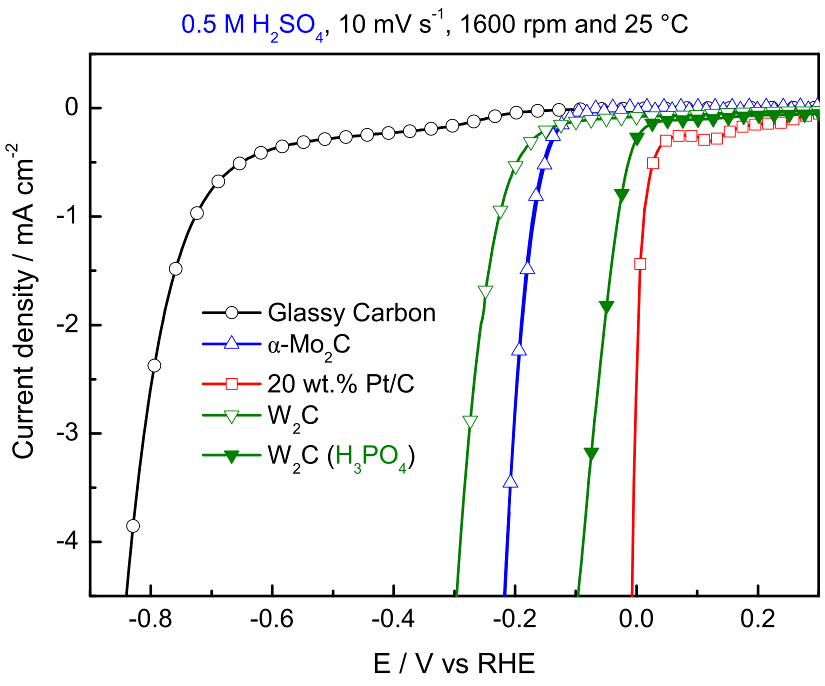


Mo$_2$C synthesized by the carbothermal route appears as one of the most active **non-noble material** toward the **HER**

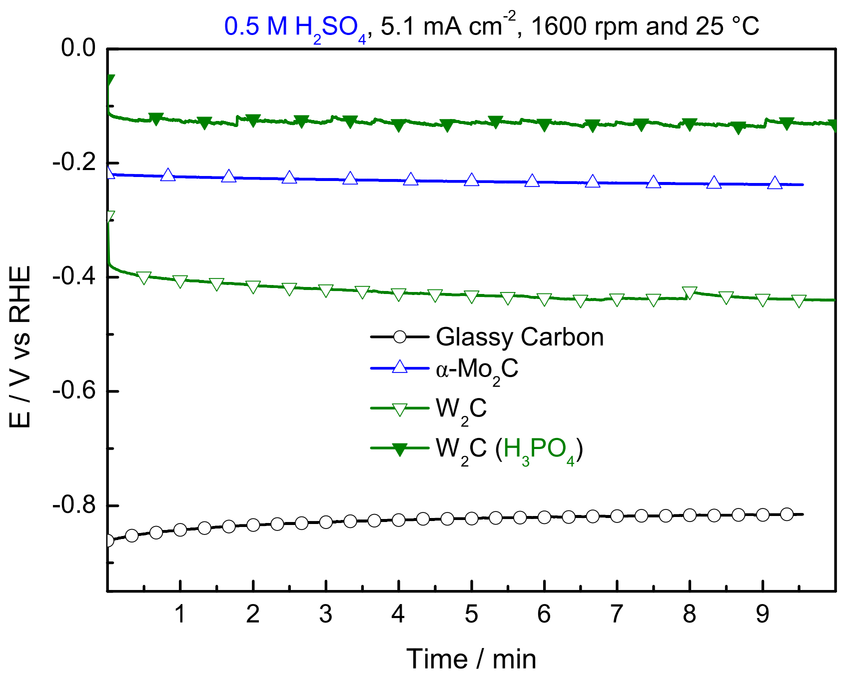
G. García et al. *Journal of Electroanalytical Chemistry* 793 (2017) 235

Hydrogen evolution reaction

Linear Sweep Voltammetry



Chronopotentiometry



Onset potential strongly depends on the nature of the ***metal*** and the **electrolyte**

G. García et al. *Journal of Electroanalytical Chemistry* 793 (2017) 235

Hydrogen evolution reaction (HER)

Mechanism

	Acid media	Basic media
Volmer →	$H_3O^+ + e^- \rightleftharpoons H_{ads} + H_2O$	$H_2O + e^- \rightleftharpoons H_{ads} + OH^-$
Heyrovski →	$H_{ads} + H_3O^+ + e^- \rightleftharpoons H_2 + H_2O$	$H_{ads} + H_2O + e^- \rightleftharpoons H_2 + OH^-$
Tafel →	$H_{ads} + H_{ads} \rightleftharpoons H_2$	$H_{ads} + H_{ads} \rightleftharpoons H_2$

Tafel slopes → η *vs* log(|j|) →

- **Volmer 120 mV·dec^{-1}**
- **Heyrovski 40 mV·dec^{-1}**
- **Tafel 30 mV·dec^{-1}**

ADVANCED ENERGY MATERIALS
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Ni-Activated Transition Metal Carbides for Efficient Hydrogen Evolution in Acidic and Alkaline Solutions

Chenfan Yang, Rong Zhao, Hui Xiang, Jing Wu, Wenda Zhong, Wenlong Li, Qin Zhang, Nianjun Yang,* and Xuanke Li**

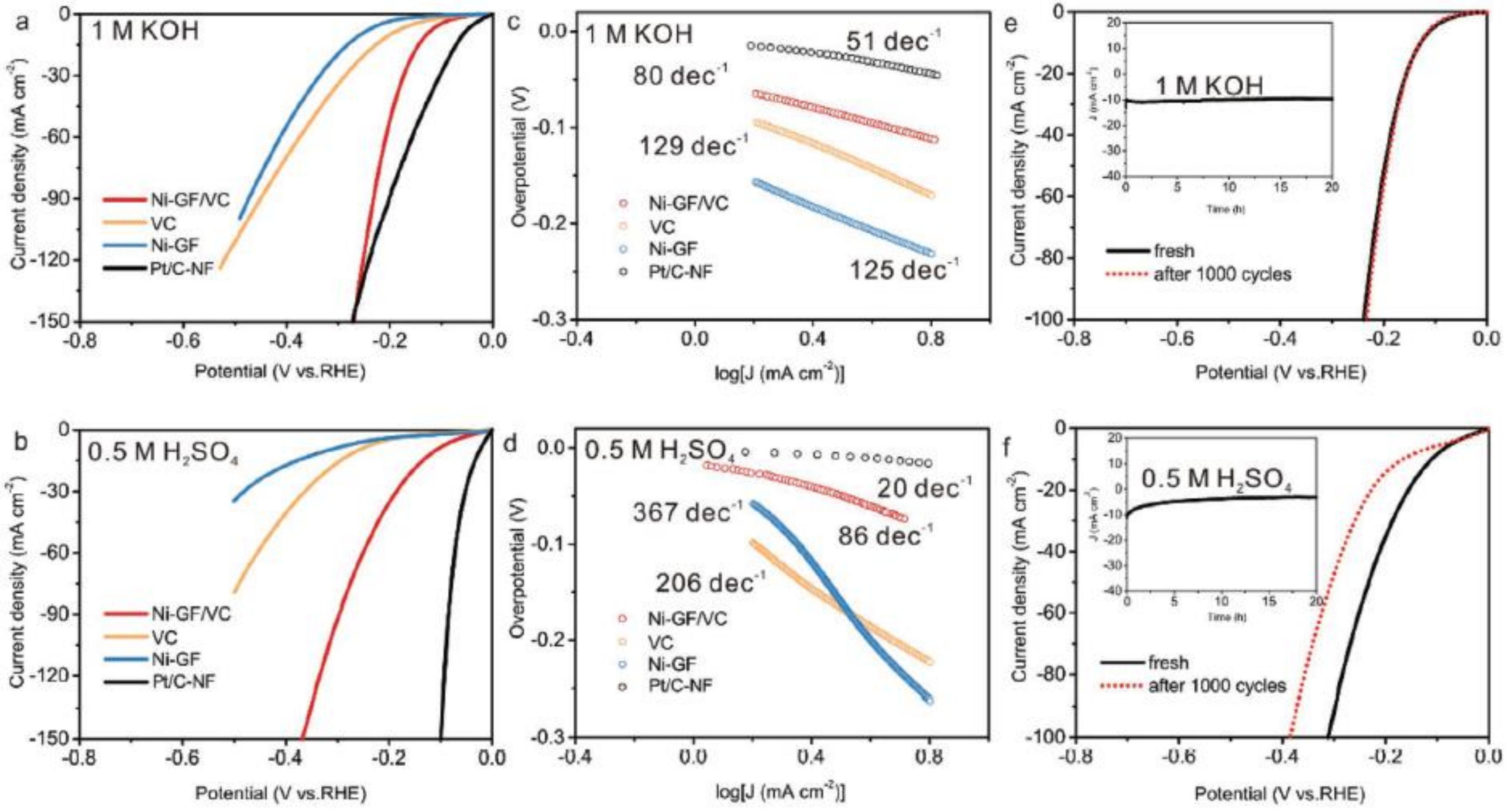
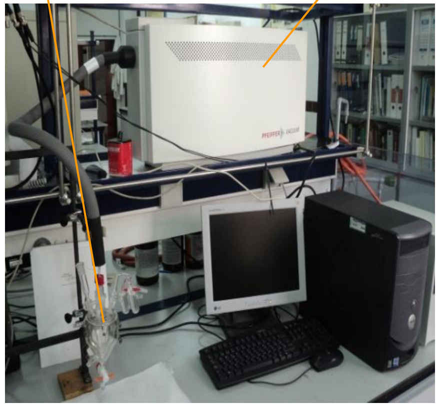
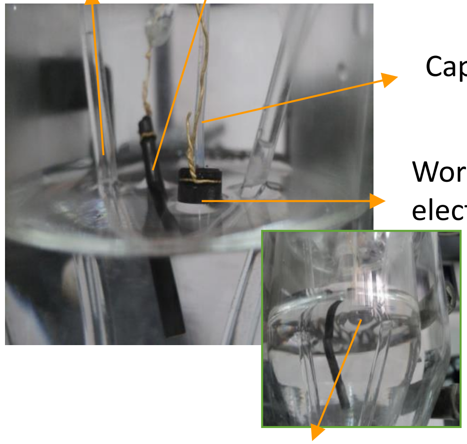


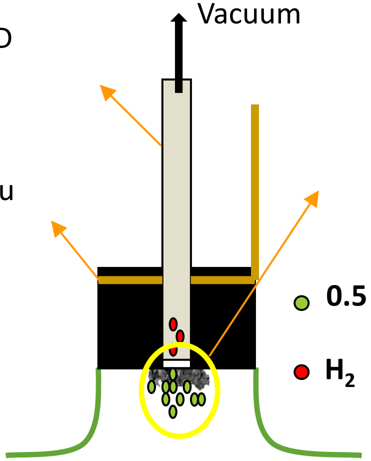
Figure 4. HER performance of the Ni-GF/VC catalyst: a,b) The *iR*-corrected LSVs and c,d) Tafel slopes of the Pt/C-NF, Ni-GF/VC, VC, and Ni-GF catalysts; e,f) the *iR*-corrected LSVs of the Ni-GF/VC catalyst in the 1st and 1000th cycles. The insets in (e) and (f) are the long-term stability tests over 20 h at a constant potential of –128 and –111 mV (vs RHE), respectively. The electrolytes were a,c,e) 1 M KOH and b,d,f) 0.5 M H_2SO_4. The scan rate in (a) and (b) is 5 mV s^{-1}.

Differential electochemical mass spectrometry (DEMS)

Pfeiffer







$$i_i = K^0 j_i$$

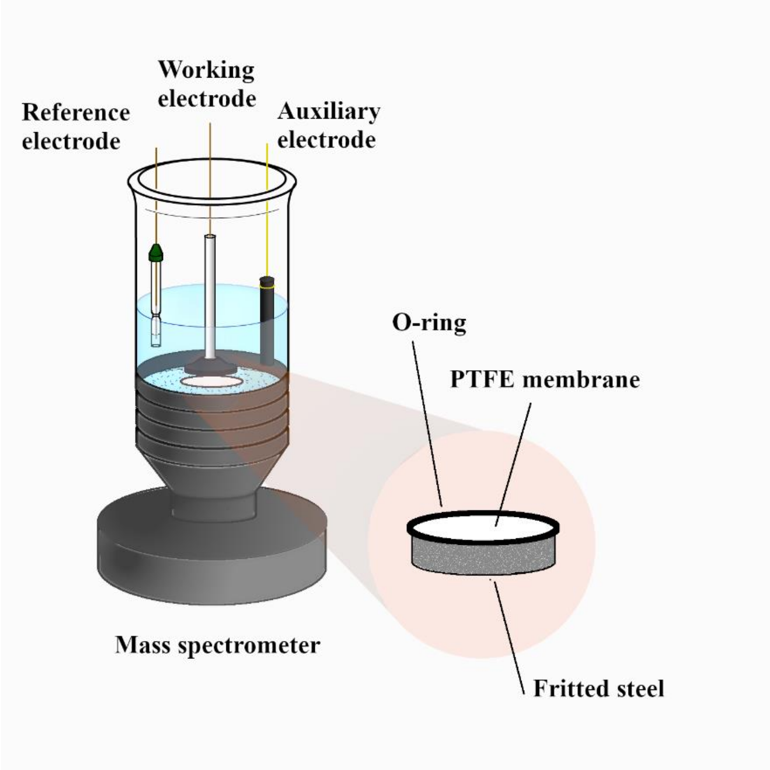
$$j_i = \frac{N\, i_f}{n\, F}$$

$$i_i \propto i_f$$

O. Guillén-Villafuerte et al.
Electrochemistry Communications 63 (2016) 48 - 51

Differential electochemical mass spectrometry (DEMS)

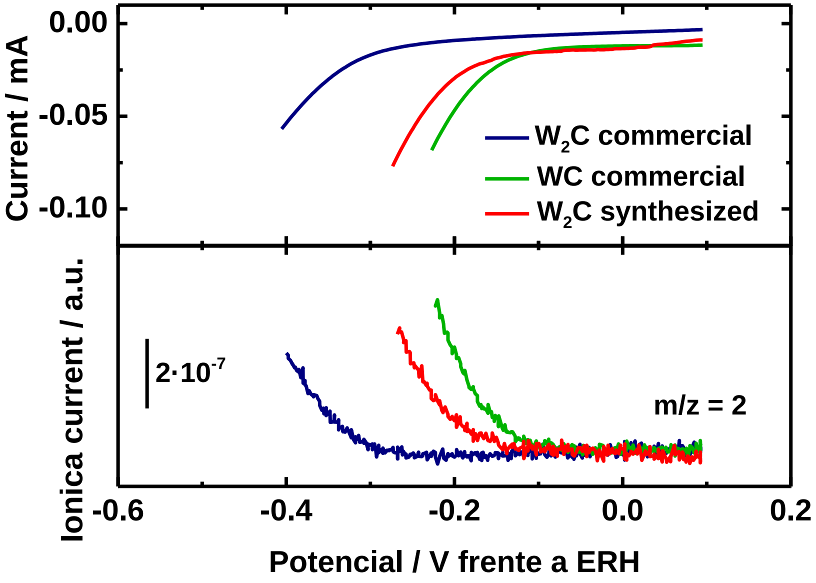
Pfeiffer
“Arturito”



Differential electochemical mass spectrometry (DEMS)

Hiden

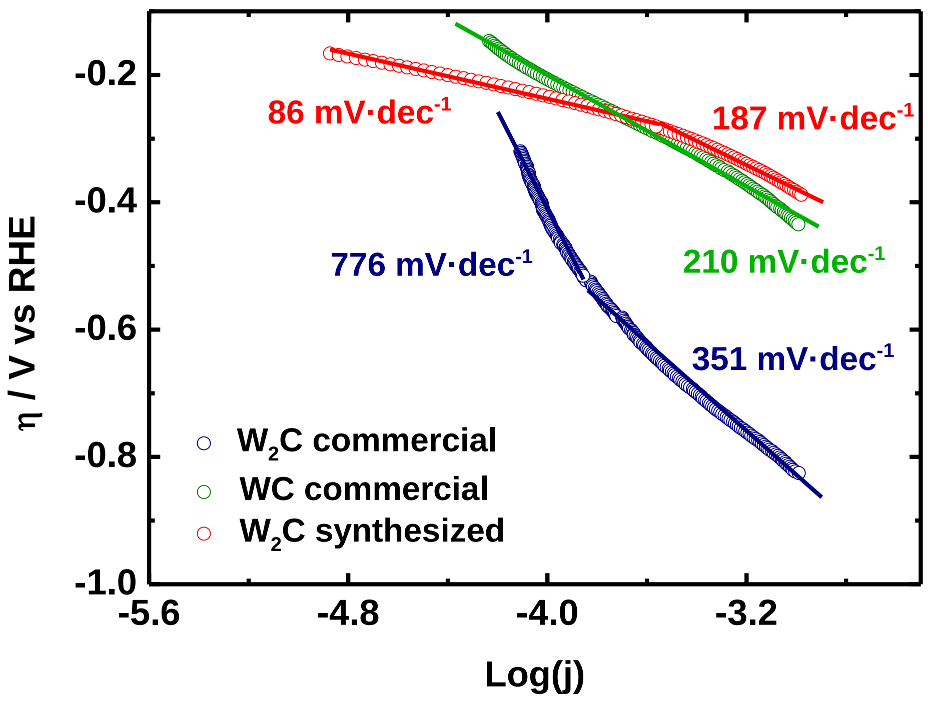
Hydrogen evolution reaction at metal carbides



0.1 M NaOH, 1 mV/s

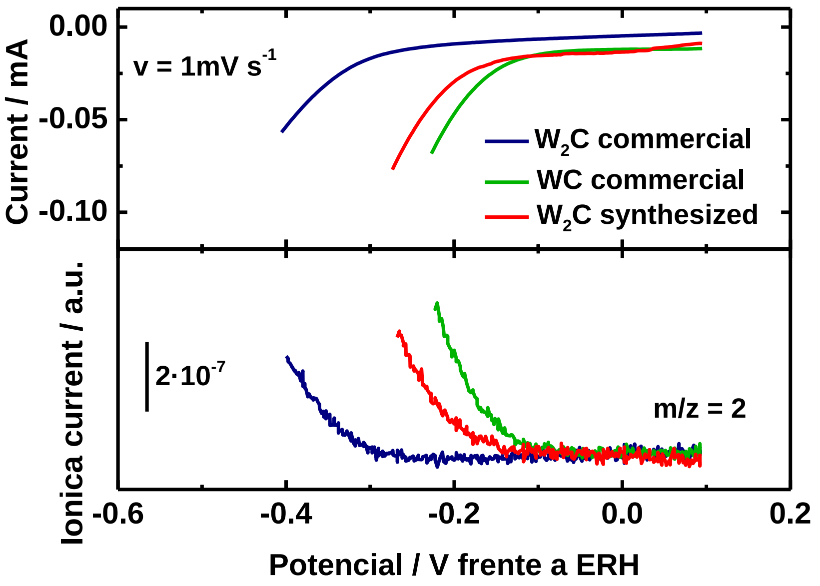
Tafel plot

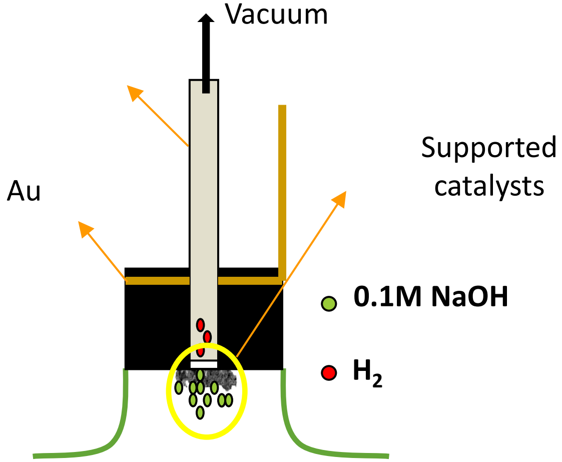
Faradaic currents



Hydrogen evolution reaction at metal carbides

DEMS

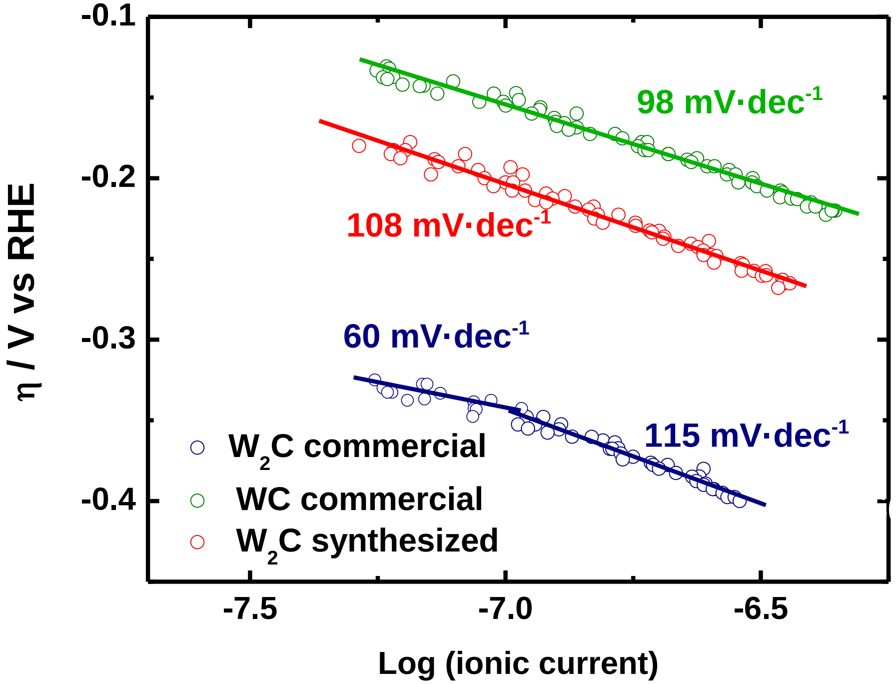




0.1 M NaOH, 1 mV/s

Ionic currents

Tafel plot



INTERNATIONAL JOURNAL OF HYDROGEN ENERGY 44 (2019) 12576–12582

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Precise determination of Tafel slopes by DEMS. Hydrogen evolution on tungsten-based catalysts in alkaline solution

S. Díaz-Coello, G. García*, M.C. Arévalo, E. Pastor

Instituto de Materiales y Nanotecnología, Departamento de Química, Universidad de La Laguna, PO Box 456, 38200, La Laguna, Santa Cruz de Tenerife, Spain

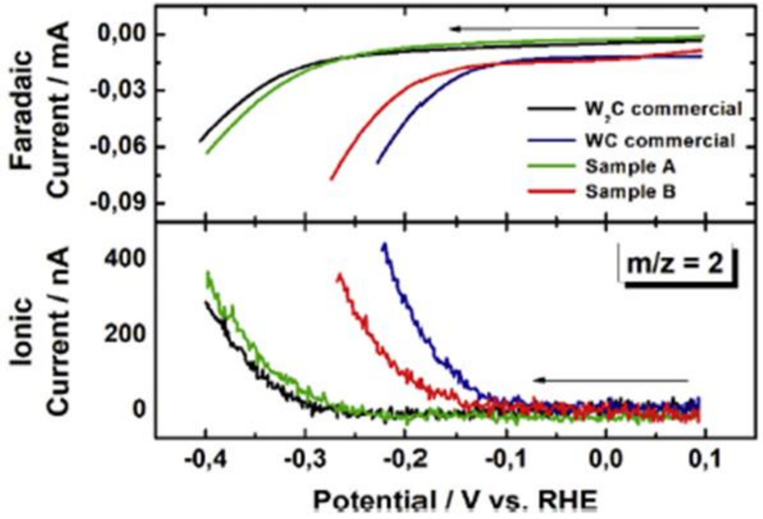


Fig. 3 – LSVs (top panel) and MSLSVs (bottom panel) for all employed catalysts in 0.1 M NaOH recorded at 1 mV s^{-1}.

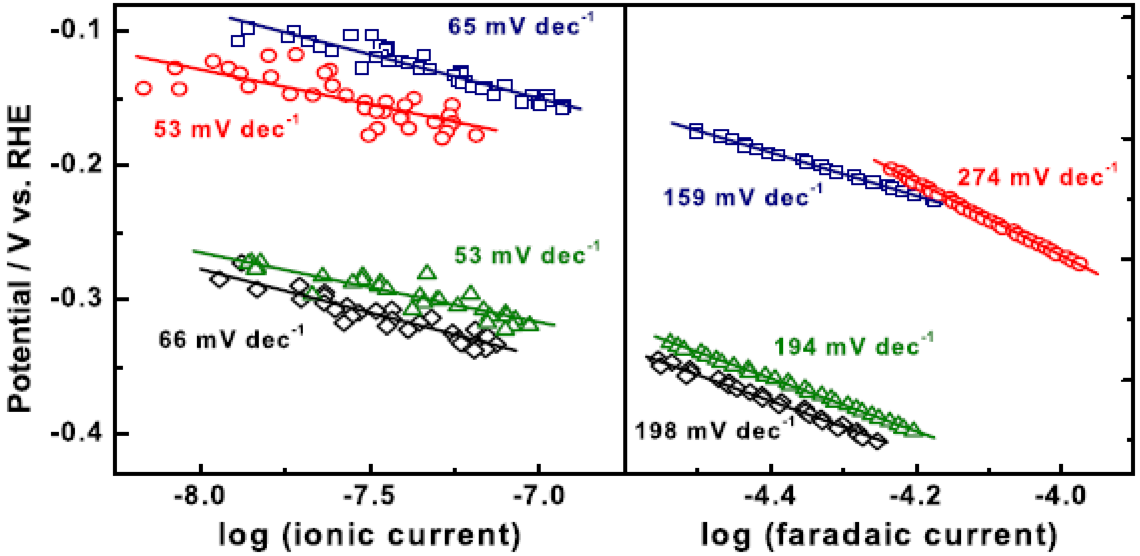


Fig. 4 – Tafel plots for the HER for commercial W_2C (black line-◇), commercial WC (blue line-□), sample A (green line-△) and sample B (red line-○) achieved from MSLSVs (left panel) and LSVs (right panel). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Hydrogen evolution reaction at metal carbides

SUMMARY

NaOH 0.1 M

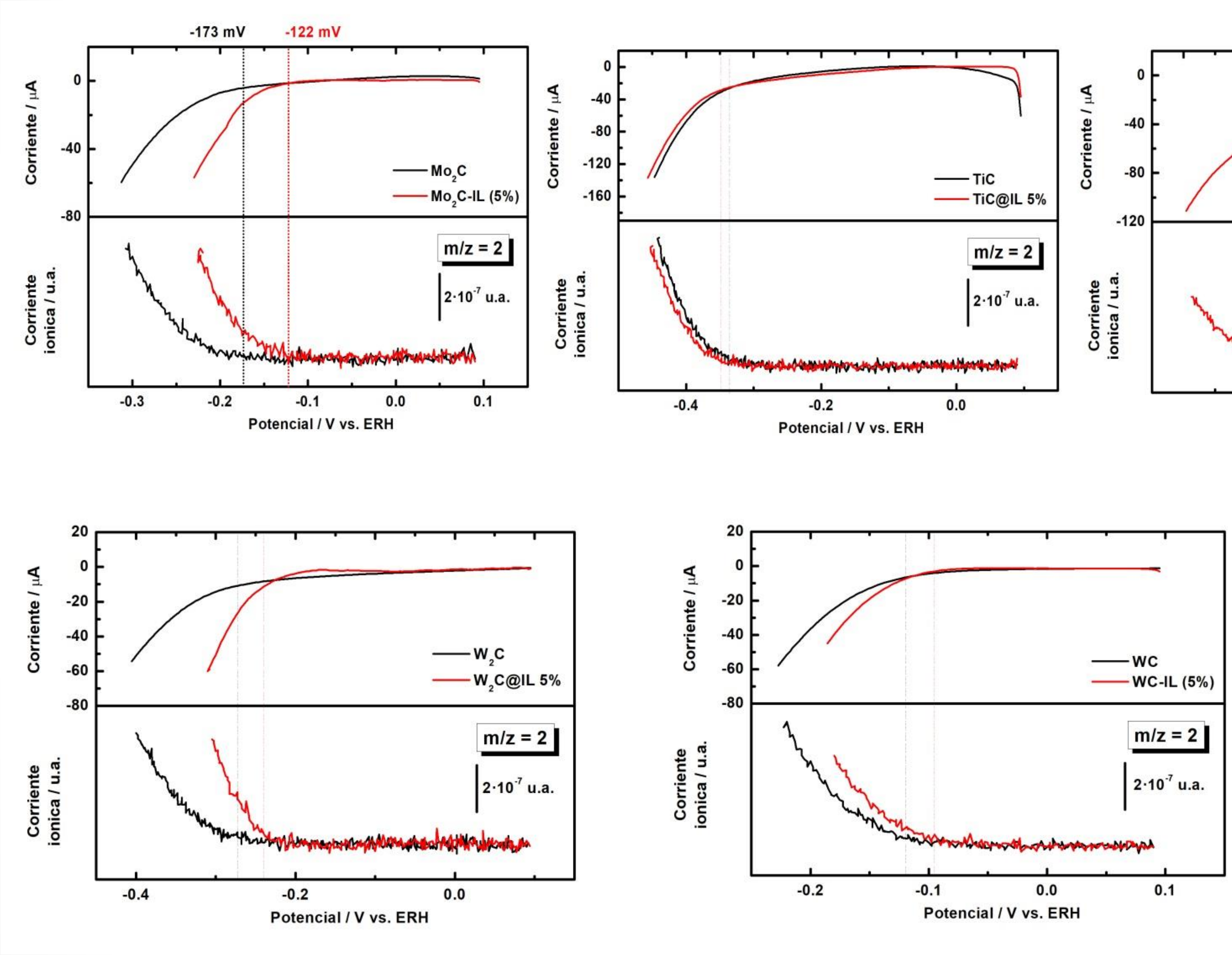
Catalyst	Onset potential	Tafel slope	rds	Faradaic current at -0.45 V vs RHE
W_2C commercial	-0.30 V vs. RHE	60, 115 mV·dec^{-1}	Heyrovsky-Volmer	100 μA
WC commercial	-0.12 V vs. RHE	98 mV·dec^{-1}	Volmer	800 μA
W_2C synthesized	-0.15 V vs. RHE	108 mV·dec^{-1}	Volmer	2.2 mA

H_3PO_4 0.5 M

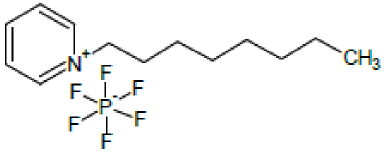
Catalyst	Onset potential	Tafel slope	rds	Faradaic current at -0.45 V vs RHE
W_2C commercial	-0.42 V vs. RHE	66,141 mV·dec^{-1}	Heyrovsky-Volmer	8 μA
WC commercial	-0.21 V vs. RHE	130 mV·dec^{-1}	Volmer	80 μA
W_2C synthesized	-0.21 V vs. RHE	113 mV·dec^{-1}	Volmer	600 μA

Hydrogen evolution reaction at metal carbides

Effect of the addition of ionic liquid



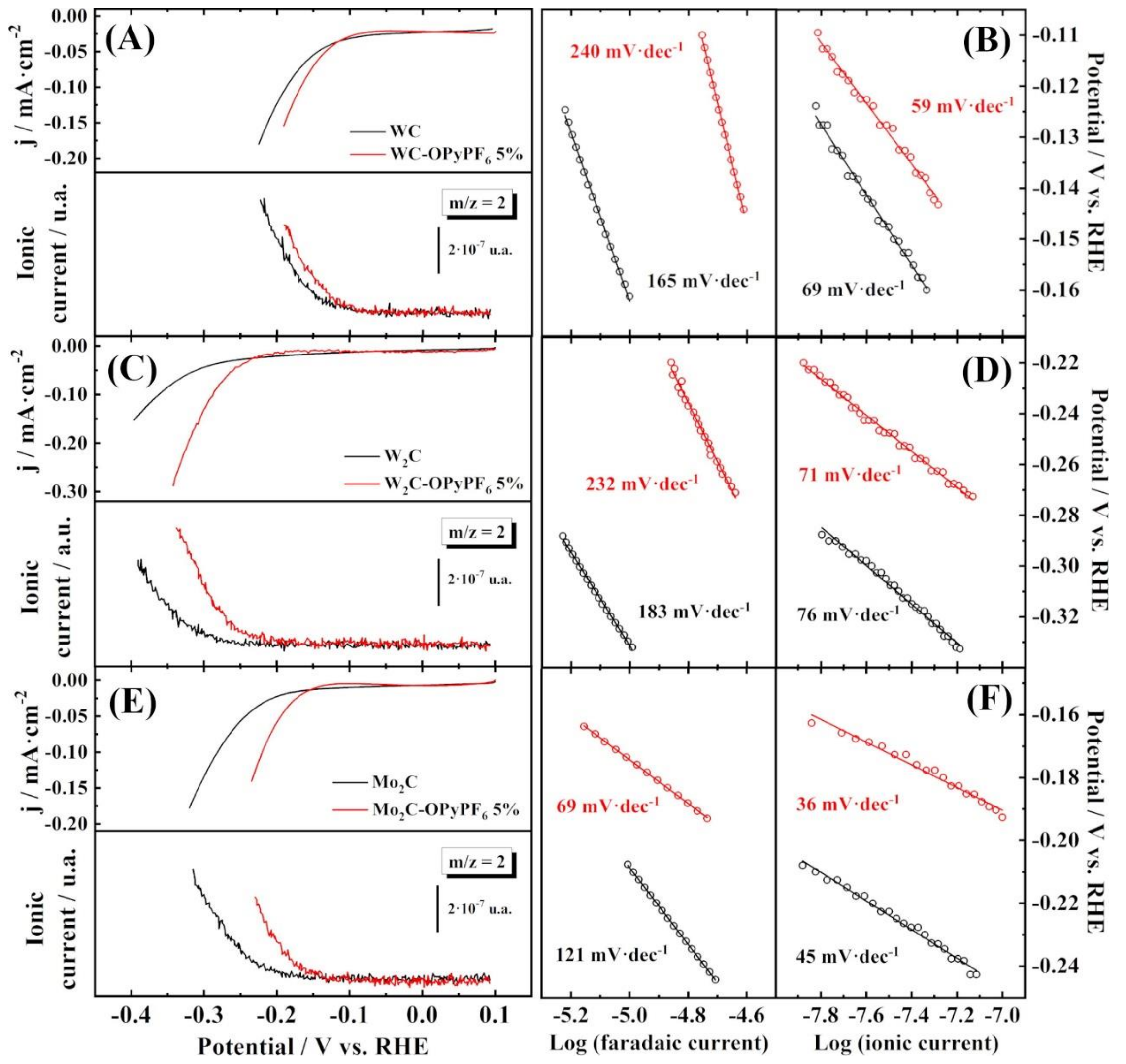
Octylpyridinium hexafluorophosphate



NaOH 0.1 M, 1 mV/s

S. Díaz-Coello et al. *J. Electroanal. Chem.* **2021**, 880, 114878.

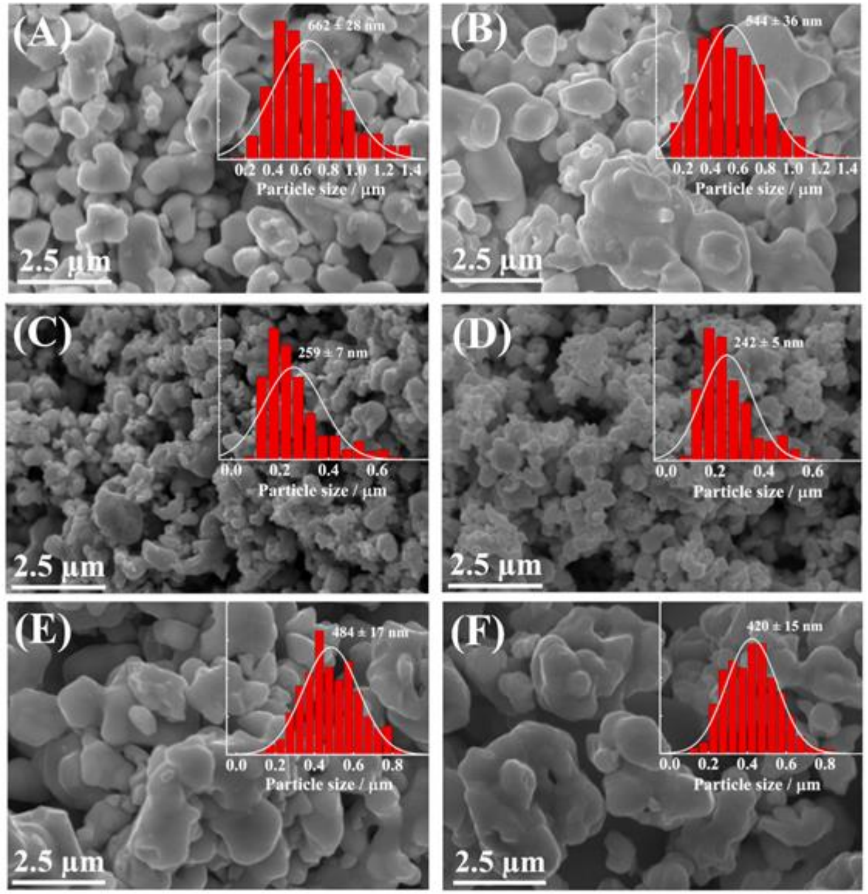
Hydrogen evolution reaction at metal carbides

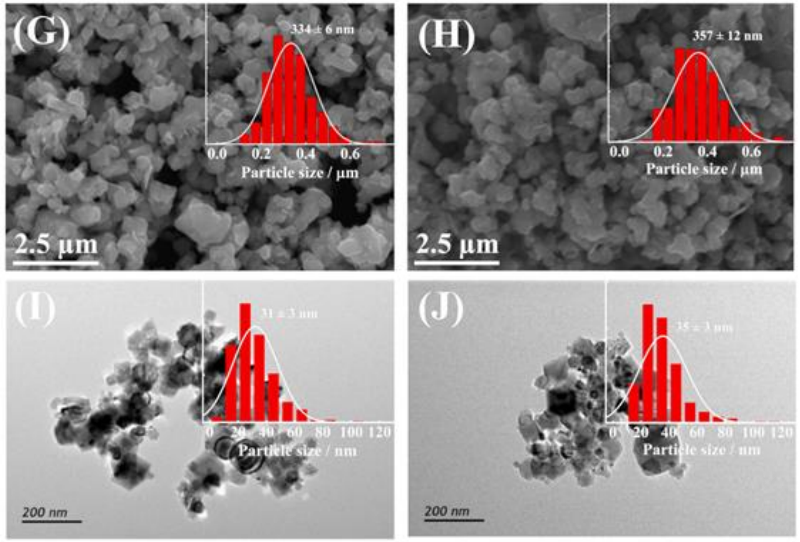


NaOH 0.1 M, 1 mV/s

S. Díaz-Coello et al. *J. Electroanal. Chem.* **2021**, 898, 115620.

SEM images of TMCs and TMCs + IL

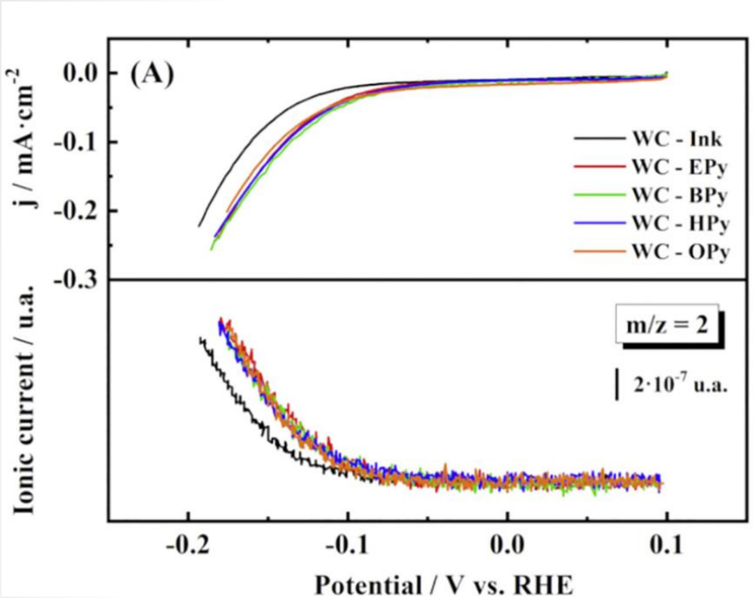


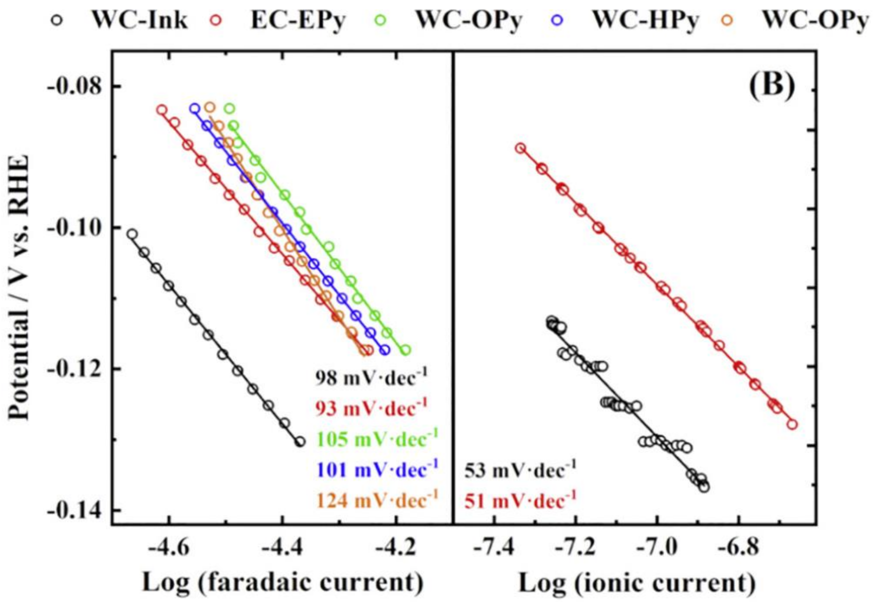


Sample	TMC / nm	TMC-IL / nm	Difference / nm
(A-B) W_2C	662	544	- 35
(C-D) WC	260	242	- 18
(E-F) Mo_2C	484	420	- 64
(G-H) VC	334	357	+ 23
(E-J) TiC	31	35	- 4

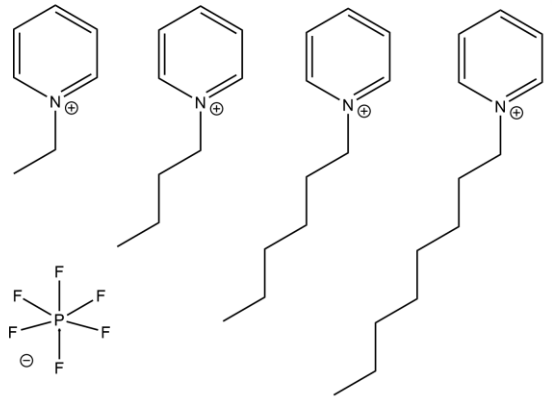
S. Díaz-Coello et al. *J. Electroanal. Chem.* **2021**, 898, 115620.

Hydrogen evolution reaction at metal carbides

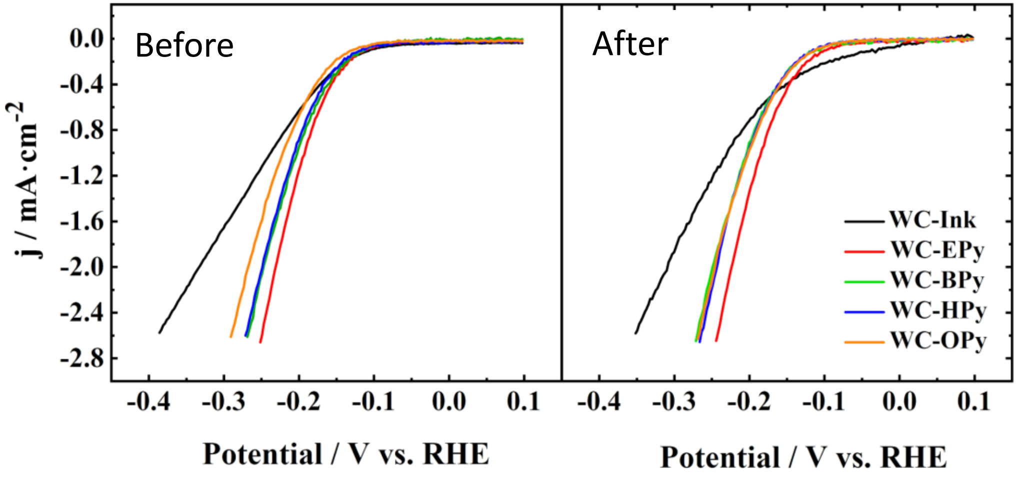




Effect of the length of the chain



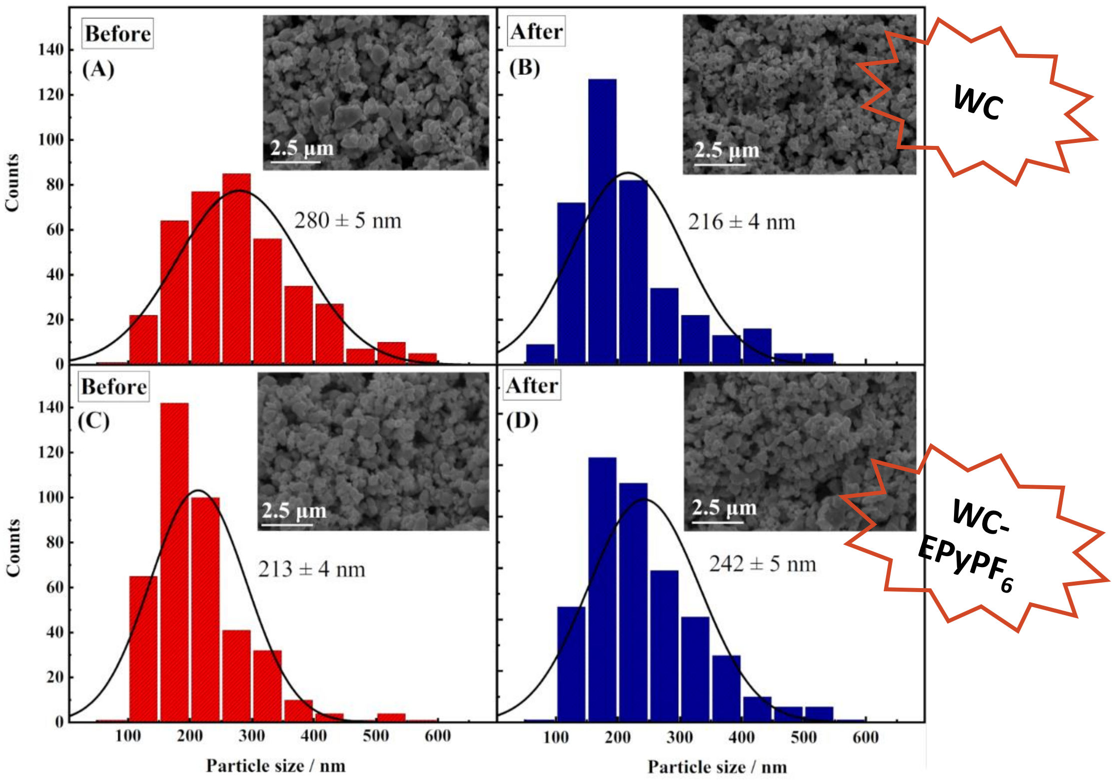
NaOH 0.1 M, 1 mV/s



Durability. *Stress tests*
500 scans, 0.2 V/s

S. Díaz-Coello et al. *J. Electroanal. Chem.* **2021**, 880, 114878.

SEM images TMCs + IL. Stress tests

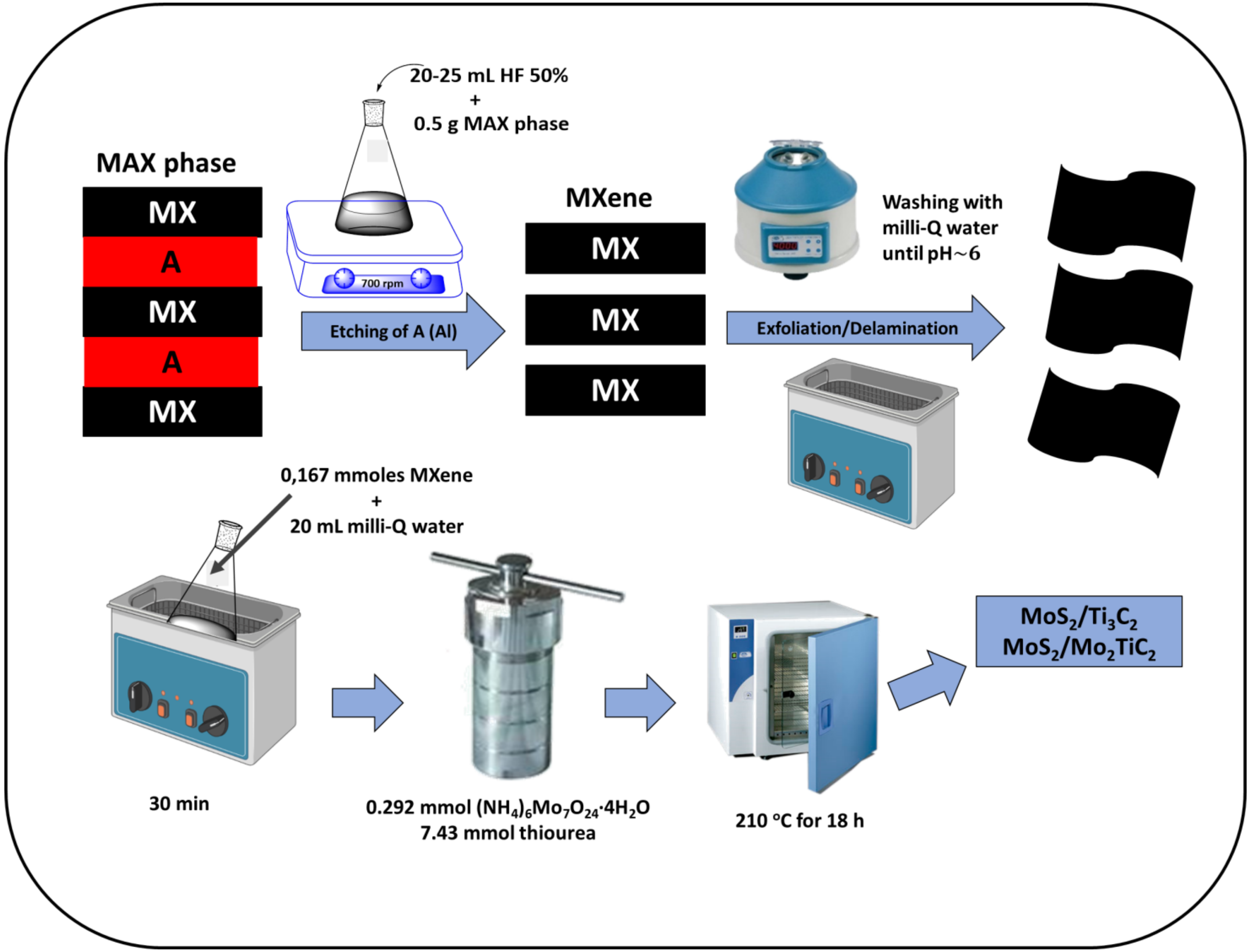


Decrease of 25 %

Increase of 12 %

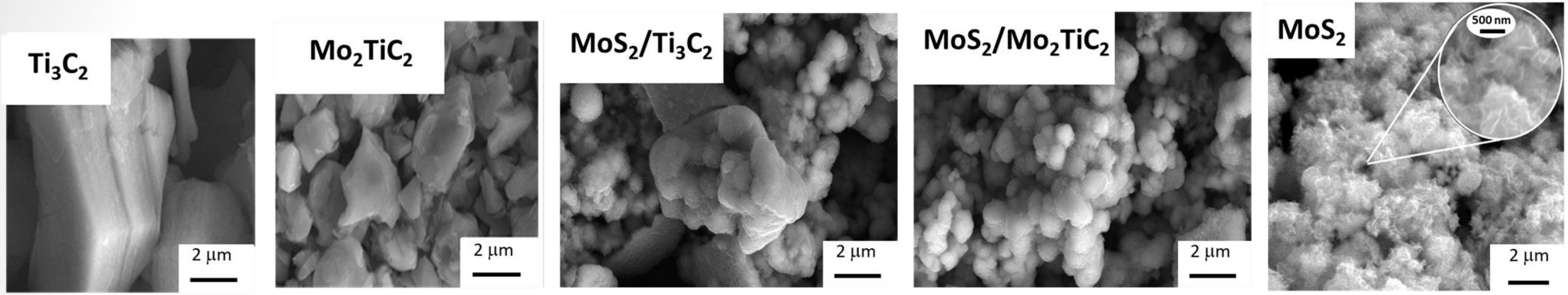
S. Díaz-Coello et al. *J. Electroanal. Chem.* **2021**, 880, 114878.

Synthesis of 2D carbides: MXenes and composites



Y. Remedios-Diaz et al. *ChemElectroChem* **2024**, e202300673

Characterization of MAX phases, MXenes and composites

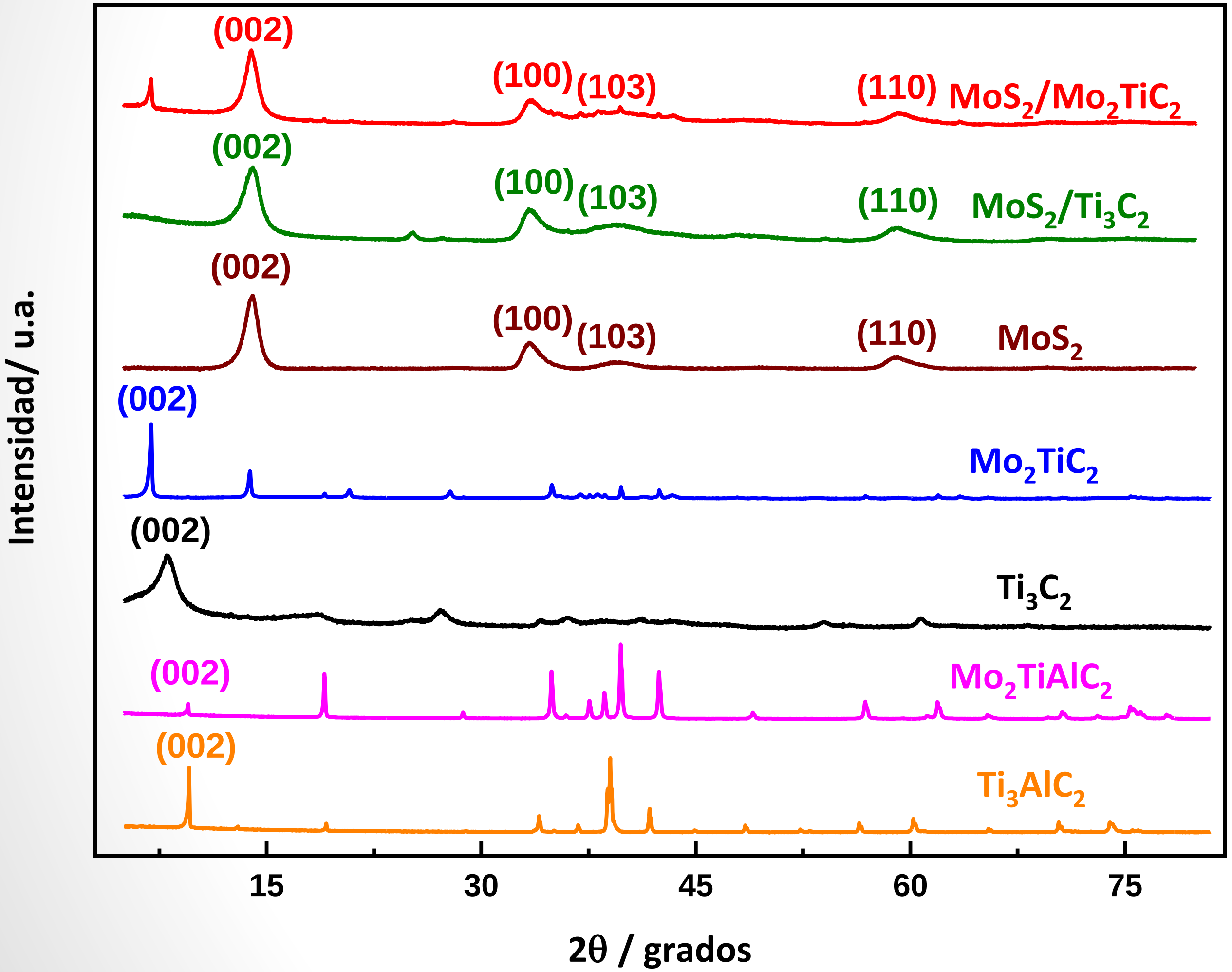


	Composition (at.)							Mo:S ratio
Material	C	O	F	Al	Mo	Ti	S	
Ti_3AlC_2	30.2	-	-	11.8	-	58.0	-	-
Mo_2TiAlC_2	48.4	10.6	-	10.6	19.8	10.6	-	-
Ti_3C_2	22.6	35.5	11.6	0.3	-	30.0	-	-
Mo_2TiC_2	45.6	22.5	3.3	0.6	18.7	9.4	-	-
MoS_2	-	10.2	-	-	29.0	-	60.8	1: 2.1
$MoS_2@Ti_3C_2$	36.2	9.3	-	-	16.4	3.5	34.6	1: 2.1
$MoS_2@Mo_2TiC_2$	37.3	7.4	-	-	18.7	0.8	35.8	1: 1.9

SEM/EDX

Y. Remedios-Diaz et al.
ChemElectroChem
2024, e202300673

Characterization of MAX phases, MXenes and composites



DRX

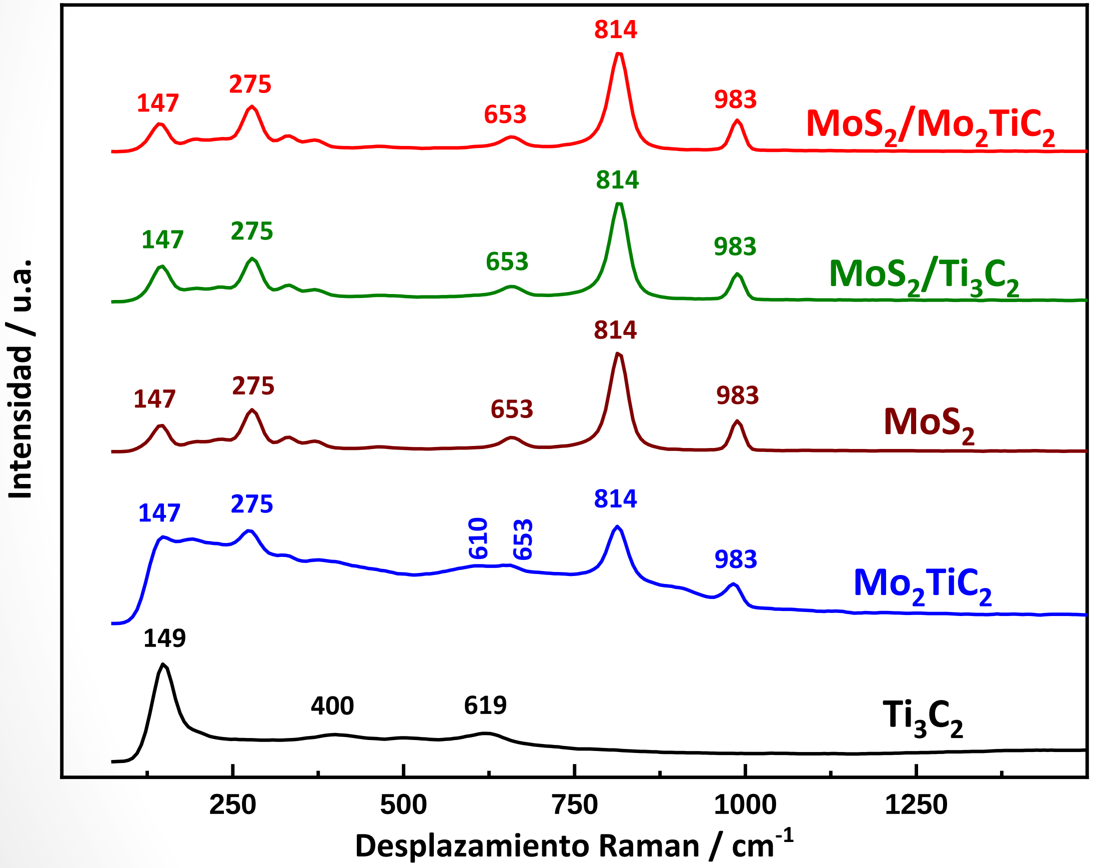
Catalyst	Crystallite size (002) /nm	Interplanar distance /nm
Ti_3AlC_2	79.6	0.92
Mo_2TiAlC_2	71.5	0.93
Ti_3C_2	5.8	1.10
Mo_2TiC_2	44.7	1.28
MoS_2	7.2*	0.63
MoS_2@Ti_3C_2	6.0*	0.63
MoS_2@Mo_2TiC_2	8.0*	0.64

*MoS_2

Y. Remedios-Diaz et al. *ChemElectroChem* 2024, e202300673

Characterization of MAX phases, MXenes and composites

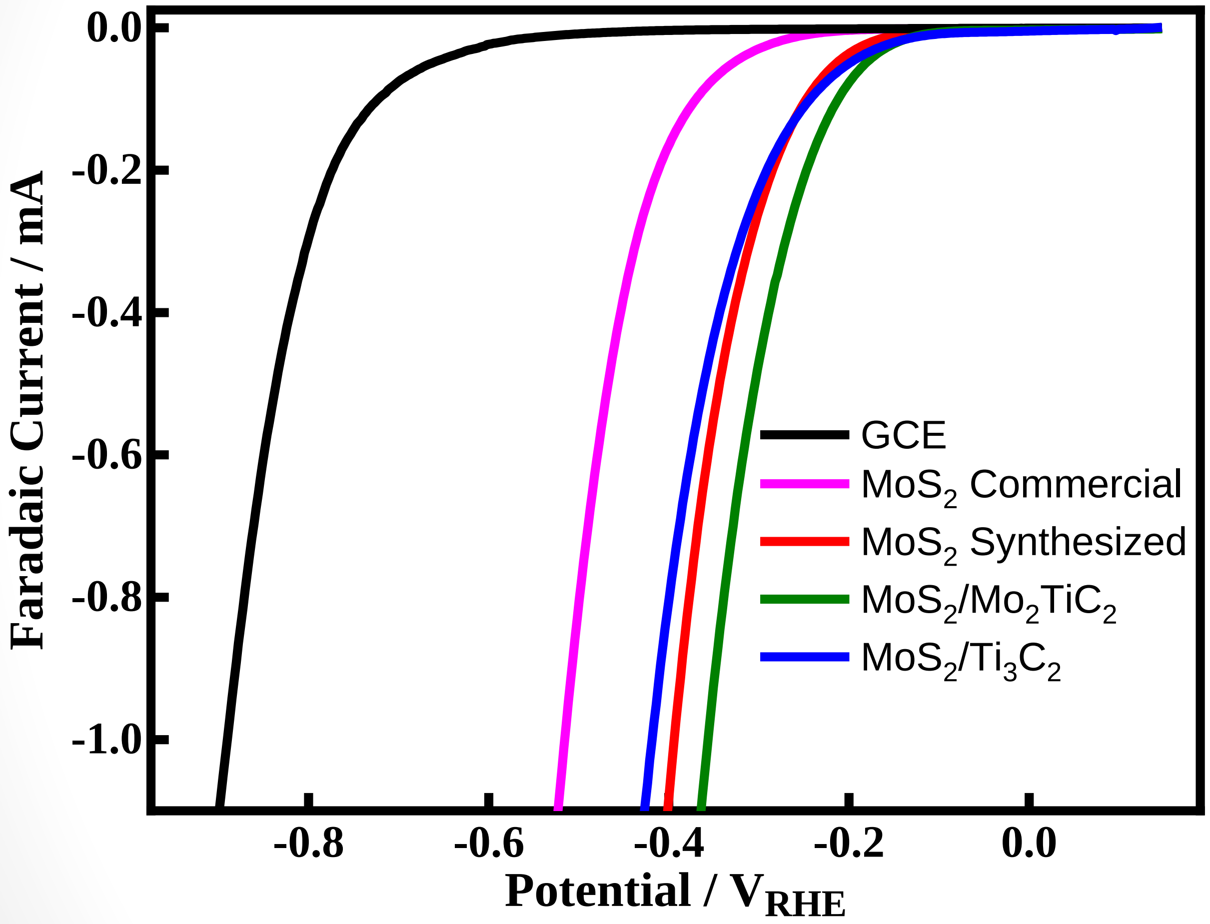
Raman Spectroscopy



Catalyst	Raman shift / cm^{-1}	Assingation	Vibration mode
Ti_3C_2	149 619	O-Ti O-Ti	B_{1g} (TiO_6) A_{1g}(TiO_6)
Mo_2TiC_2	147 275 653 814 983	*Lattice mode* O=Mo O-Mo_3 O-Mo_2 O=Mo	- Bending Stretching Stretching Stretching
MoS_2	147 275 370 400 653 814 983	*Lattice mode* O=Mo MoS_2 MoS_2 O-Mo_3 O-Mo_2 O=Mo	- Bending in-planeE_{2g} out-of-plane A_{1g} Stretching Stretching Stretching
MoS_2@Ti_3C_2	147 275 370 400 653 814 983	*Lattice mode* O=Mo MoS_2 MoS_2 O-Mo_3 O-Mo_2 O=Mo	- Bending in-planeE_{2g} out-of-plane A_{1g} Stretching Stretching Stretching
MoS_2@Mo_2TiC_2	147 275 370 400 653 814 983	*Lattice mode* O=Mo MoS_2 MoS_2 O-Mo_3 O-Mo_2 O=Mo	- Bending in-planeE_{2g} out-of-plane A_{1g} Stretching Stretching Stretching

Y. Remedios-Diaz et al. *ChemElectroChem* 2024, e202300673

HER for MoS_2/MXene composites



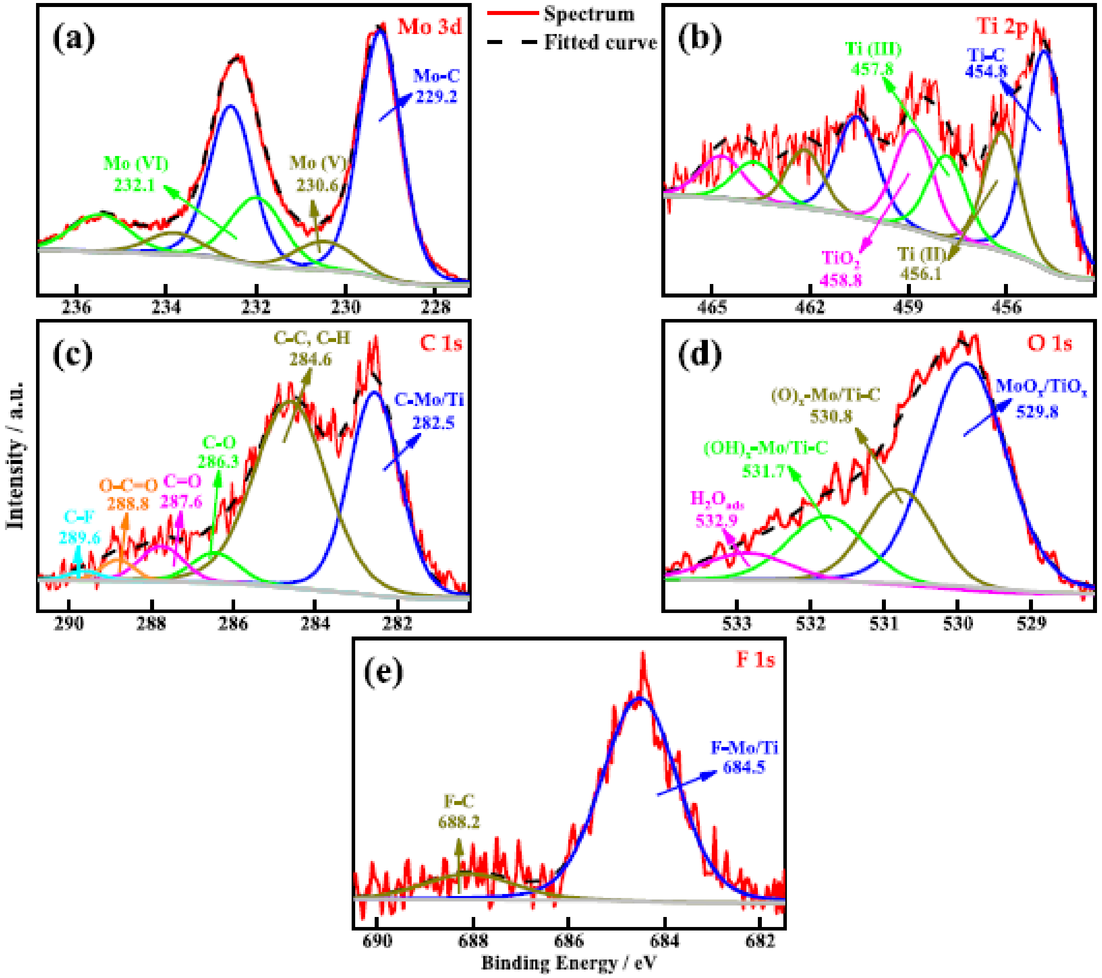
LSV, 0.5 M H_2SO_4, 2 $mV\cdot s^{-1}$

S. Delgado et al. *In preparation*

Characterization of MAX phases, MXenes and composites

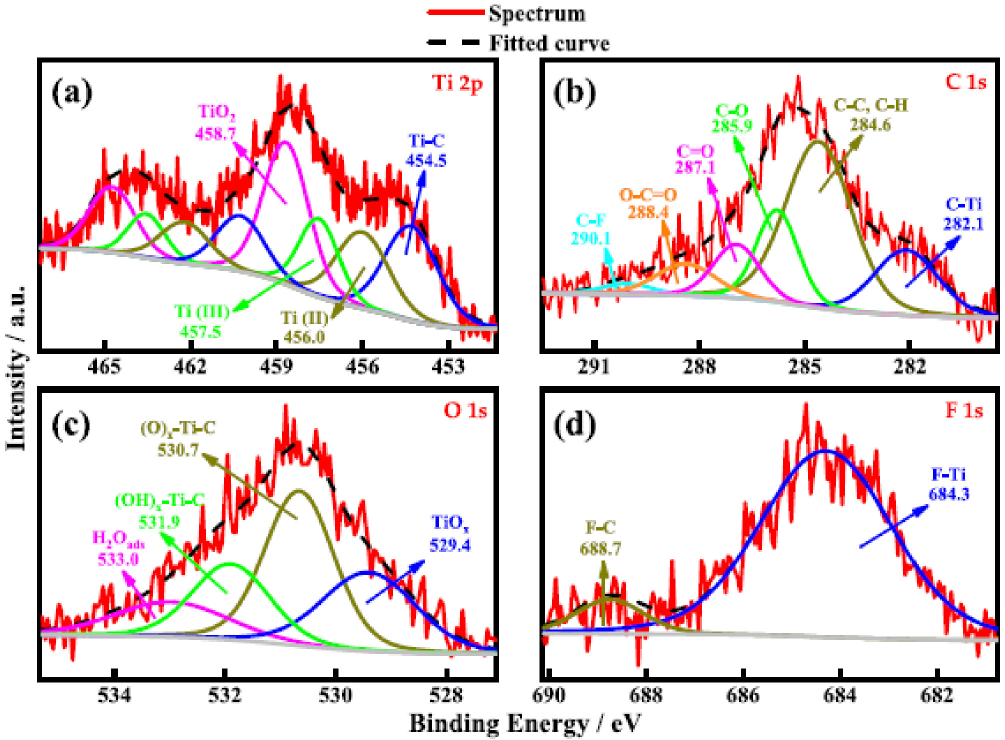
XPS

Mo_2TiC_2



XPS spectra of Mo_2TiC_2: (a) Mo 3d, (b) Ti 2p, (c) C 1s, (d) O 1s and (e) F 1s.

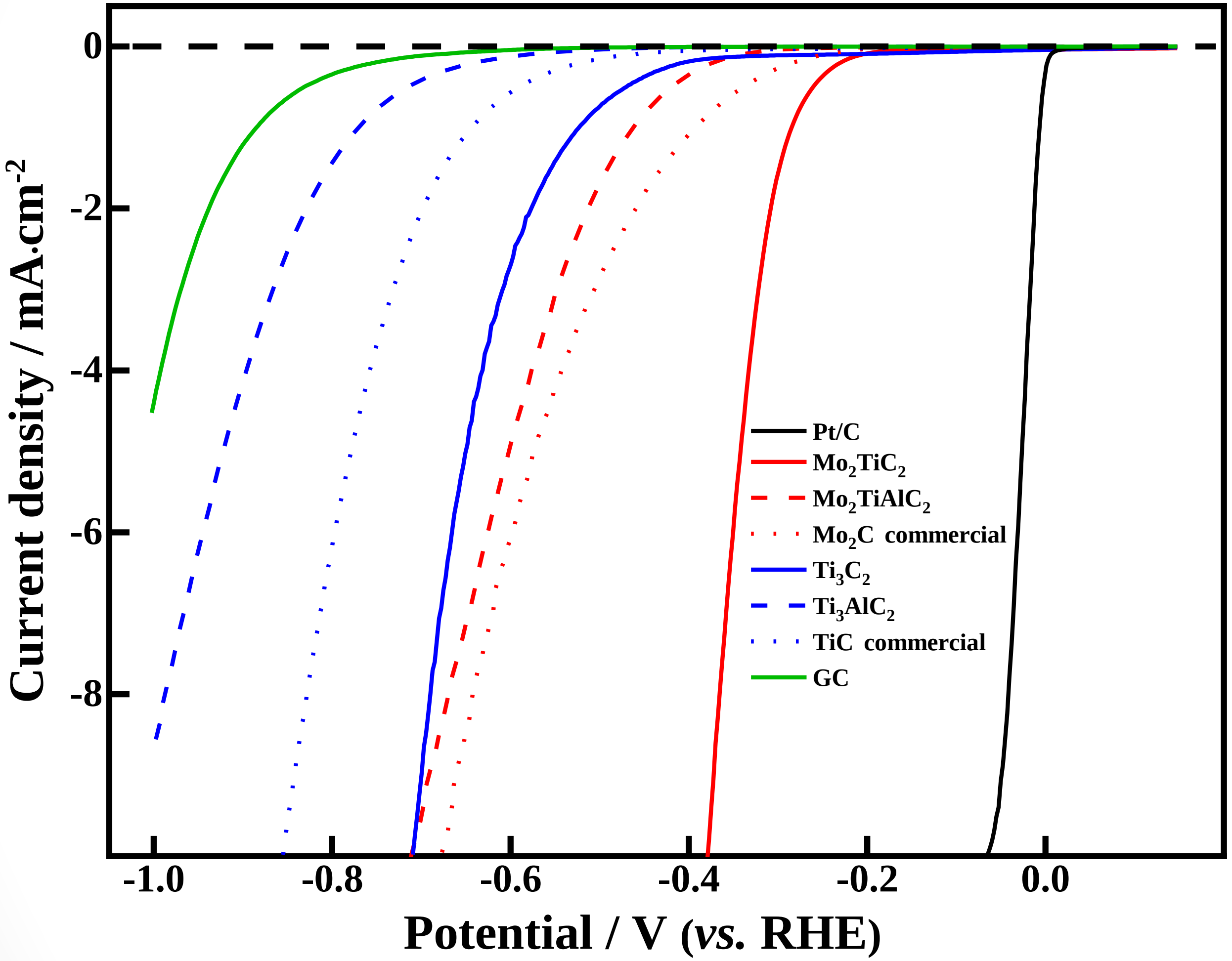
Ti_3C_2



XPS spectra of Ti_3C_2: (a) Ti 2p, (b) C 1s, (c) O 1s and (d) F 1s.

Y. Remedios-Diaz et al. *ChemElectroChem* 2024, e202300673

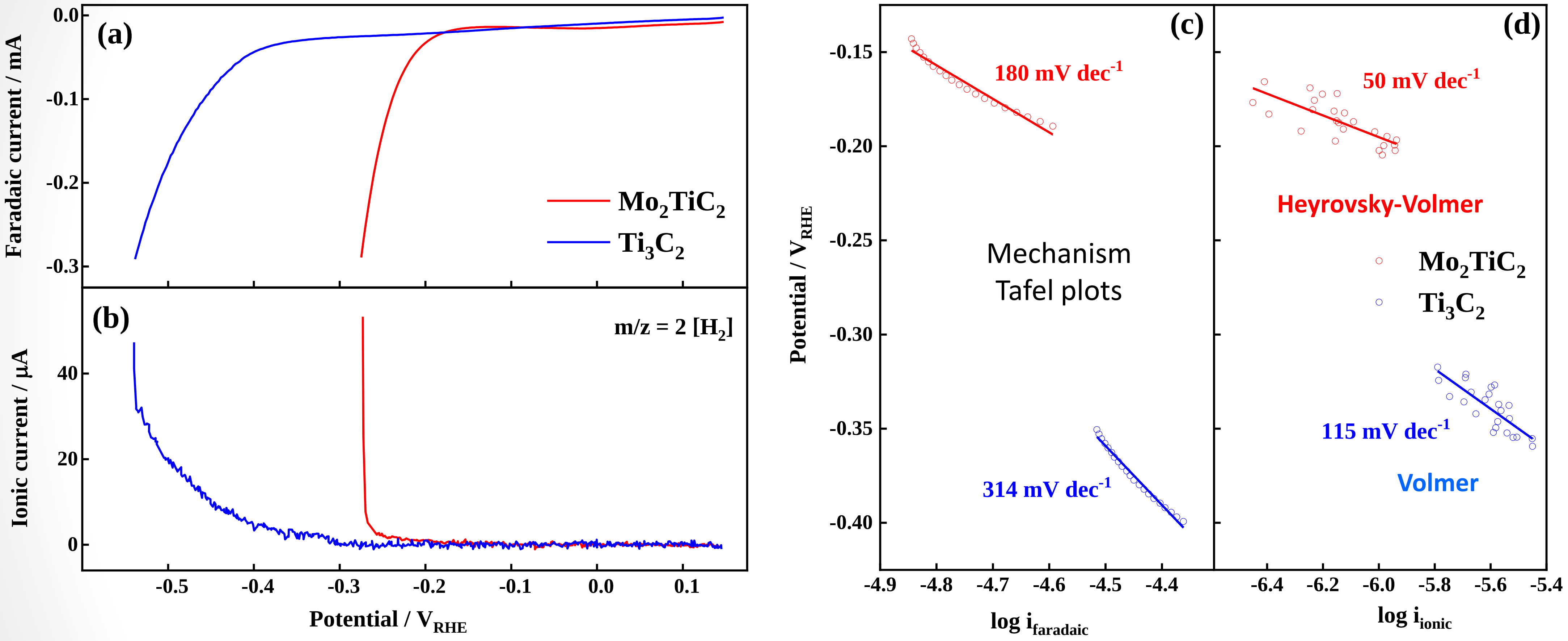
HER for MAX phases and MXenes



LSV, 0.5 M H_2SO_4, 2 mV·s^{-1}

S. Delgado et al. *International Journal of Hydrogen Energy* 55 (2024) 1050

HER studied by DEMS

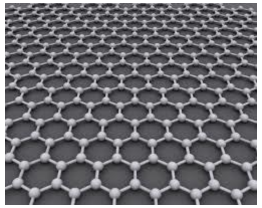


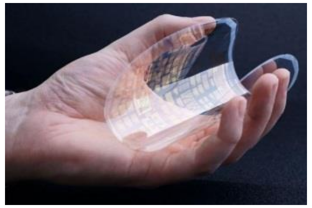
2 mV·s^{-1}, 0.5 M H_2SO_4

S. Delgado et al. *International Journal of Hydrogen Energy* 55 (2024) 1050

2D materials: graphene as catalyst support

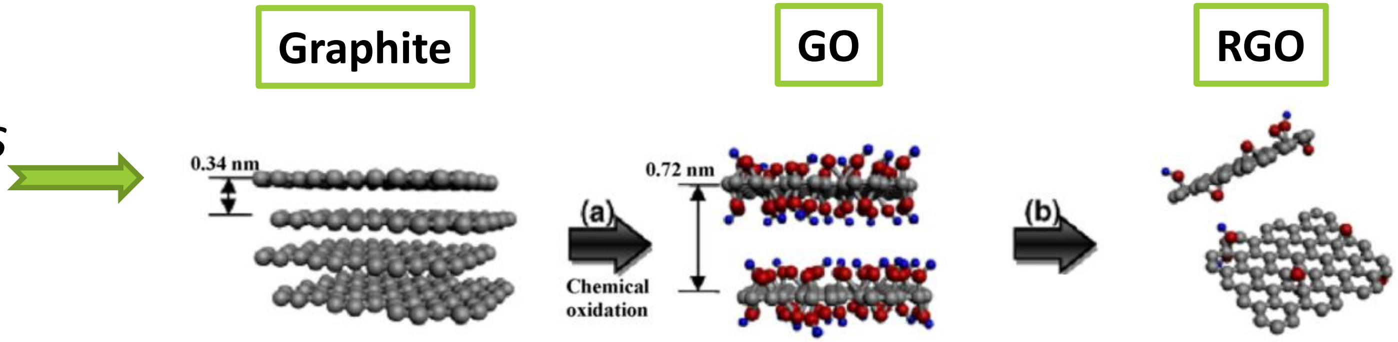
- Graphene is a bidimensional nanometric material obtained by micromechanical exfoliation in 2004.
- It shows excellent mechanical, magnetic and electrical properties.





Synthesis of reduced graphene oxide

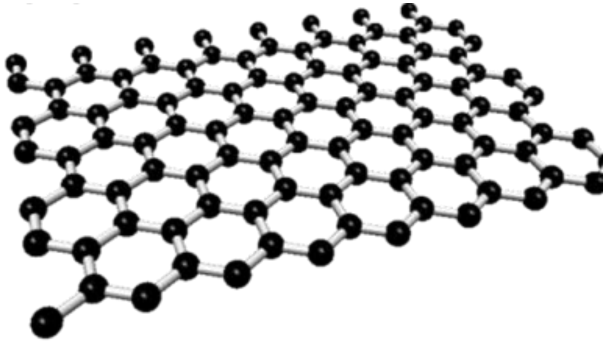
Hummer´s Method



Graphene as catalyst support

Properties of a support for electrochemistry systems

- Stable ✓
- Good electric conductivity ✓
- Suitable surface chemistry (metal/support interaction) ✗
- High surface area (2630 $m^2\ g^{-1}$) ✓



Graphene (G)

- The strong π-π interaction produces a weak interaction with the metals at the surface

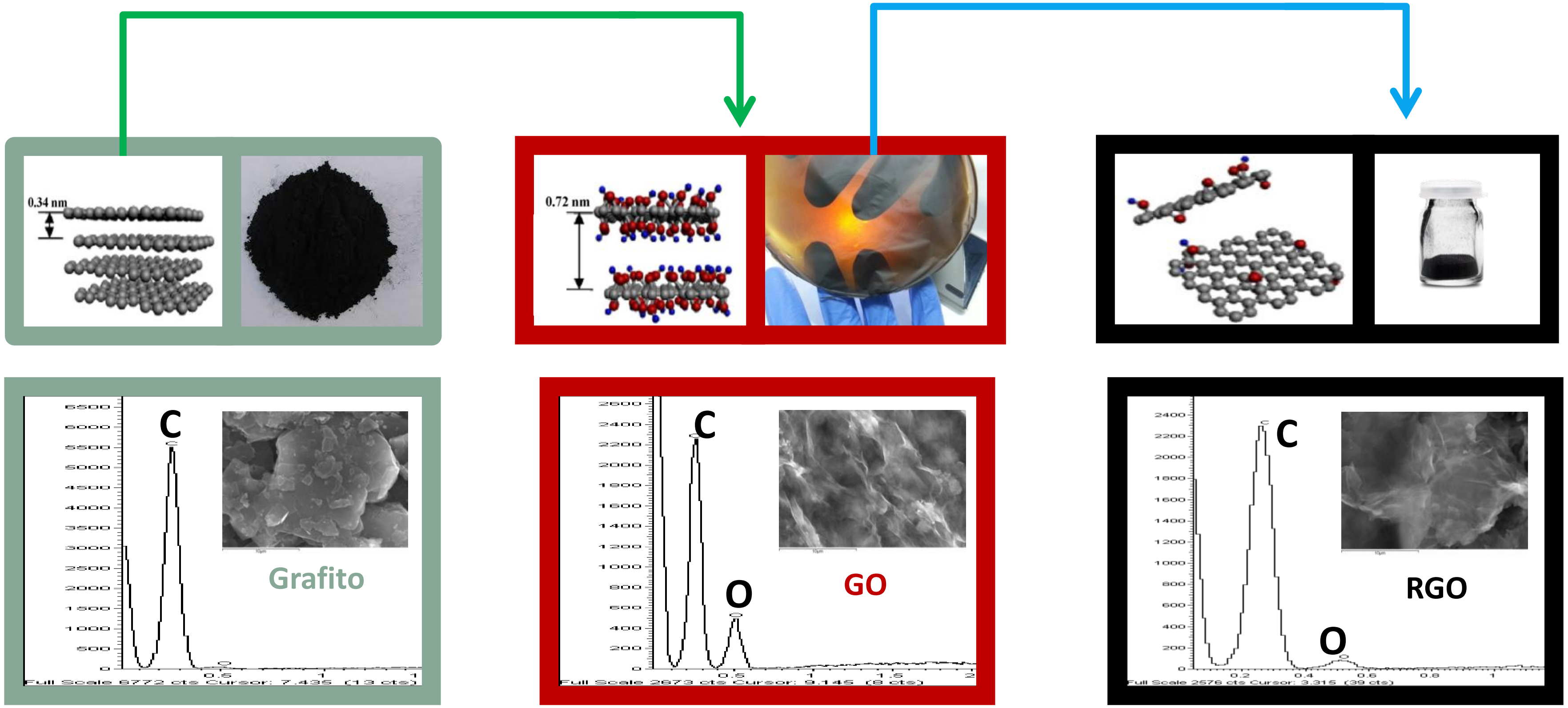
Synthesis of graphenic materials

Synthesis of GO

Synthesis of RGO

$H_2SO_4/KMnO_4$ (Hummers method)

Thermal treatment 450 ºC, N_2/H_2 5%



Reduced graphene oxide as catalyst support

Properties of a support for electrochemistry systems

- Stable ✓
- Good electric conductivity ✓
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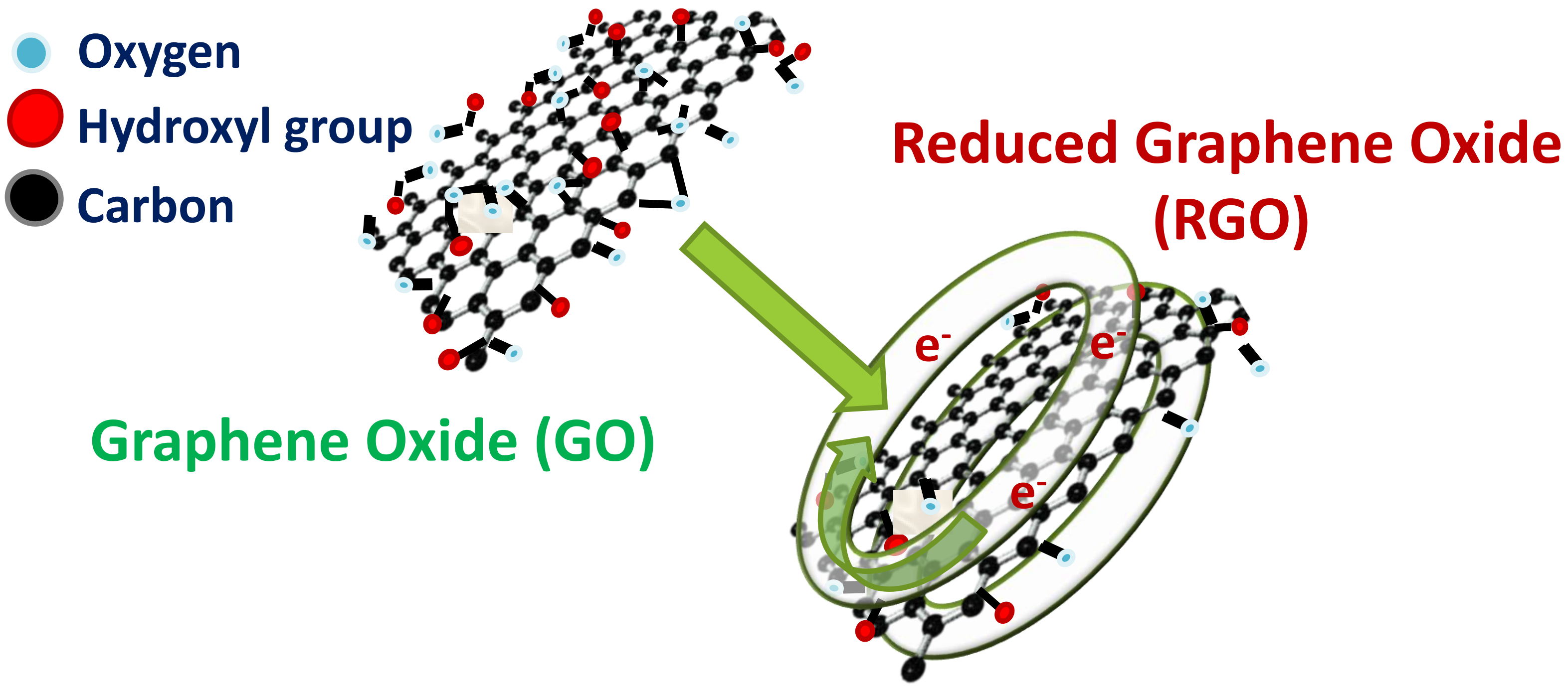


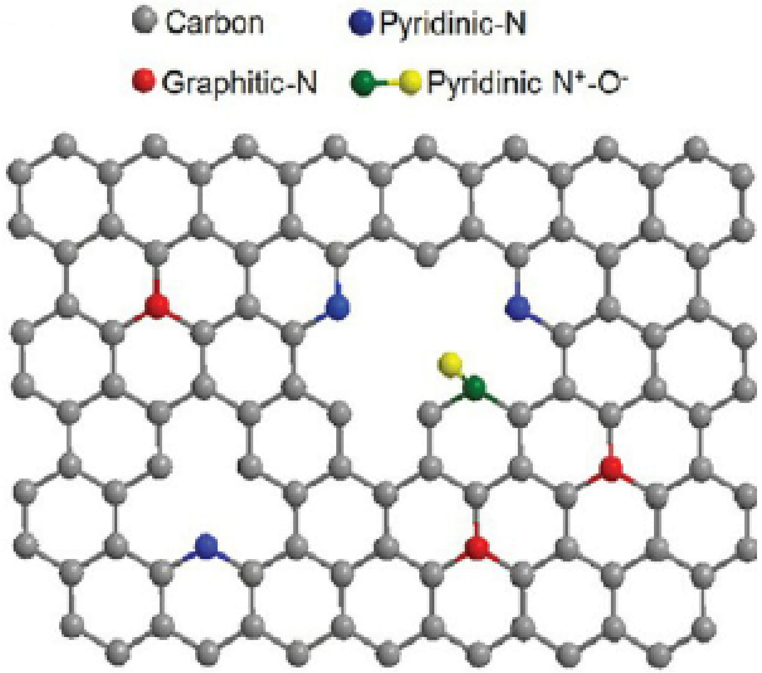
Graphene materials (GMs)

Graphene based electrocatalysts:

- Inexpensive, widely available, high electrical conductivity, stability in acid/alkaline environments

- Doping process modifies electronic structure and tailor surface chemistry (can be used as catalyst)

- Defects could be active sites for electrocatalysis



Synthesis of GMs

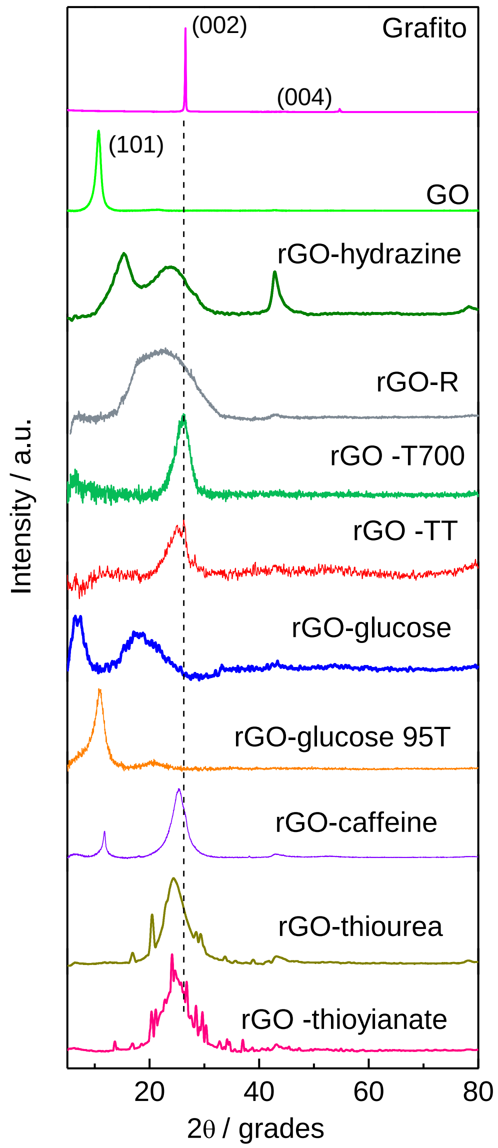
Green reduction agents

- sodium citrate
  - rGO - reflux
  - rGO –T700 reflux
- glucose
  - rGO -G Hidro
  - rGO -G 95T
- thermal treatment synthesis (rGO –TT)

N and/or S doping

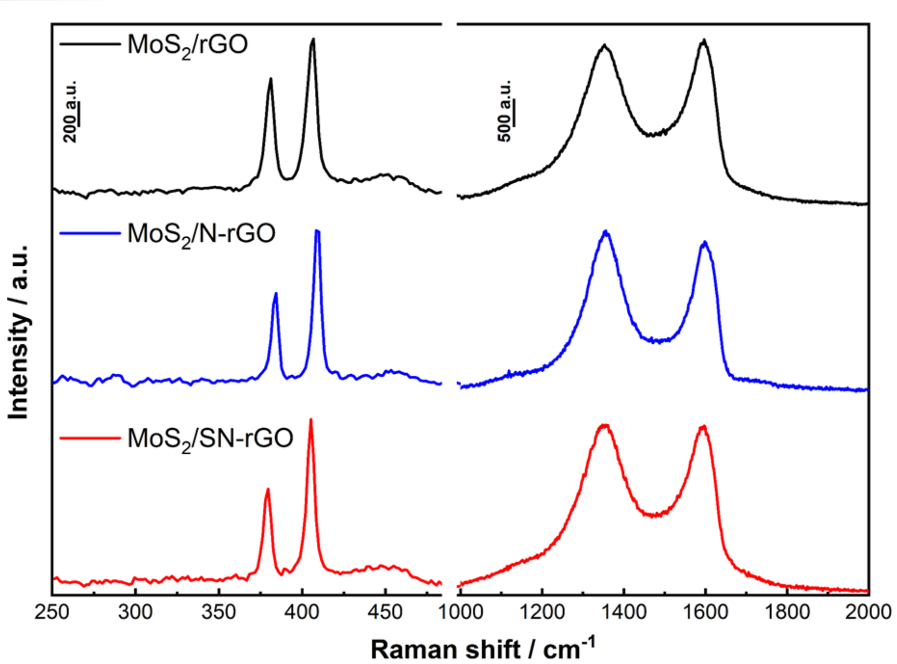
- hydrazine (rGO –hydrazine)
- caffeine (rGO –caffeine)
- dimethyl sulfoxide (S-rGO)
- thiourea (rGO –thiourea)
- ammonium thiocyanate (rGO –thiocyanate)

DRX



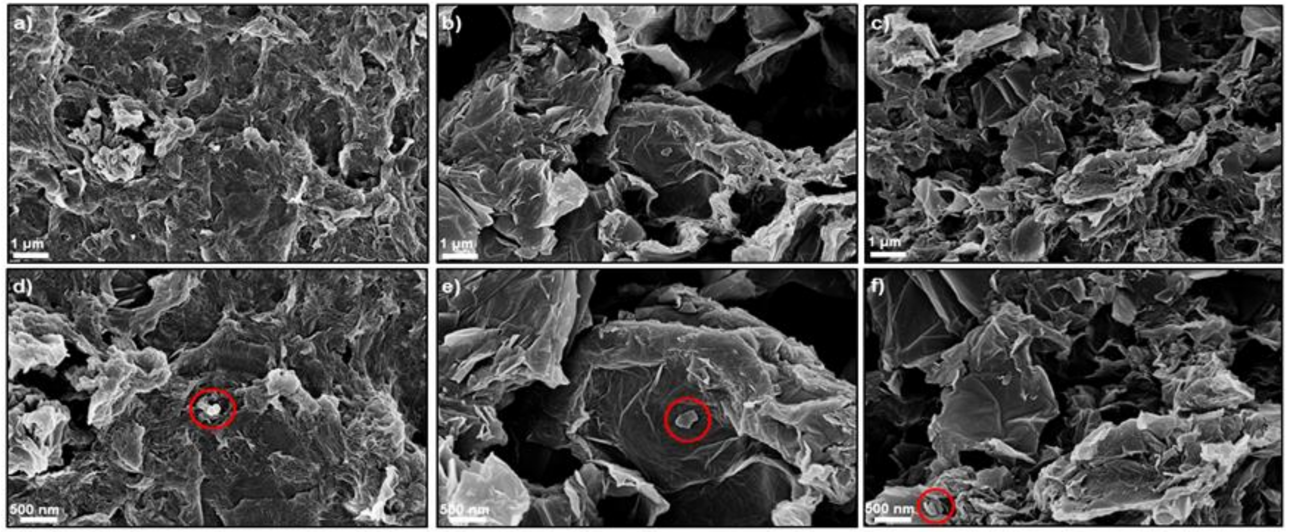
Physicochemical caracterización of GMs and Mo_2S (1 %) composites

Raman Spectroscopy



SEM

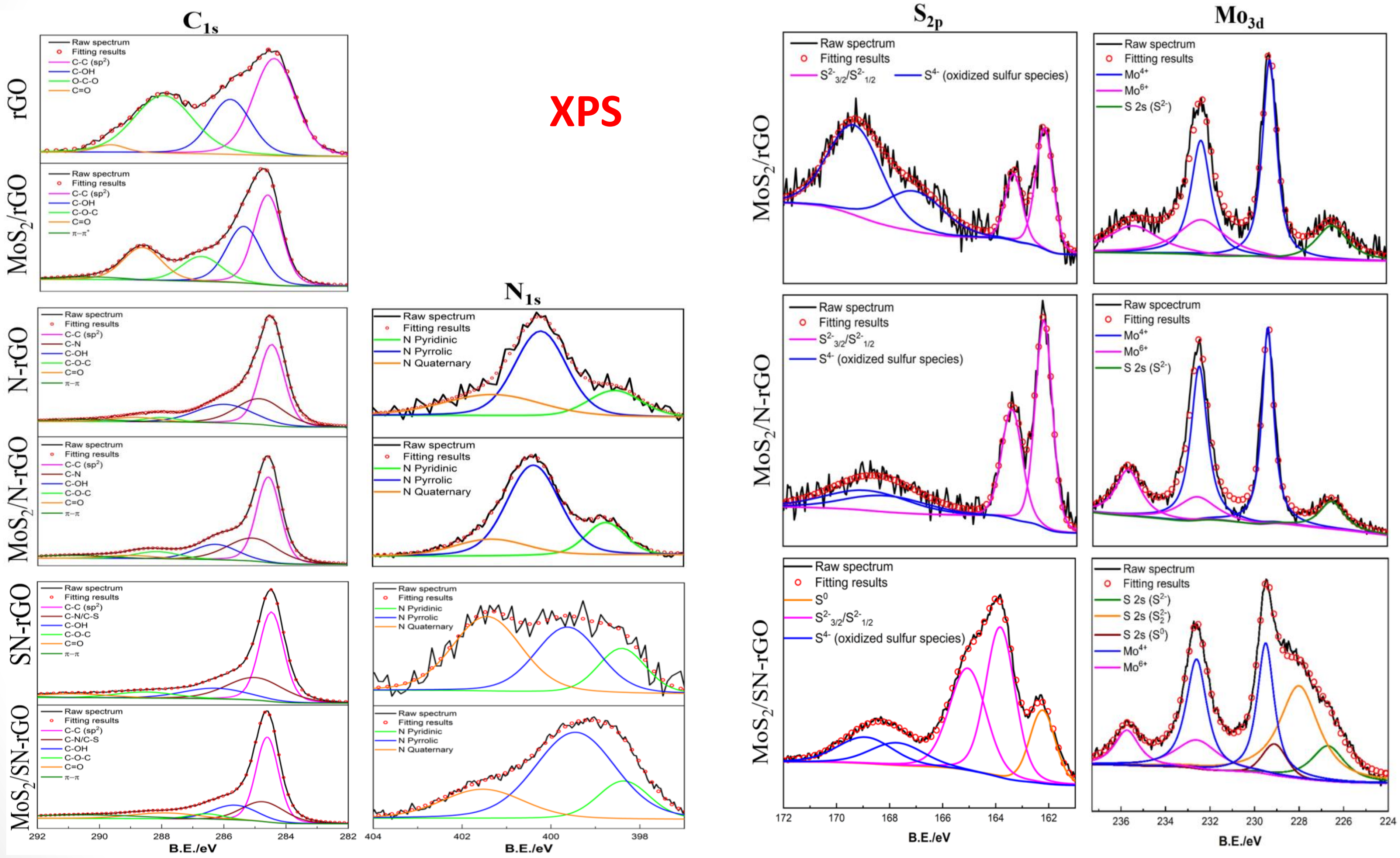
MoS_2/rGO MoS_2/N-rGO MoS_2/SN-rGO



F. Fioravanti et al. *Electrochimica Acta* 441 (2023) 141781

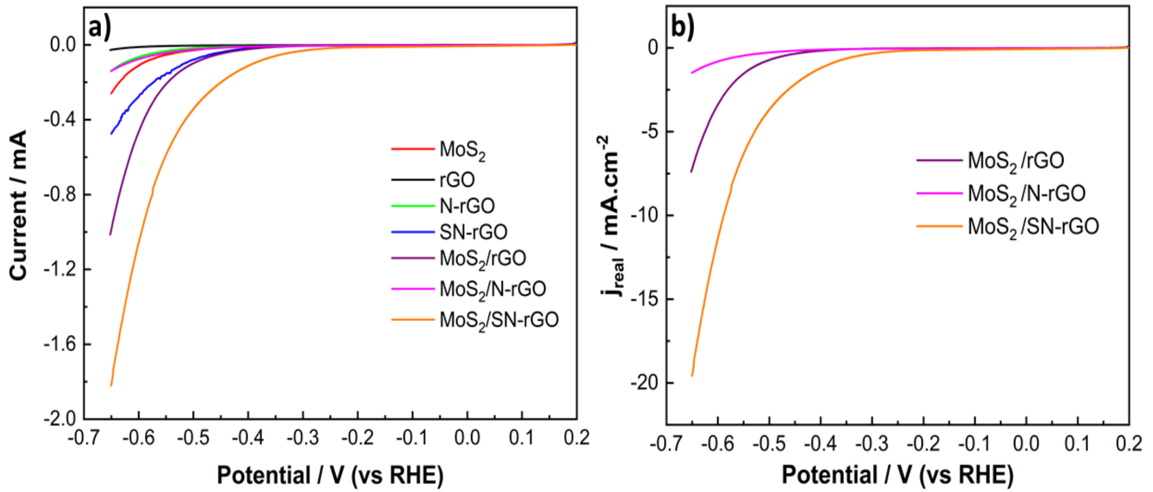
Physicochemical caracterización of GMs and Mo_2S (1 %) composites

XPS

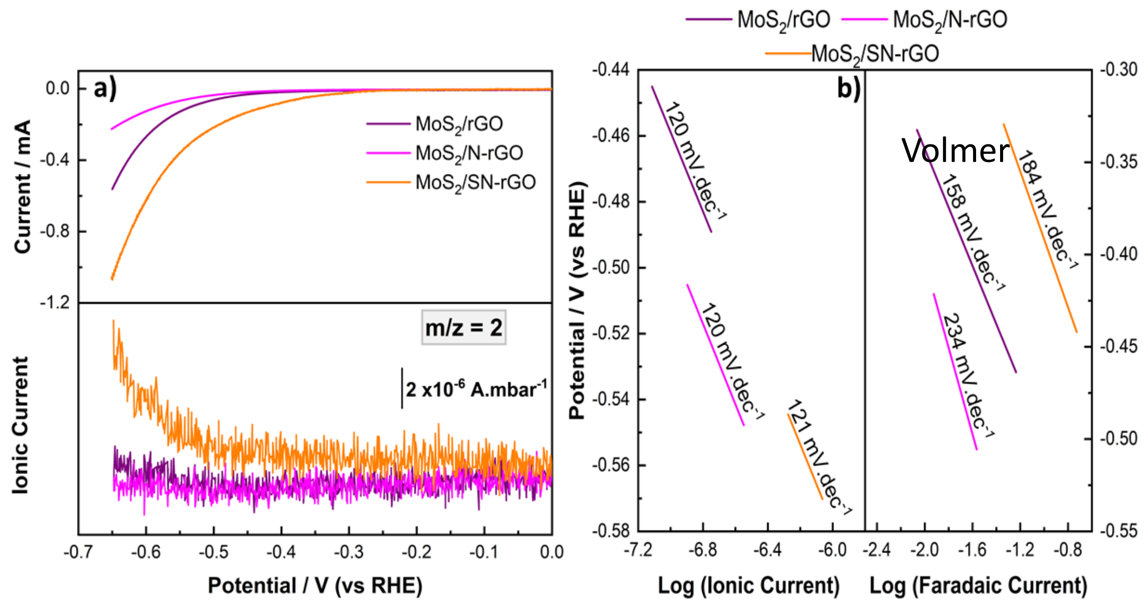


F. Fioravanti et al. *Electrochimica Acta* 441 (2023) 141781

HER for GMs and Mo_2S (1 %) composites



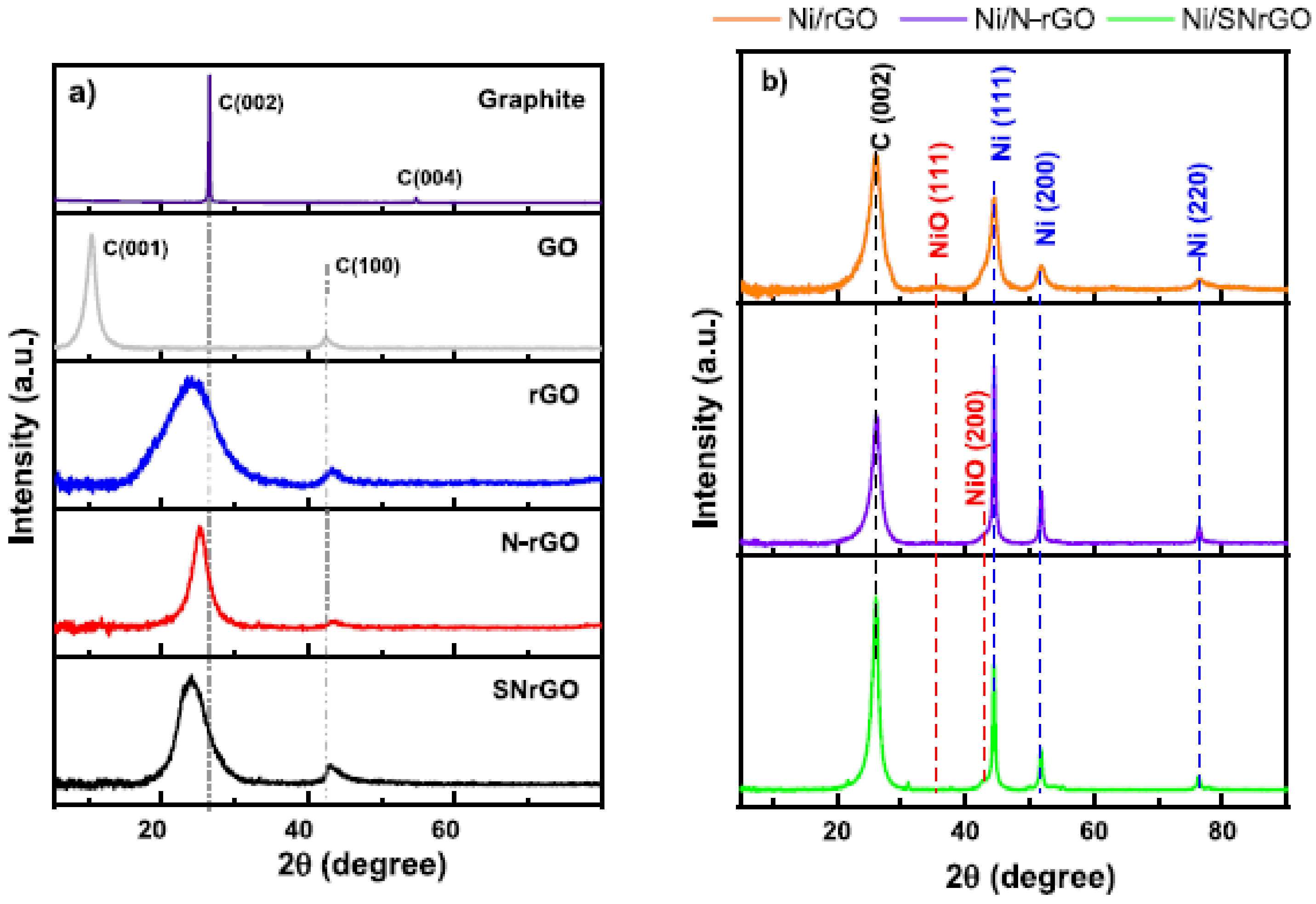
LSV, 0.5 M H_2SO_4, 2 $mV{\cdot}s^{-1}$



F. Fioravanti et al. *Electrochimica Acta* 441 (2023) 141781

Physicochemical characterization of Ni NPs/GMs

DRX



XRD patterns for a) supporting materials and b) Ni-based catalysts.

S.J. Martínez et al. *Inorganica Chimica Acta* 566 (2024) 122008

Physicochemical characterization of Ni NPs/GMs

XPS

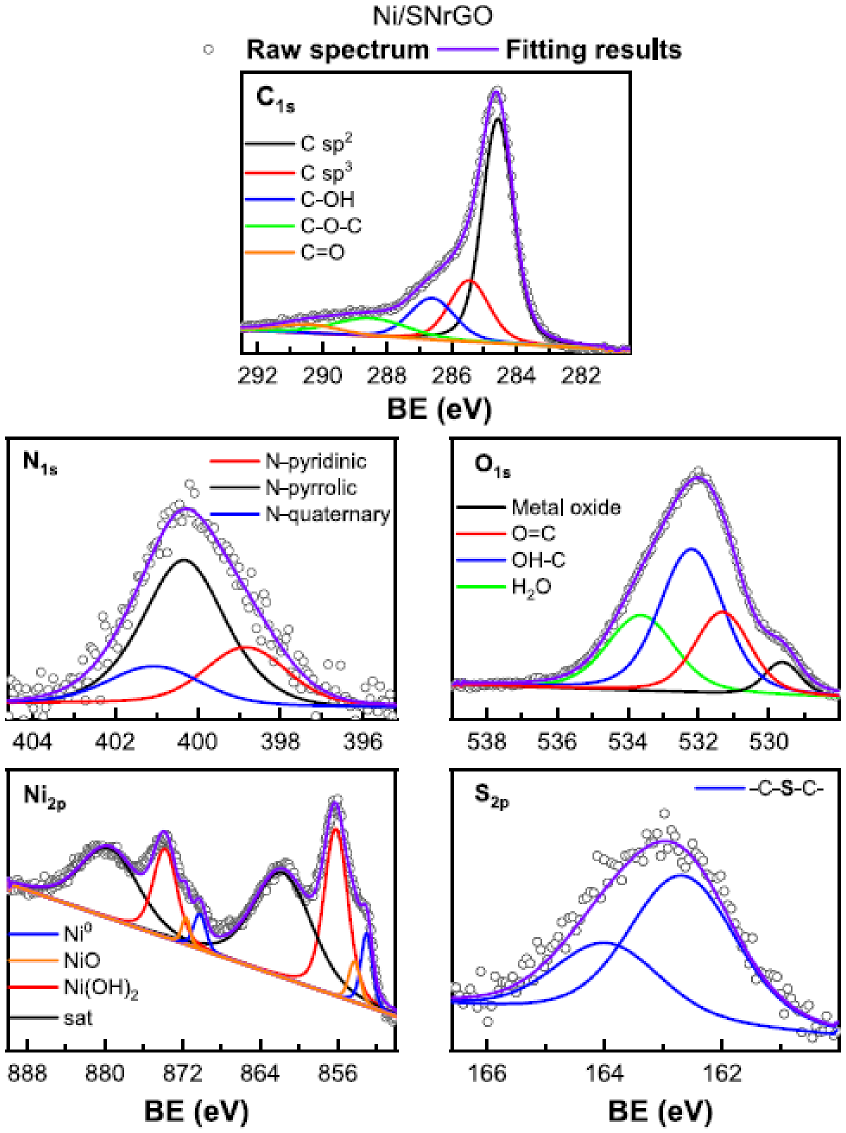


Fig. 3. XPS spectra of Ni/SNrGO.

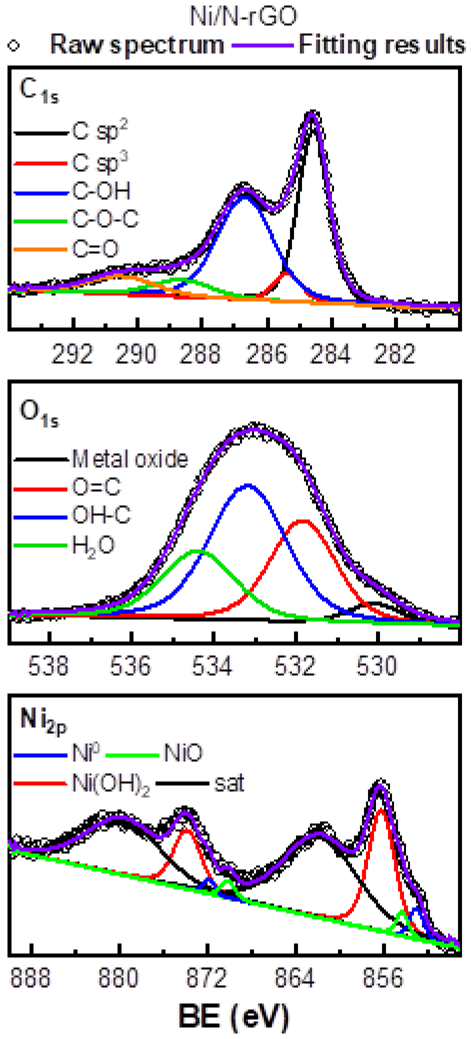
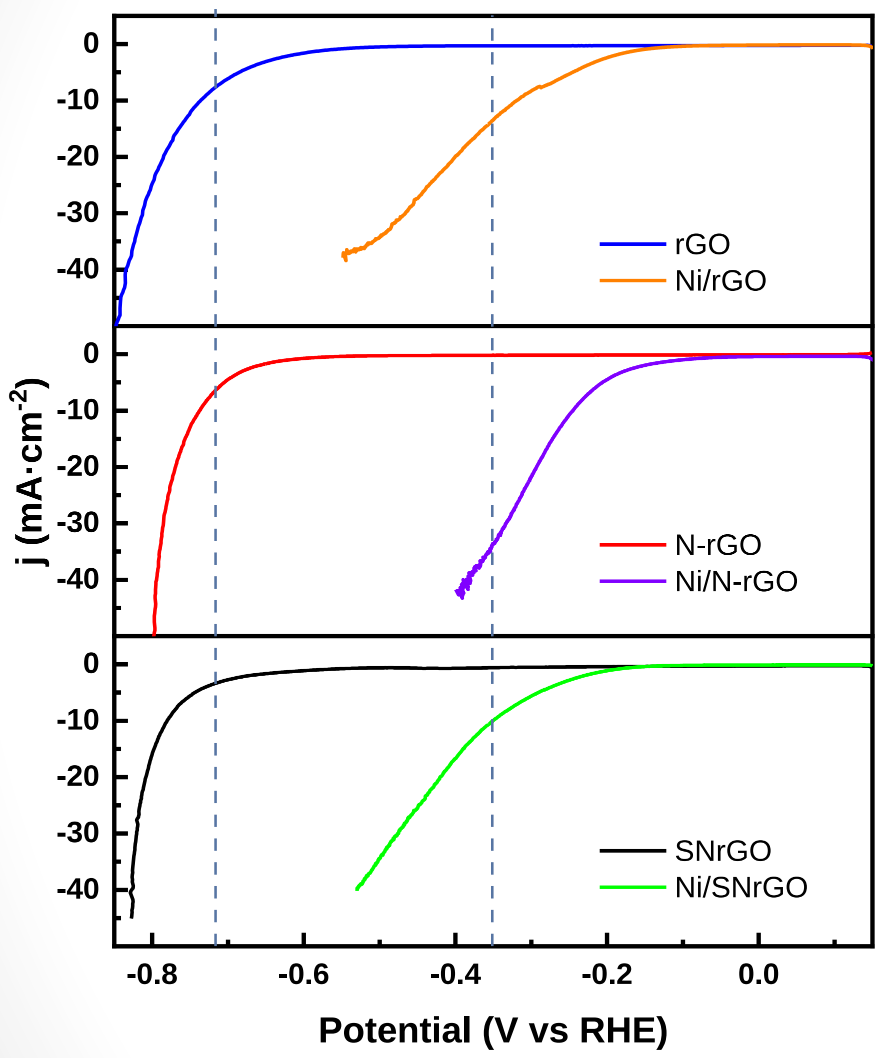
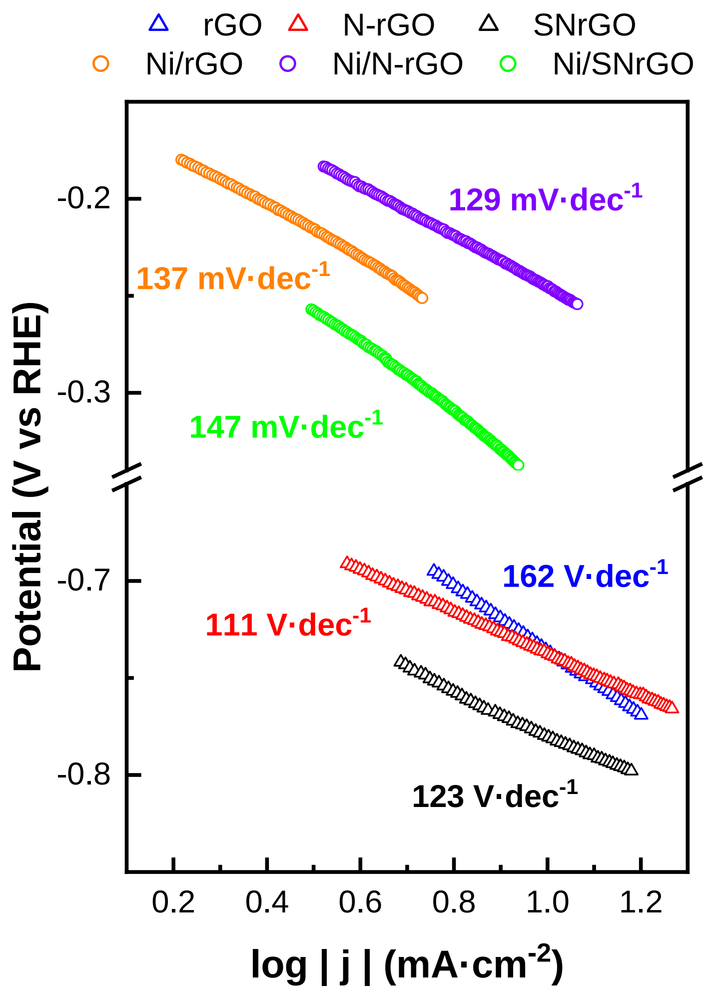


Figure S7. XPS spectra for Ni/N-rGO

S.J. Martínez et al. *Inorganica Chimica Acta* 566 (2024) 122008

HER reaction for Ni NPs/GMs

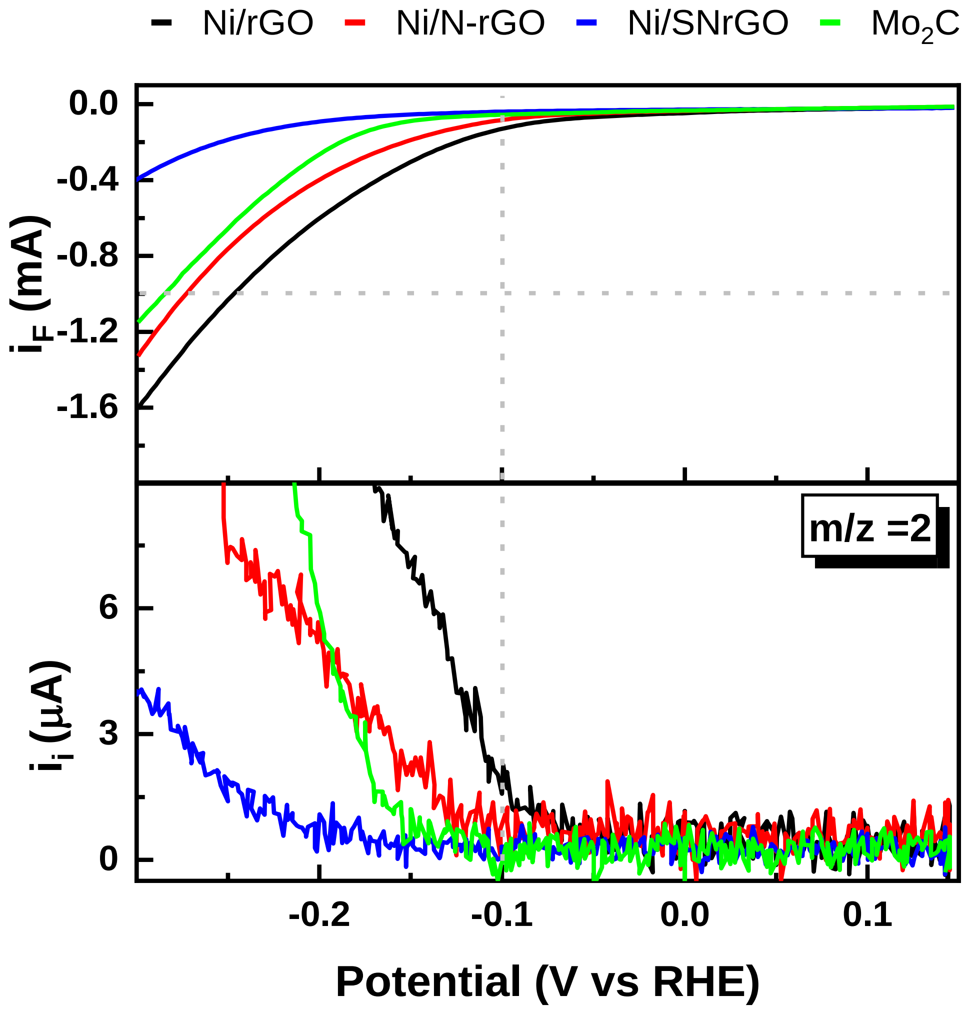




LSV, 2 mV·s^{-1}, 1 M NaOH

Stephanie J. Martínez et al. *Inorganica Chimica Acta* 566 (2024) 122008

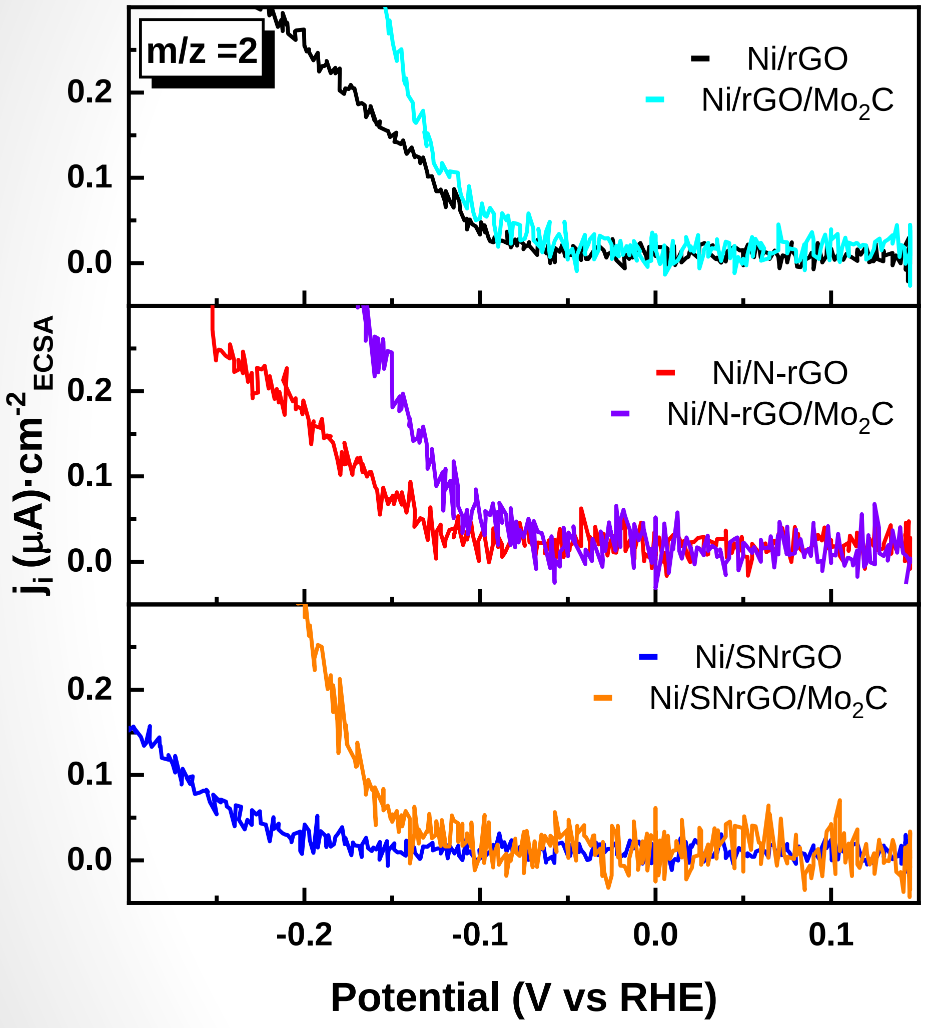
HER reaction for Ni NPs/GMs



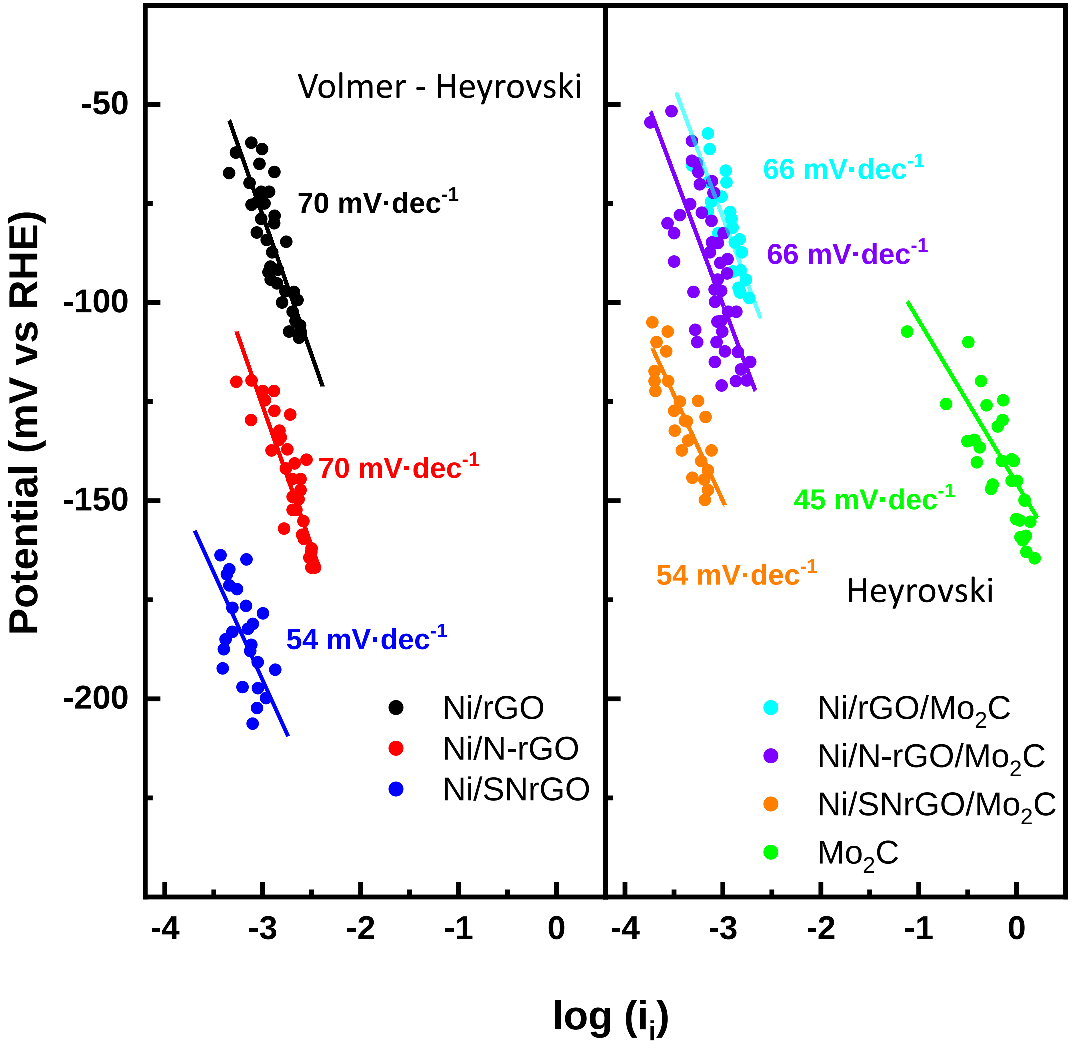
LSV, 2 mV·s^{-1}, 1 M NaOH

Stephanie J. Martínez et al. *Electrochim. Acta*, submitted

HER reaction for Ni NPs/GMs/Mo_2C



LSV, 1 M NaOH, 2 mV·s^{-1}



Stephanie J. Martínez et al. *Electrochim. Acta*, submitted

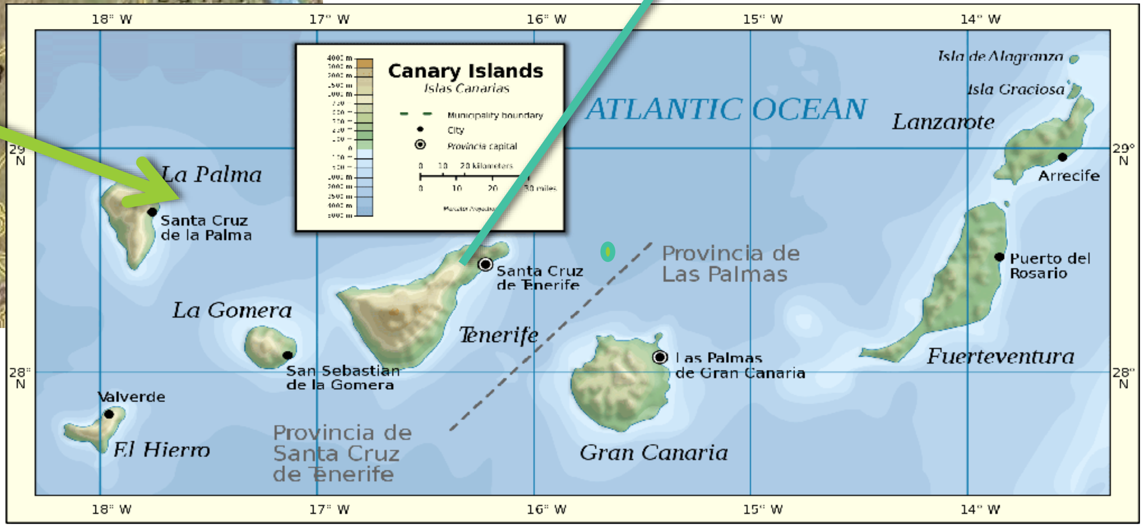
Conclusions

- MC_x are suitable to be used as catalysts for the HER. Optimization of the synthesis can enhance the performance.
- Surface structure, nature of the metal and the electrolyte appear as main responsible for their electrochemical behavior.
- Ionic liquids seems to modified the activity towards the HER. This effect is strongly related with the modification of particle sizes after interaction with the IL.
- MXenes and dichalcogenides show good activity towards the HER.
- Doping of graphene materials allows to modulate the response of the catalysts towards the HER when used as supports (or co-catalysts).
- Design of 2D composites materials seems to be a good opportunity for increasing the activity of catalyst towards the HER.
- DEMS appears as an important tool for the elucidation of the mechanism of the HER.

Where are we?

2nd LATAM MEETING ON GREEN AMMONIA AND POWER-to-X

University of La Laguna



Life in Tenerife!!









The ULL is located in north-eastern Tenerife, in the city of San Cristobal de La Laguna, declared by the UNESCO as World Heritage site in December, 1999.

Surface Science and Electrocatalysis Group

Grupo de Ciencia de Superficies y Electrocatálisis

Prof. Elena Pastor Tejera

Prof. Carmen Arévalo Morales

Prof. José Luis Rodríguez Marrero

Dra. Ana L. López Machado

Dr. Sergio Díaz Coello

Prof. Juan Carlos Calderón Gómez

Prof. Gonzalo García Silvestro

Surface Science and Electrocatalysis Group

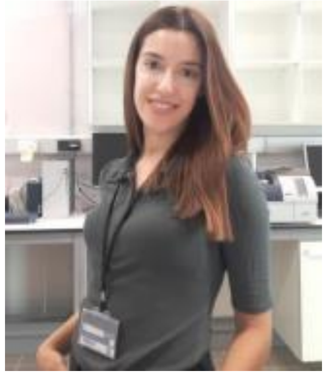
Grupo de Ciencia de Superficies y Electrocatálisis

Sthephanie Martínez

Sergio Fajardo Rodríguez

Yapci Remedios Díaz

Antonella Loiacono



Jennifer Rodríguez Díaz

Abraham Castilla Silvestre

Stefan Delgado Barreto

Canarias avanza con Europa

Gobierno de Canarias

Unión Europea

Fondo Europeo de Desarrollo Regional

Instituto de Materiales y Nanotecnología
Universidad de La Laguna

Laboratorio electroquímico de tecnologías del hidrógeno y almacenamiento energético (LABH2)

Elena Pastor
Grupo de Ciencia de Superficies y Electrocatálisis

2nd LATAM MEETING ON GREEN AMMONIA AND POWER-to-X

Acknowledgements

Federico Fiovaranti, Esteban Francheschini, Gabriela Lacconi (UNC, Argentina)

ProID2021010098

Gobierno de Canarias
Consejería de Economía, Conocimiento y Empleo
Agencia Canaria de Investigación, Innovación y Sociedad de la Información

Vicerrectorado de Investigación y Transferencia
Universidad de La Laguna

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AGENCIA ESTATAL DE INVESTIGACIÓN

ÁREAS TEMÁTICAS

- Electroquímica de materiales
- Bioelectroquímica
- Conversión y almacenamiento de energía electroquímica
- Tecnología e ingeniería de los procesos electroquímicos
- Electroquímica fundamental
- Electroquímica molecular
- Electroanálisis

FECHAS CLAVE

- 10 de febrero 2025: apertura del envío de comunicaciones y la web
- 30 de abril 2025: límite para el envío de comunicaciones
- 25 de mayo 2025: límite para la realización de inscripciones anticipadas
- 25 de junio 2025: cierre de inscripciones
- 15-18 de julio 2025: 45 GERSEQ

El Área de Química Física del Departamento de Química de la ULL organiza la XLV Reunión del Grupo Especializado de Electroquímica de la RSEQ del 15 al 18 de julio de 2025 en el Campus de Guajara. Les esperamos en la ULL.

https://gerseq45.webs.ull.es

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María Escudero (ICN2)

INSCRIPCIÓN

	"Early" (hasta el 25 de mayo)	"Late" (desde el 25 de mayo)
Miembros RSEQ, GE-RSEQ	400 €	450 €
No miembros RSEQ, GE-RSEQ	480 €	530 €
Estudiantes (Miembros RSEQ, GE-RSEQ	250 €	300 €
Estudiantes (No miembros RSEQ, GE-RSEQ)	330 €	380 €
Acompañantes	200 €	200 €
Estudiantes del Máster y Doctorado en "Electroquímica. Ciencia y Tecnología"	100 €	100 €

grupo especializado electroquímica

RSEQ Real Sociedad Española de Química
El Sitio de la Química en España


2nd LATAM MEETING ON GREEN AMMONIA AND POWER-to-X
¡¡¡Thank you for your attention!!!