



1<sup>ST</sup> LATAM ON GREEN AMMONIA  
POWER-to-X MEETING

# New Ideas for Energy Generation and Storage

**Gonzalo García Silvestro**

Bioinorganic and Spectroelectrochemistry

Materials Invisible To Oxygen - MITO

Ciencias de Superficies y Electrocatalisis

Deputy Director of the Institute of Nanotechnology and Materials

**Faculty of Science, University of La Laguna, Tenerife, Spain.**



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**Institute of Nanotechnology and Materials**

- + **1.- Electroquímica y corrosión**
- + **2.- Energías renovables y óptica**
- + **3.- Espectroscopía laser y altas presiones**
- + **4.- Nanoscopías, superficies y electroquímica molecular**
- + **5.- Estructura electrónica y simulación de materiales y nanomateriales**
- + **6.- Materiales cerámicos y pilas de combustible de óxidos sólidos**
- + **7.- Ciencia de superficies y electrocatalisis**
- + **8.- Crecimiento, caracterización de materiales dieléctricos (CCDD)**
- + **9.- Espectroscopía óptica y molecular**
- + **10.- Materiales magnéticos**
- + **11.- Nanomateriales y espectroscopía**
- + **12.- Ingeniería de materiales**
- + **13.- Laboratorios de materiales para análisis químicos**
- + **14.- Bioinorgánica y Espectroelectroquímica**

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+ 14.- Bioinorgánica y Espectroelectroquímica

## Research lines

- Electrocatalysis
- Design and synthesis of catalysts for generation and storage of energy.
- Fuel Cells
- Electrolyzers
- Design and development of spectroelectrochemical techniques.





# Ciencias de Superficies y Electrocatalisis



- + 1.- Electroquímica y corrosión
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- + 3.- Espectroscopía laser y altas presiones
- + 4.- Nanoscopías, superficies y electroquímica molecular
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- + 14.- Bioinorgánica y Electroquímica

## Bioinorganic & Spectroelectrochemistry “BIOS”

### Research Lines

- Multidisciplinary and Fundamental Research
- Biomimetic
- Molecular Electrochemistry
- Spectroelectrochemistry
- Model Systems





# Bioinorganic and Spectroelectrochemistry





# Bioinorganic and Spectroelectrochemistry

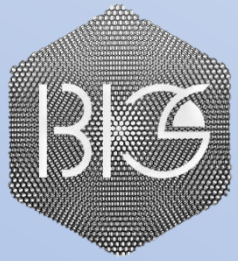
Nature uses electrochemistry in the presence and absence of light to control the redox cycle of compounds crucial for life, such as carbon, oxygen and nitrogen.

The BIOS research group studies how to control the rates of such redox reactions, combining theory, electrode design and the development of spectroelectrochemical techniques.

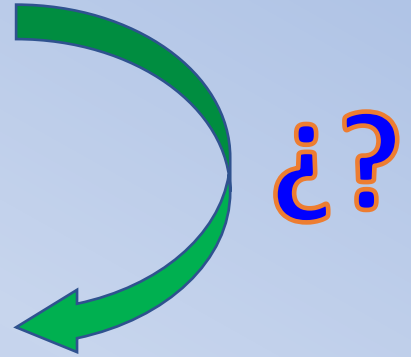


# Materials Invisible To Oxygen - MITO



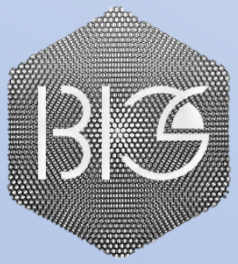


**Clean Energy**

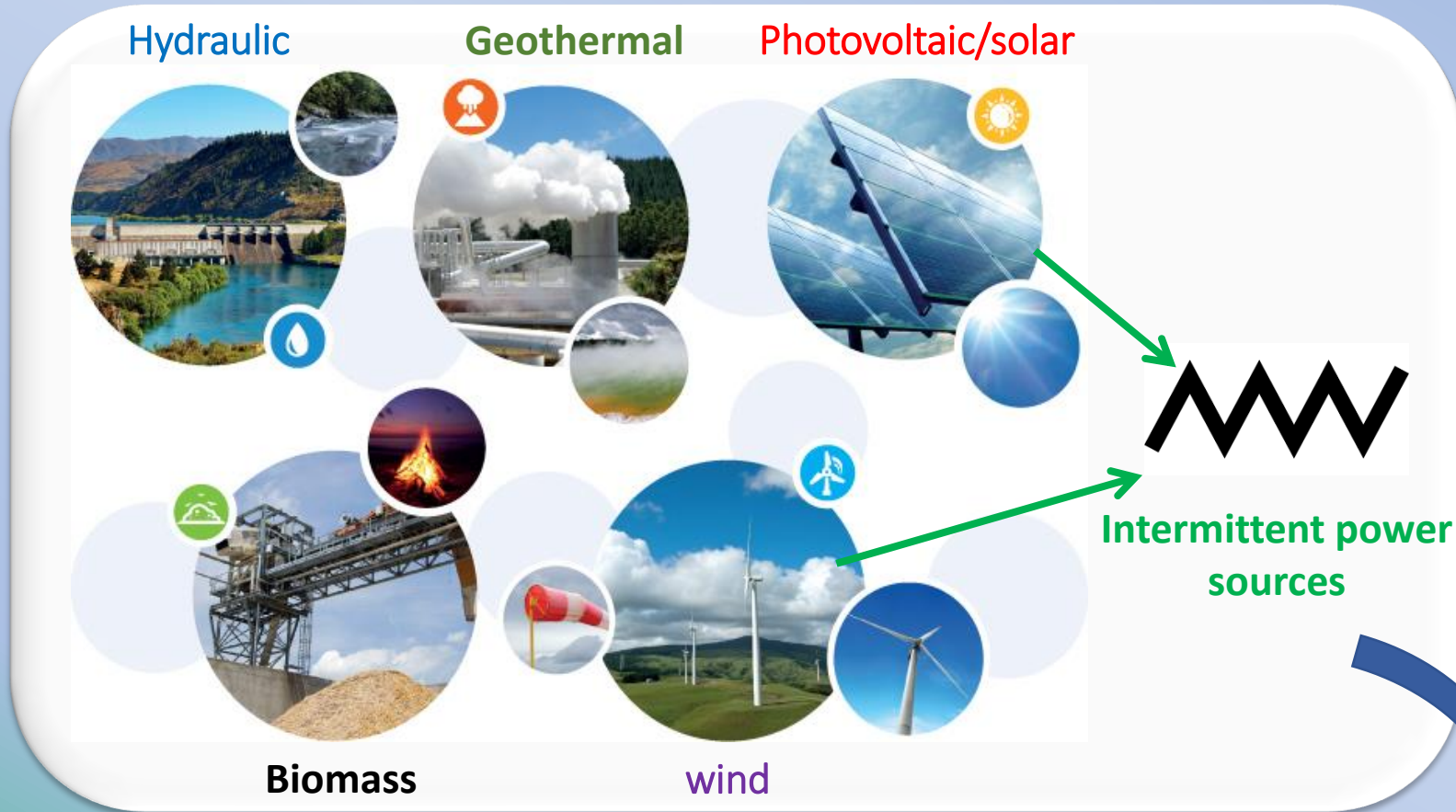


**Decontamination**

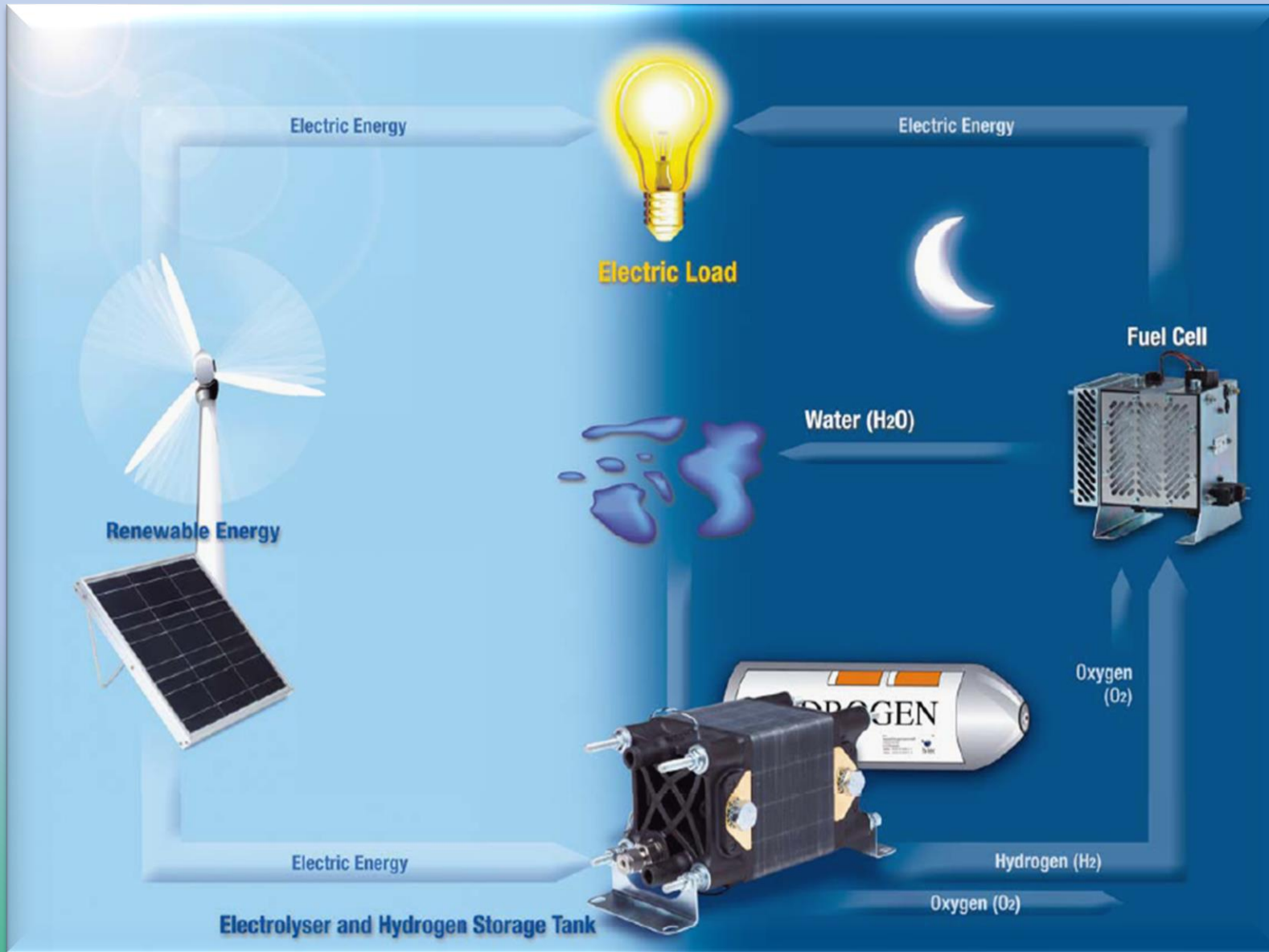
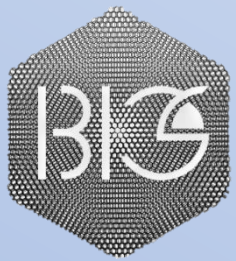


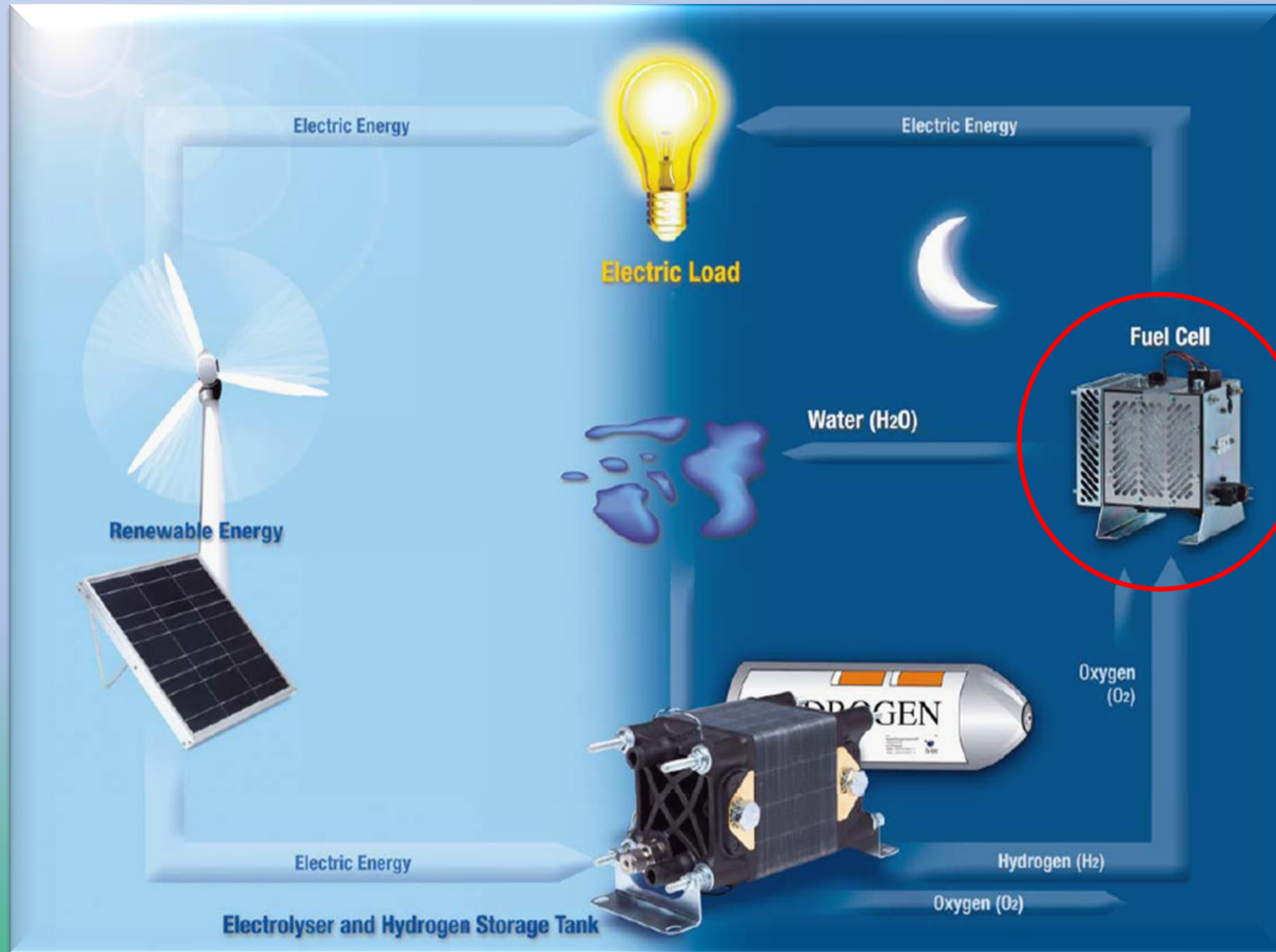
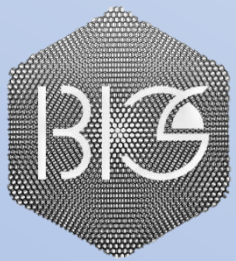


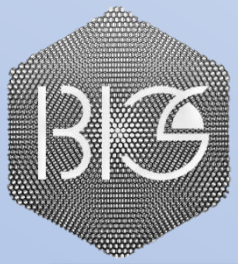
# Renewable energy



It is necessary to connect them to systems that allow energy to be stored to consume it when necessary.







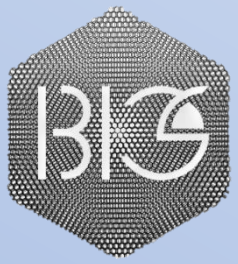
## Fuel Cell

**Cathode (ORR):**  $4\text{H}^+ + 4\text{e}^- + \text{O}_2 \rightarrow 2\text{H}_2\text{O}$   $E^0 = 1.23 \text{ V vs. SHE}$

**Anode (HOR):**  $2\text{H}_2 \rightarrow 4\text{H}^+ + 4\text{e}^-$   $E^0 = 0.00 \text{ V vs. SHE}$

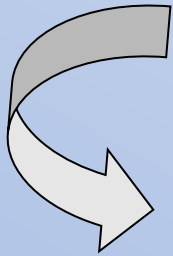
**Global Reaction:**  $\text{O}_2 + 2\text{H}_2 \rightarrow 2\text{H}_2\text{O}$   $E^0 = 1.23 \text{ V vs. SHE}$  ↑

Anode: MeOH, EtOH, **NH<sub>3</sub>**....

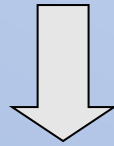


## Synthesis of Catalysts

## Catalyst Requirements

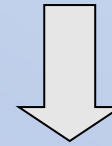


### Catalyst support



- High Surface Area
- Appropriate Porosity
- High Electrical Conductivity

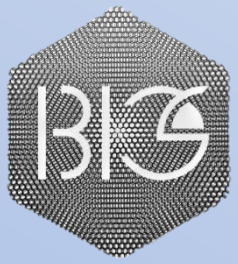
### Metal Nanoparticles



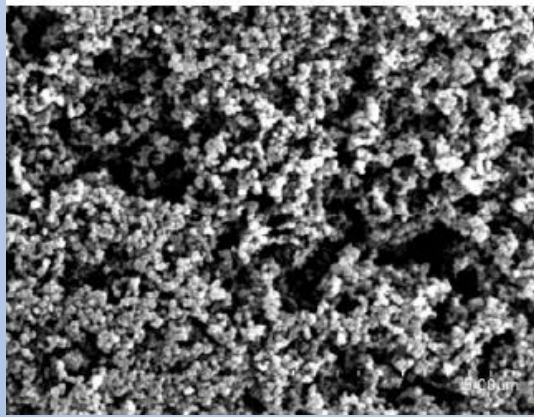
- Small Particle Size
- Uniform Distribution / Strong Interaction
- High Density of Active Sites



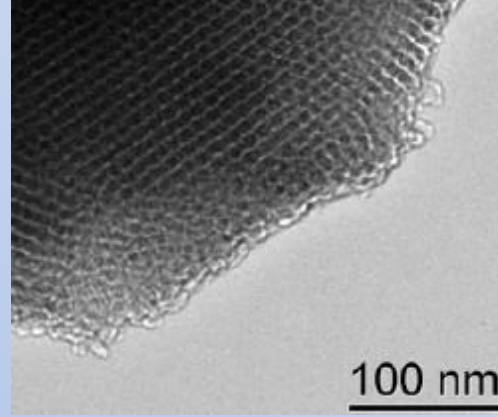
Low Cost!!!!



# Carbon-Based Nanomaterials



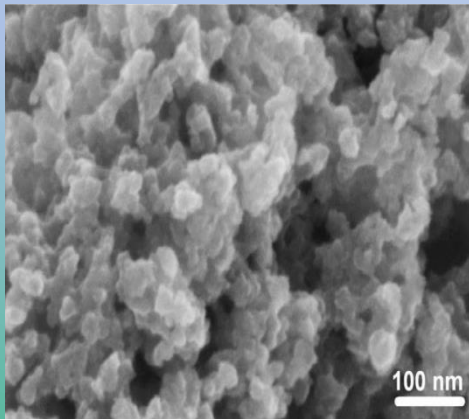
**Carbon Black**



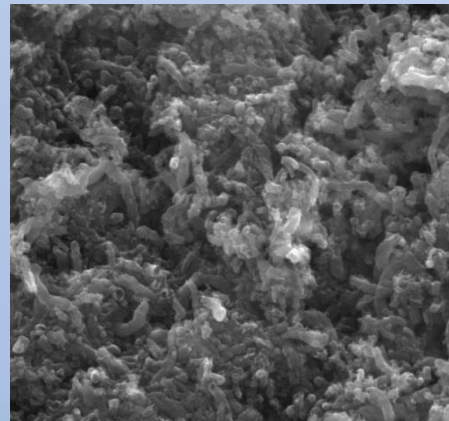
**Mesoporous**



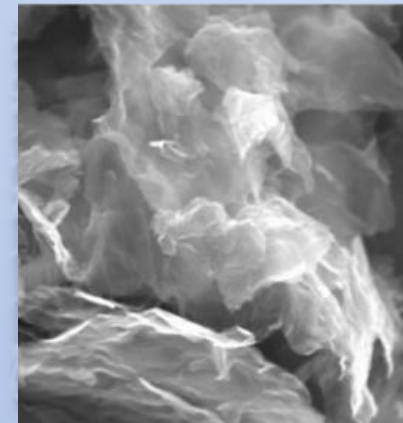
**Nanotubes**



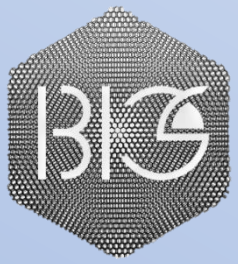
**Xerogel**



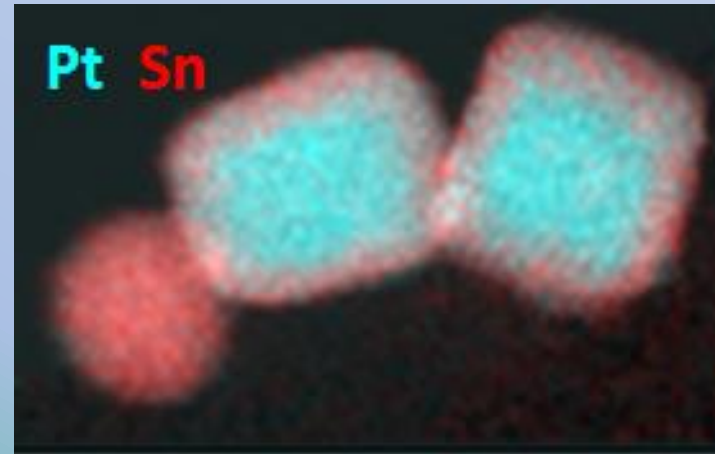
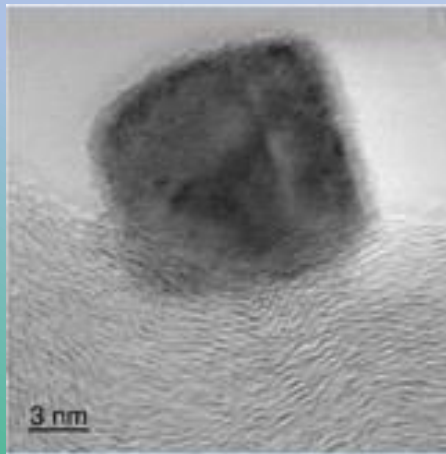
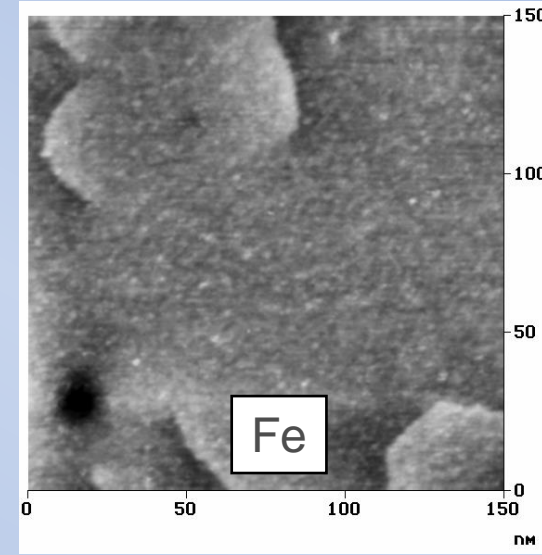
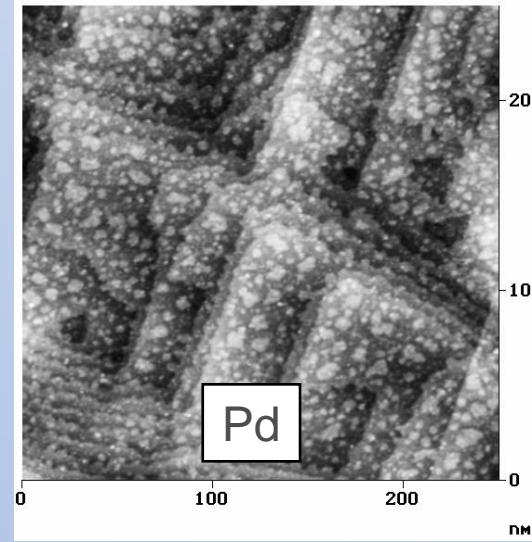
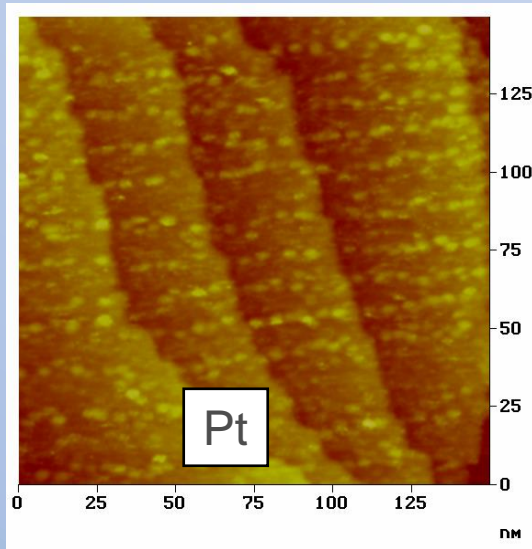
**Nanofibres**

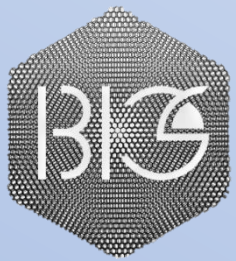


**Graphene**

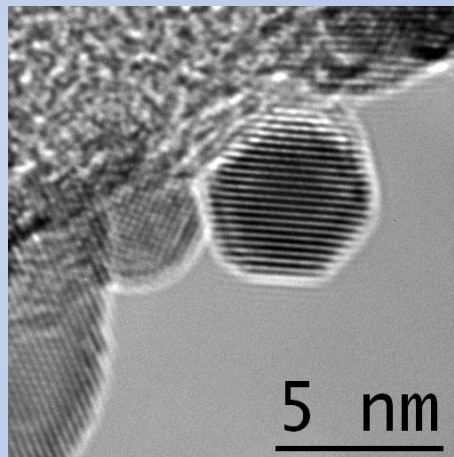
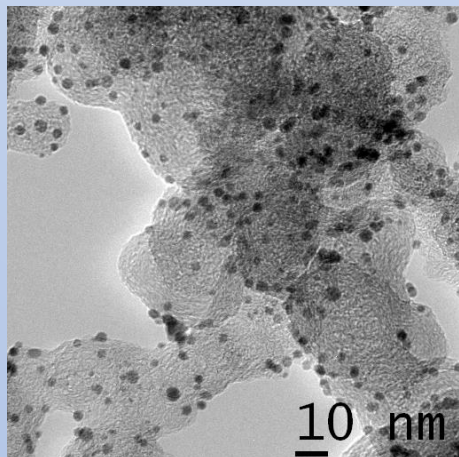


# Metal-Based Nanomaterials

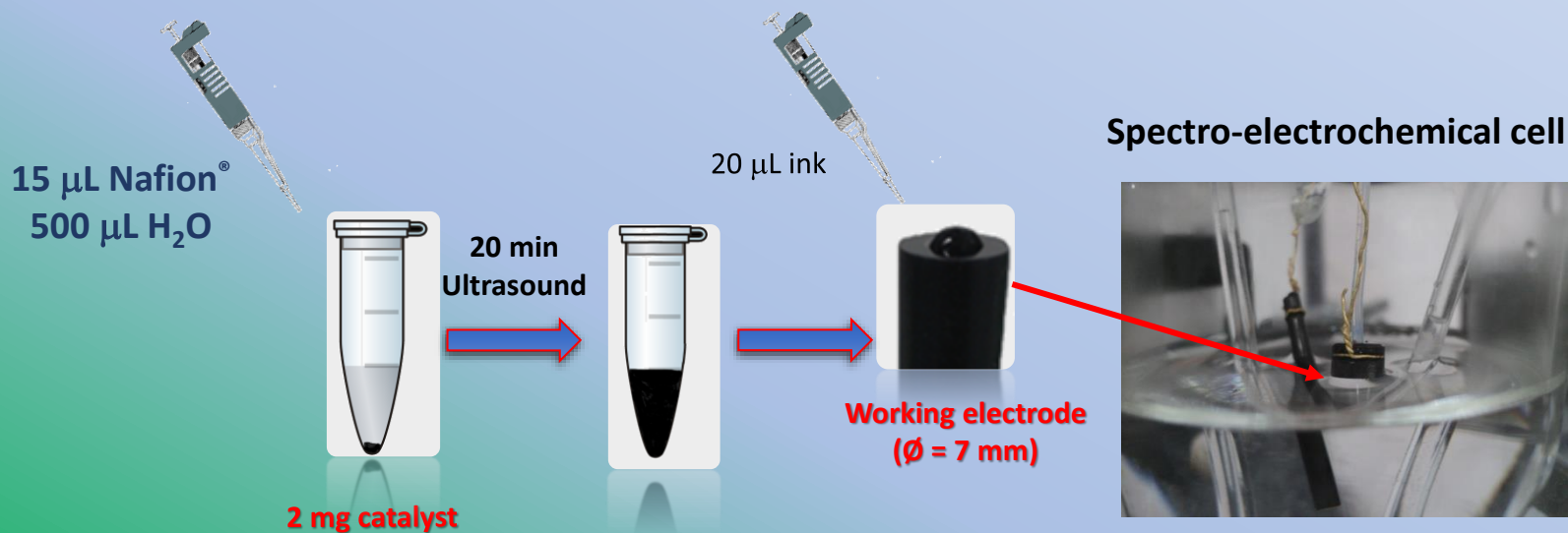




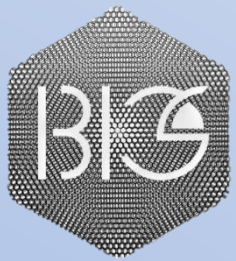
# Carbon-Supported Metal Nanocatalysts



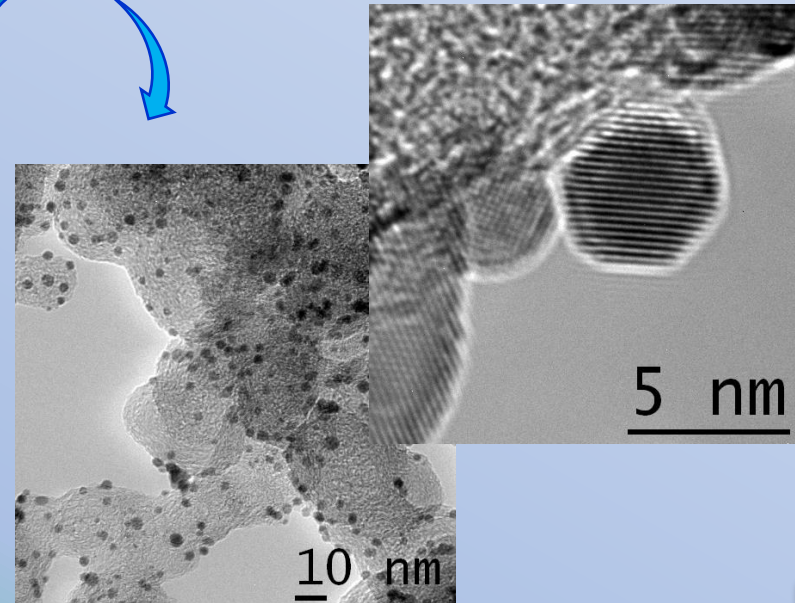
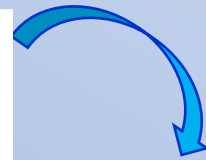
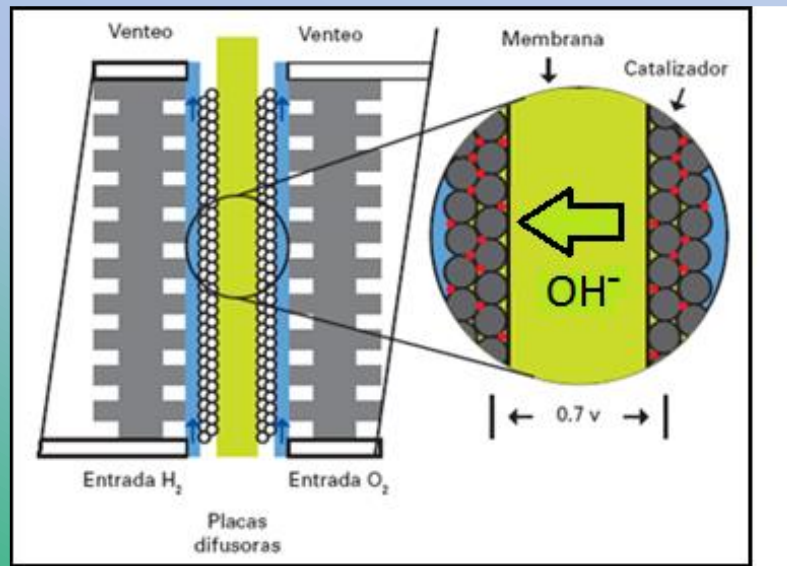
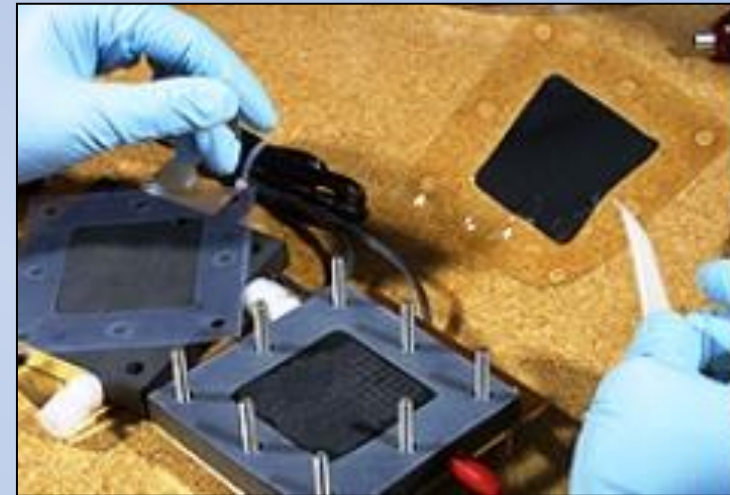
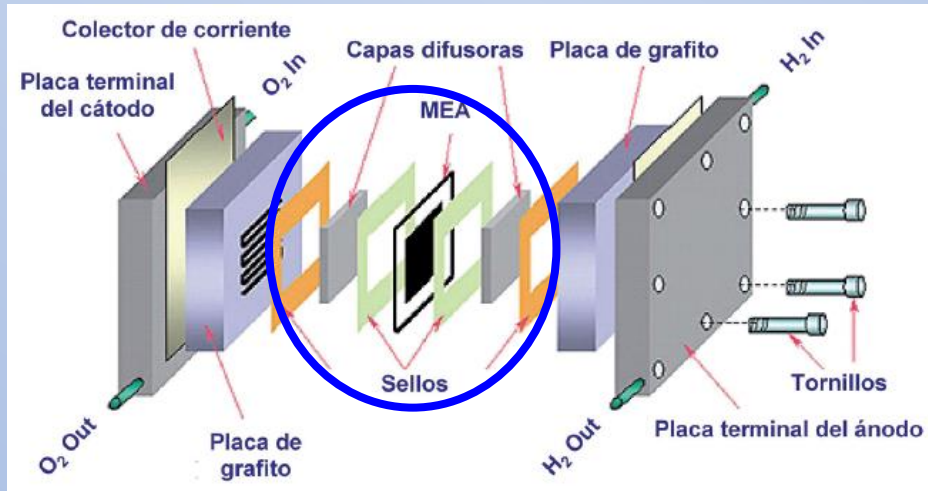
## Catalyst-Ink Preparation



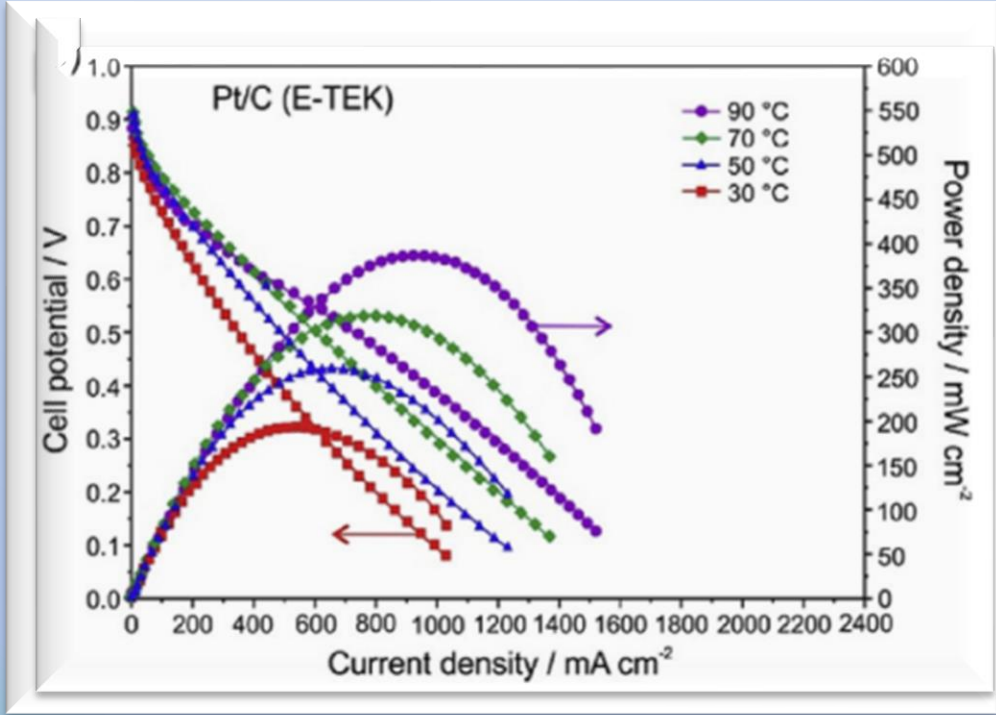
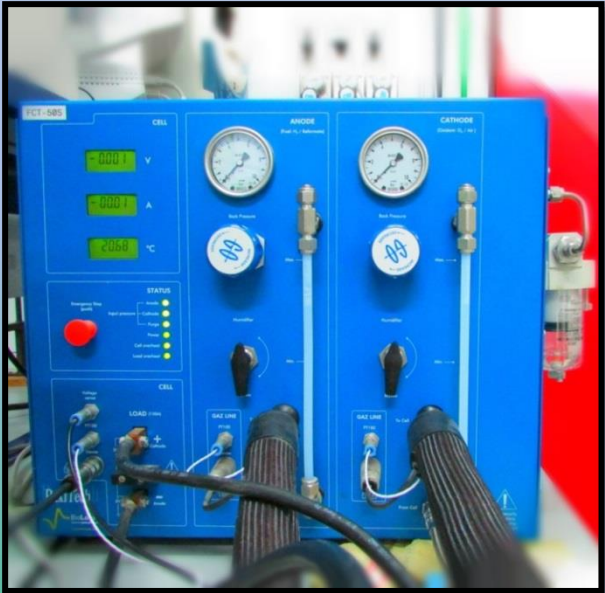
- CV
- LSV
- IES
- RRDE
- RDE
- DEMS
- FTIRS
- EC-Raman
- ....

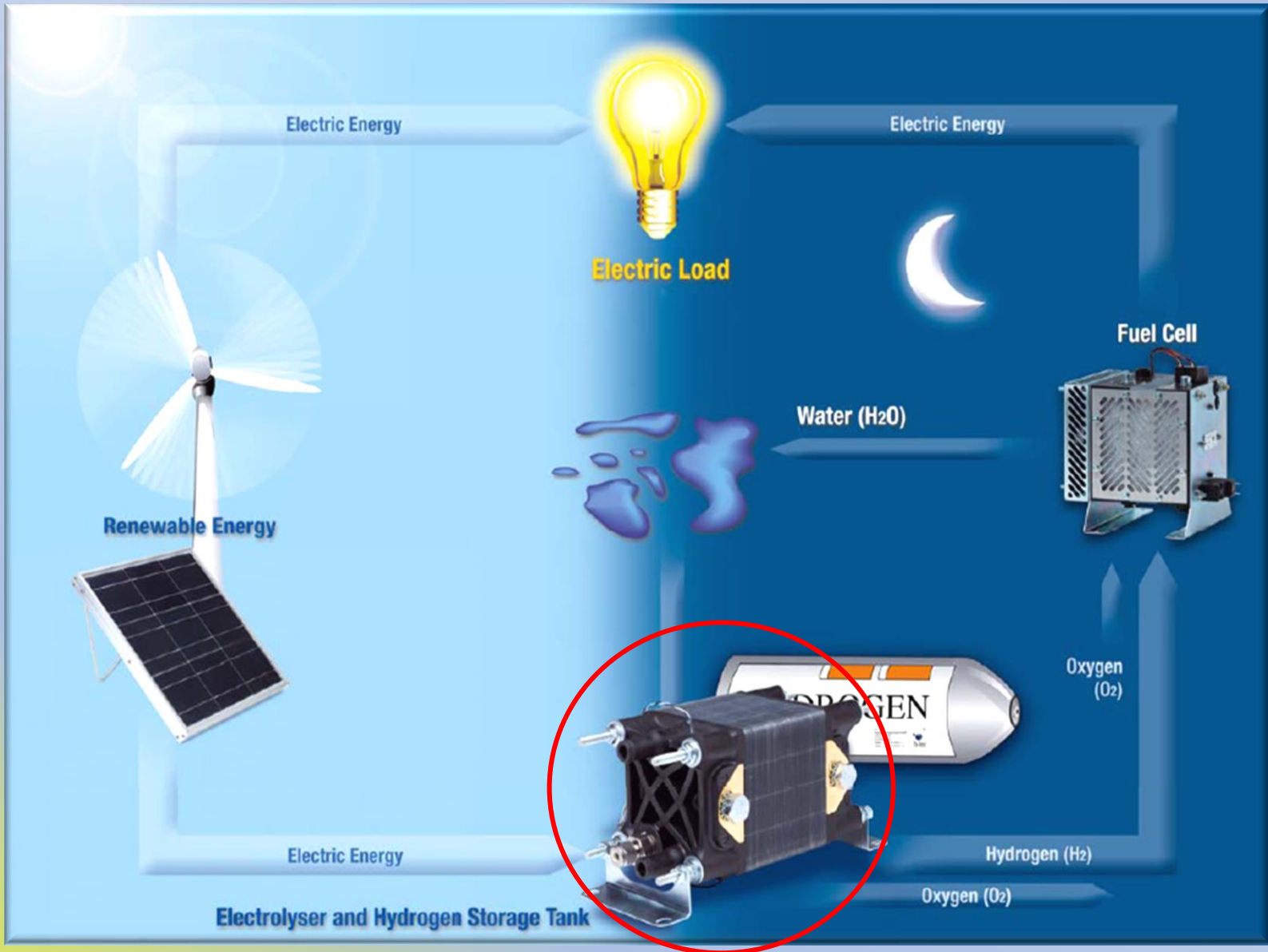
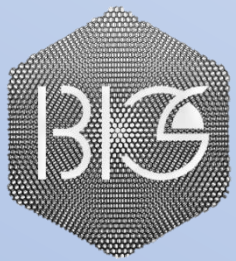


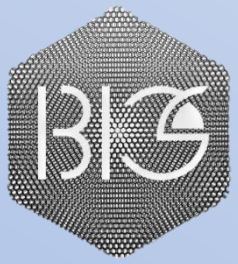
# PEMFC - DAFC



# PEMFC - DAFC



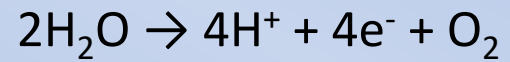




# Electrolysers

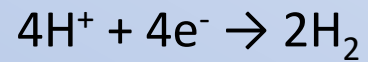


**Anode (OER):**



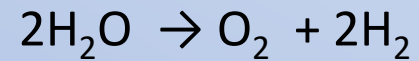
$$E^0 = -1.23 \text{ V vs. SHE}$$

**Cathode (HER):**



$$E^0 = 0.00 \text{ V vs. SHE}$$

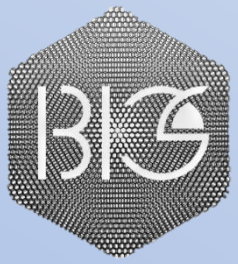
**Global Reaction:**



$$E^0 = -1.23 \text{ V vs. SHE}$$

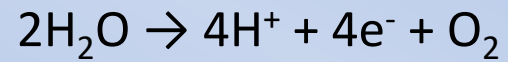


Cathode: Nitrogen Oxides....



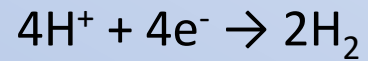
# Electrolysers

**Anode (OER):**



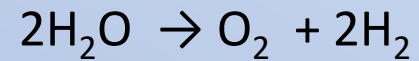
$$E^0 = 1.23 \text{ V vs. SHE}$$

**Cathode (HER):**



$$E^0 = 0.00 \text{ V vs. SHE}$$

**Global Reaction:**

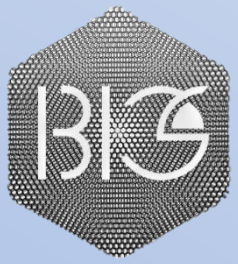


$$E^0 = 1.23 \text{ V vs. SHE}$$



Cathode: Nitrogen Oxides....

¿¿¿& now???



# Electrolysers



**Anode (OER):**  $2\text{H}_2\text{O} \rightarrow 4\text{H}^+ + 4\text{e}^- + \text{O}_2$   $E^0 = 1.23 \text{ V vs. SHE}$

**Cathode (HER):**  $4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2$   $E^0 = 0.00 \text{ V vs. SHE}$

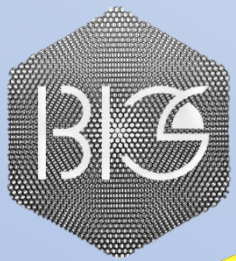
**Global Reaction:**  $2\text{H}_2\text{O} \rightarrow \text{O}_2 + 2\text{H}_2$   $E^0 = 1.23 \text{ V vs. SHE}$



Photo-Electrochemistry

¿¿¿& now???

- Photosensible.
- High electrical conductivity.
- Corrosion and wear resistant.
- Low-cost.



# Electrolysers



GOBIERNO DE ESPAÑA | MINISTERIO DE INDUSTRIA, COMERCIO Y TURISMO | Oficina Española de Patentes y Marcas

**c.e.o.** Consulta de Expedientes OEPM

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**Patente nacional P202000050(4) - NANOMATERIALES ALTAMENTE EFICIENTES**

- Nº Solicitud: P202000050 | Fecha presentación: 06/04/2020
- Nº publicación: ES2862426 | Fecha publicación: 07/10/2021 | Fecha concesión: 21/02/2022
- Título: NANOMATERIALES ALTAMENTE EFICIENTES
- Estado: Concesión | Última actualización de la base de datos: 09/09/2022

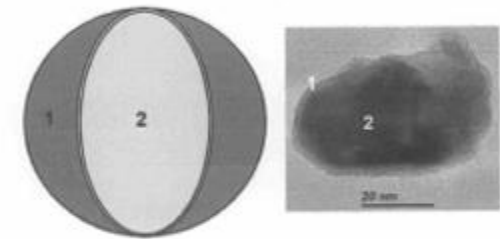
**Solicitante / Titular:**

Nombre: Light Bridges, SL (100,00%)  
 Dirección: Avda Alcalde Ramírez Bethencourt 17,  
 Localidad: Las Palmas de Gran Canaria  
 Provincia: Las Palmas  
 Código Postal: 35004  
 País de residencia: ES ESPAÑA

**Inventor/es:**  
 Gonzalo GARCÍA SILVESTRO

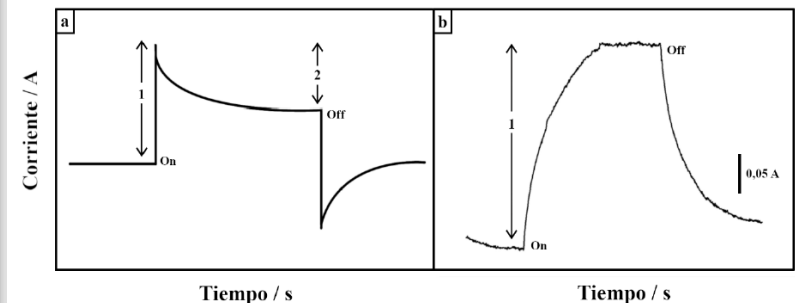
1<sup>ST</sup> LATAM ON GREEN AMMONIA POWER-to-X MEETING

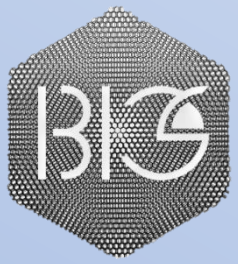
Figura 1



## 57 Resúmen:

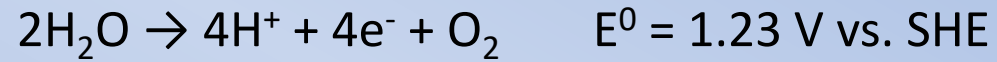
Uso de nanopartículas con actividad fotoelectrocatalítica altamente eficiente. La obtención de morfologías del tipo núcleo-coraza formada por carburos-óxidos de metales de transición produce un efecto de sinergia para evitar los procesos de recombinación electrón/hueco, aumentar la conductividad eléctrica y la actividad electrocatalítica y fotoelectrocatalítica. Así, se aumenta el rendimiento fotoelectrocatalítico que presentan hacia distintas reacciones fotoelectroquímicas de interés, como puede ser la generación de hidrógeno, reducción de dióxido de carbono y de nitrato.



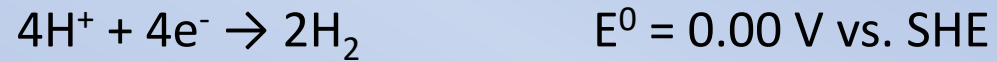


# Electrolysers

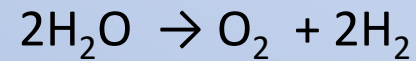
**Anode (OER):**



**Cathode (HER):**



**Global Reaction:**



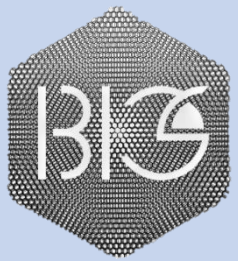
Anodic reaction

## Photo-Electrochemistry

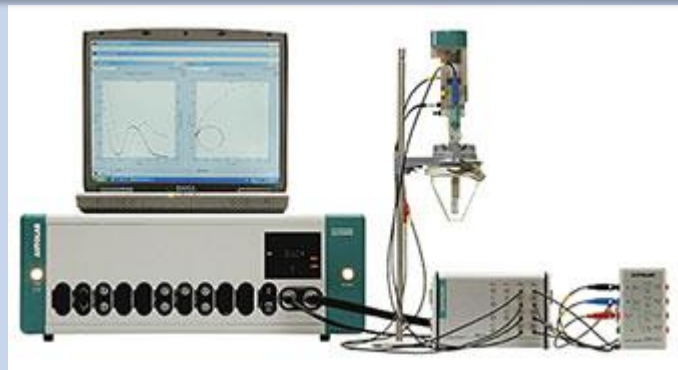
- Photosensible.
- High electrical conductivity.
- Corrosion and wear resistant.
- Low-cost.

**Degradation of pollutants in water e.g.:**

- Alcohols, Paracetamol, Urea, Hydrocarbons...



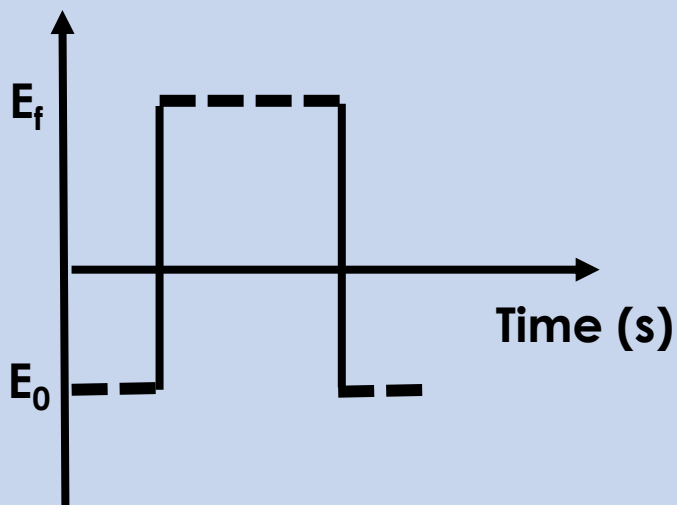
# Spectro-Electrochemical techniques



Potentiostat  
Galvanostat

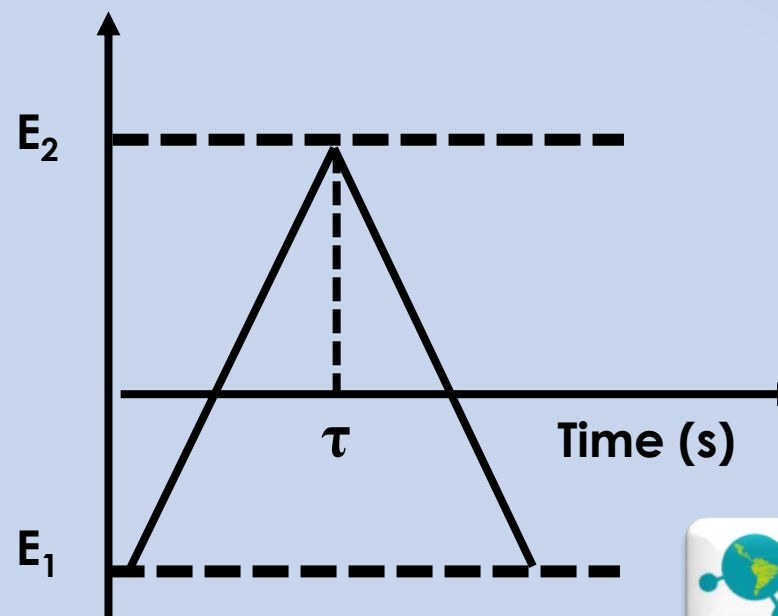
## Potentiostatic Techniques

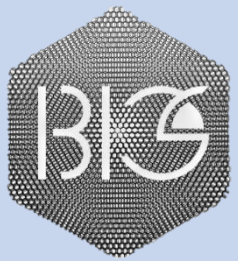
Potential (V)



## Potentiodynamic Techniques

Potential (V)

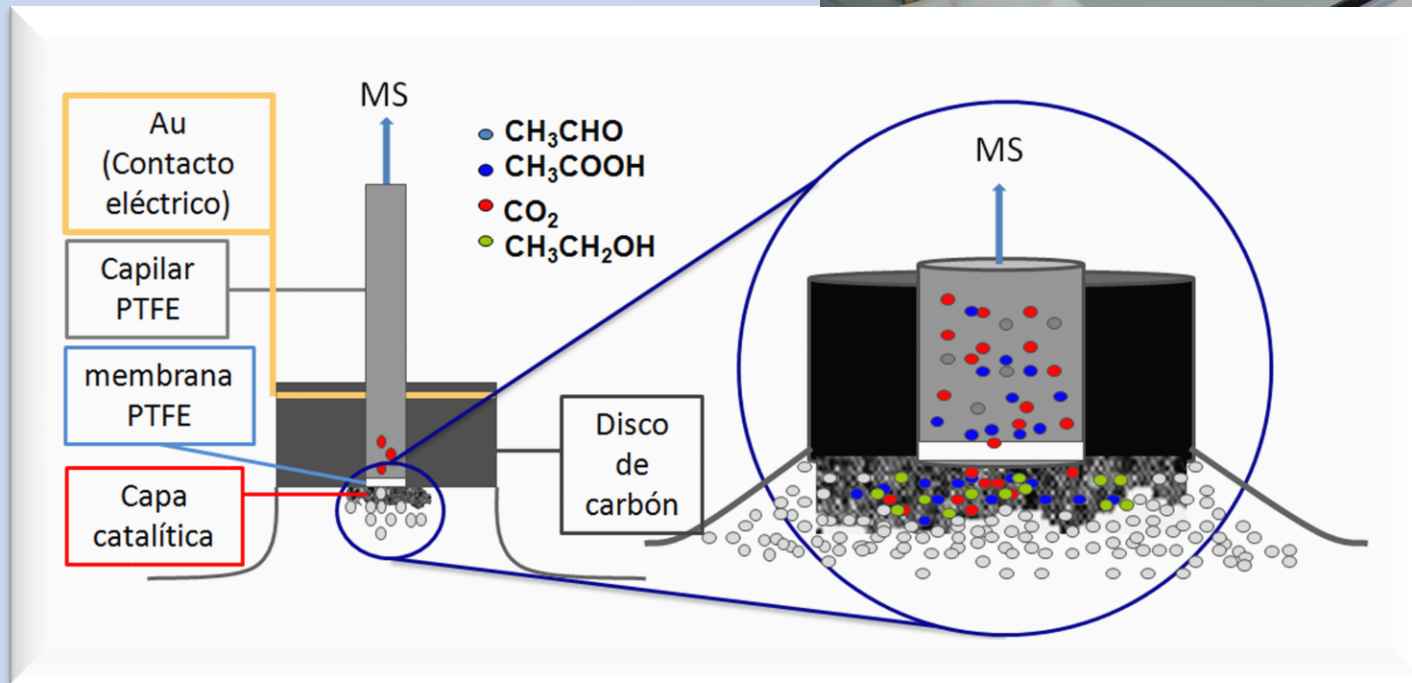
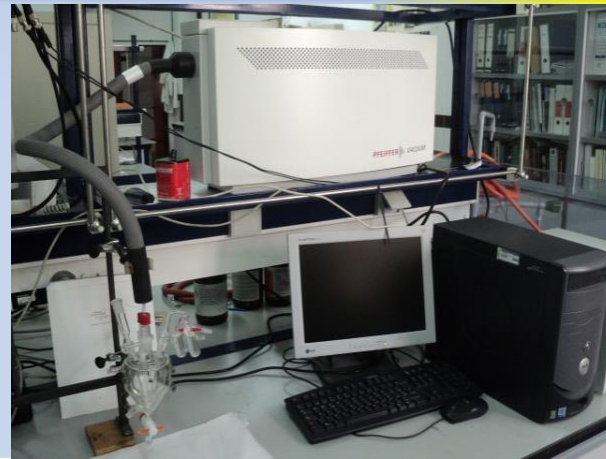




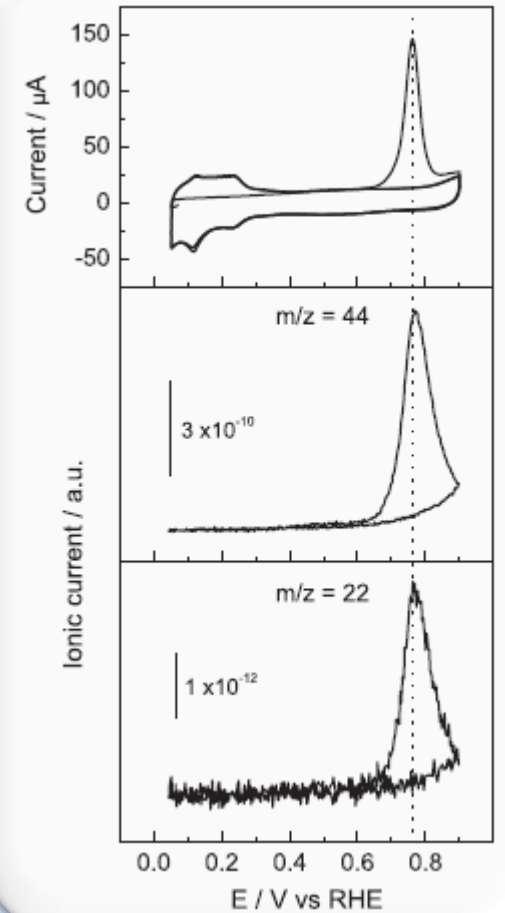
# Spectro-Electrochemical techniques

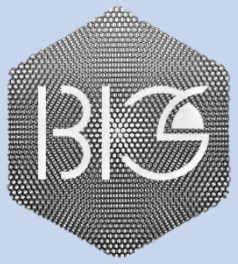


## Differential Electrochemical Mass Spectrometry



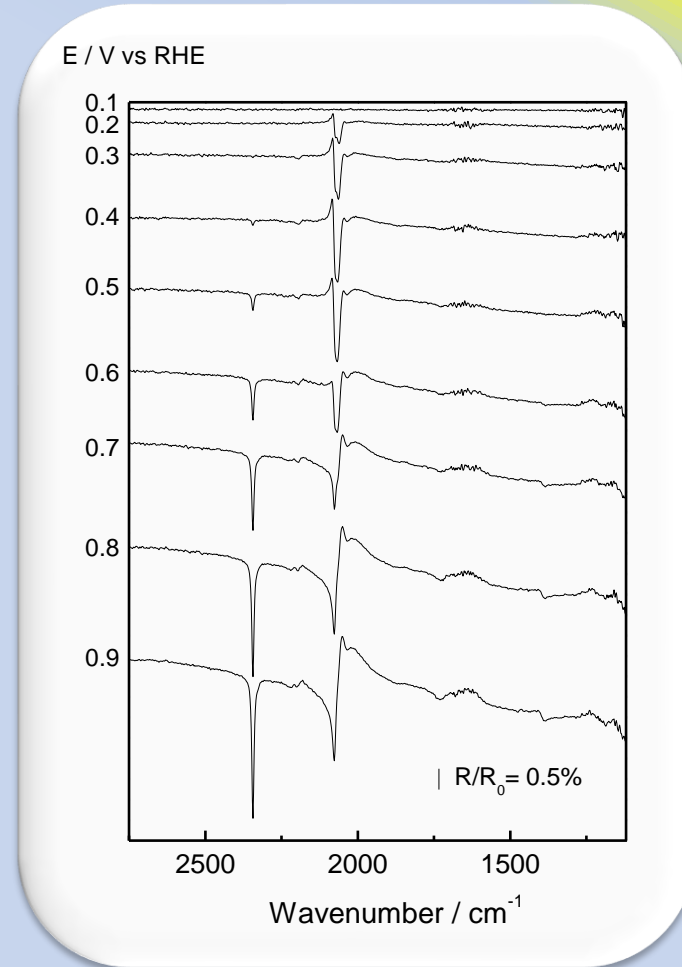
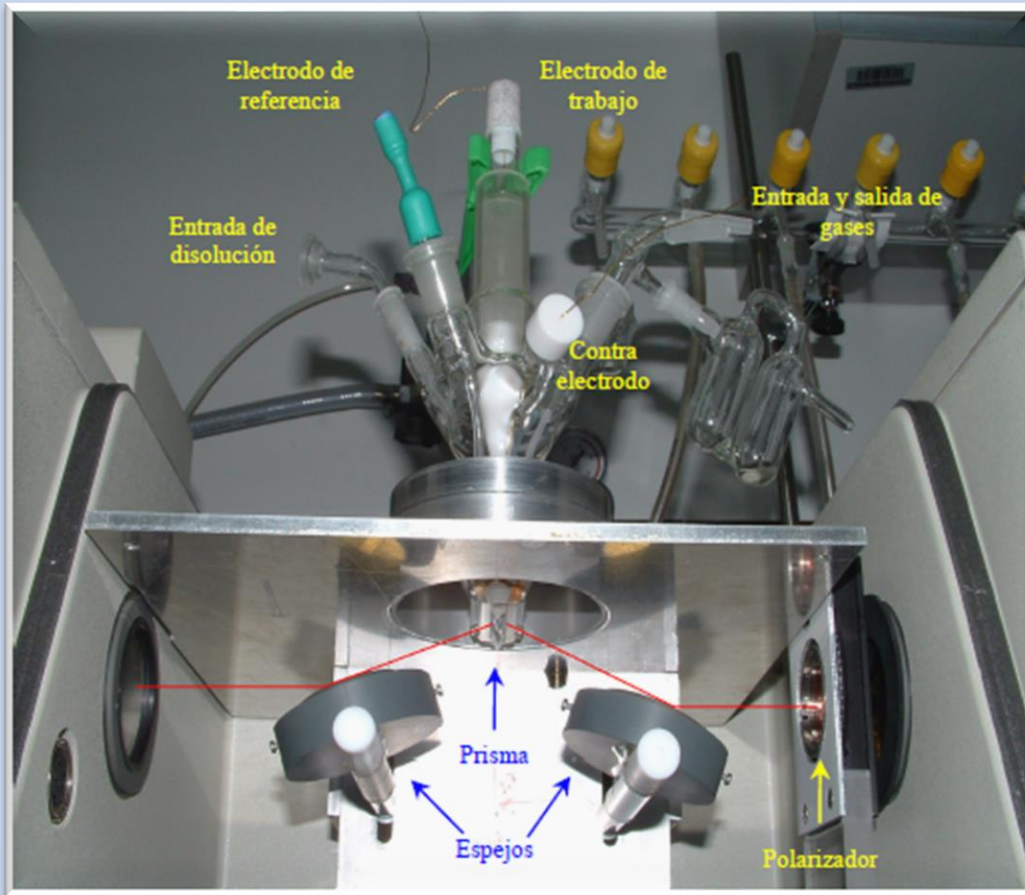
Several Configurations!

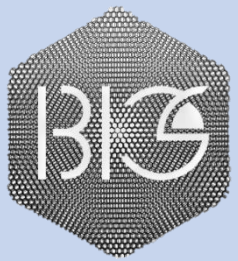




# Spectro-Electrochemical techniques

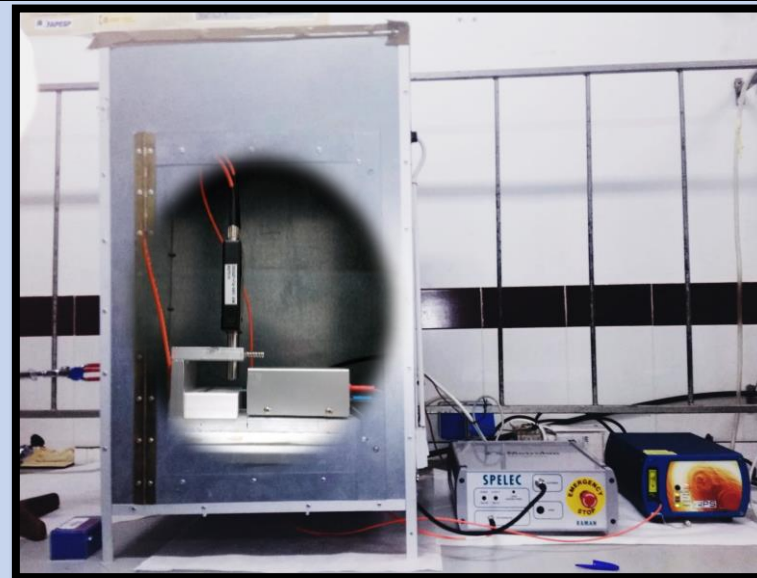
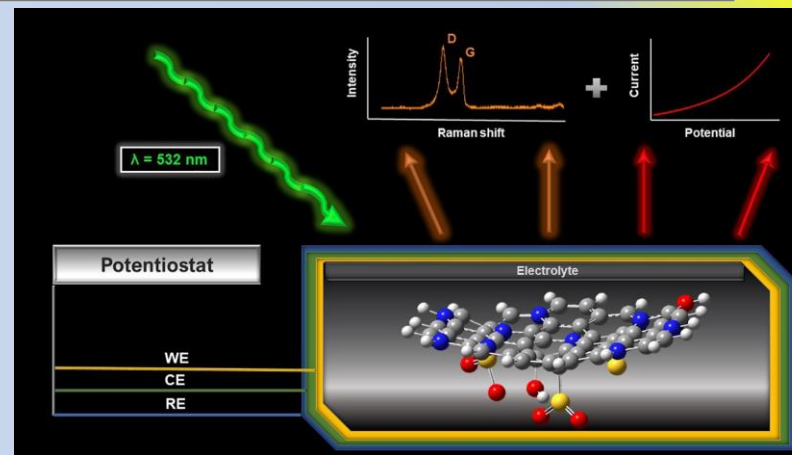
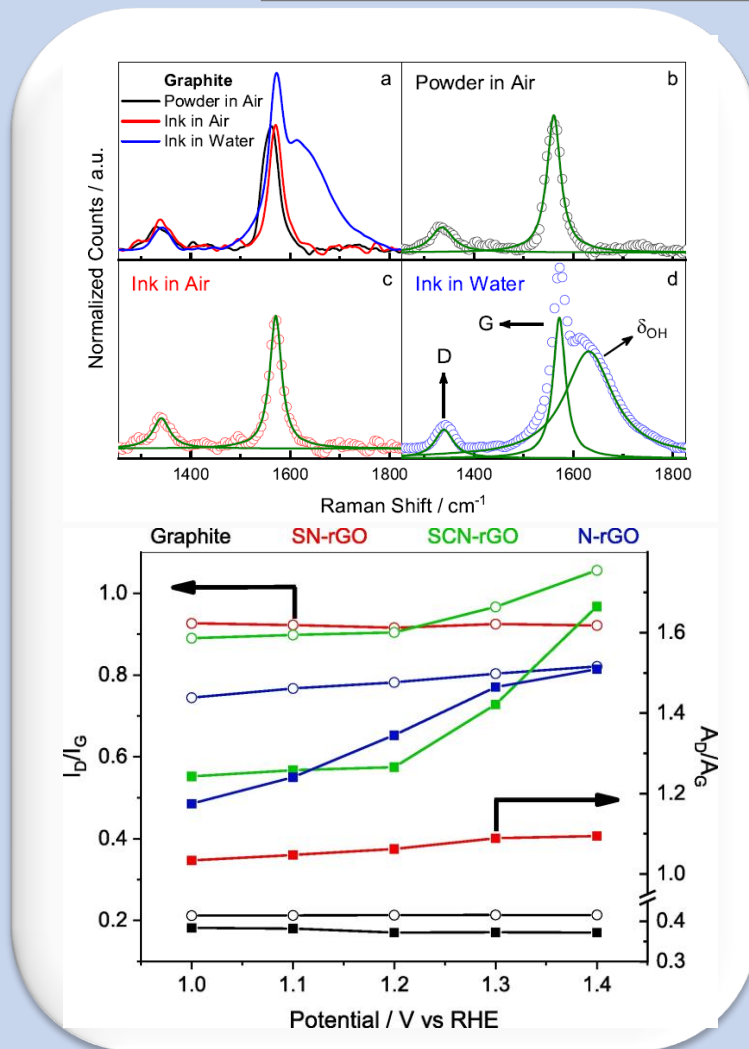
## In Situ Fourier Transform Infrared Spectroscopy





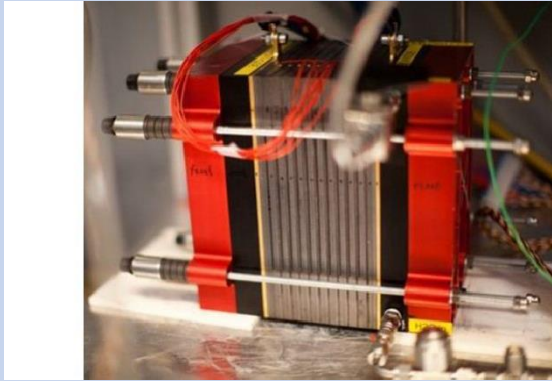
# Spectro-Electrochemical techniques

## Raman spectroelectrochemistry



# In summary

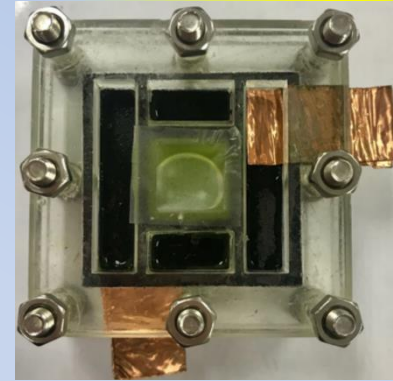
Fuel Cells



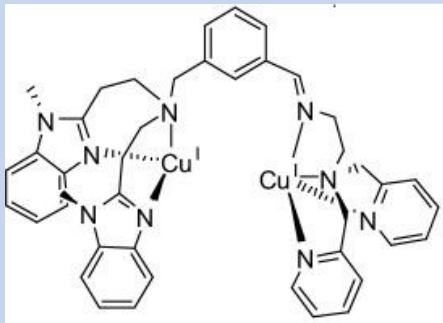
Electrolysers



CO<sub>2</sub> reduction



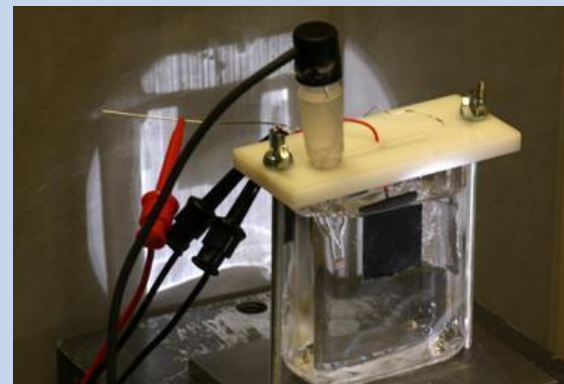
Biomimetic / Biosensor



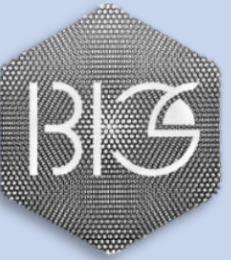
Single Crystals



Photo(electro)chemistry



Corrosion





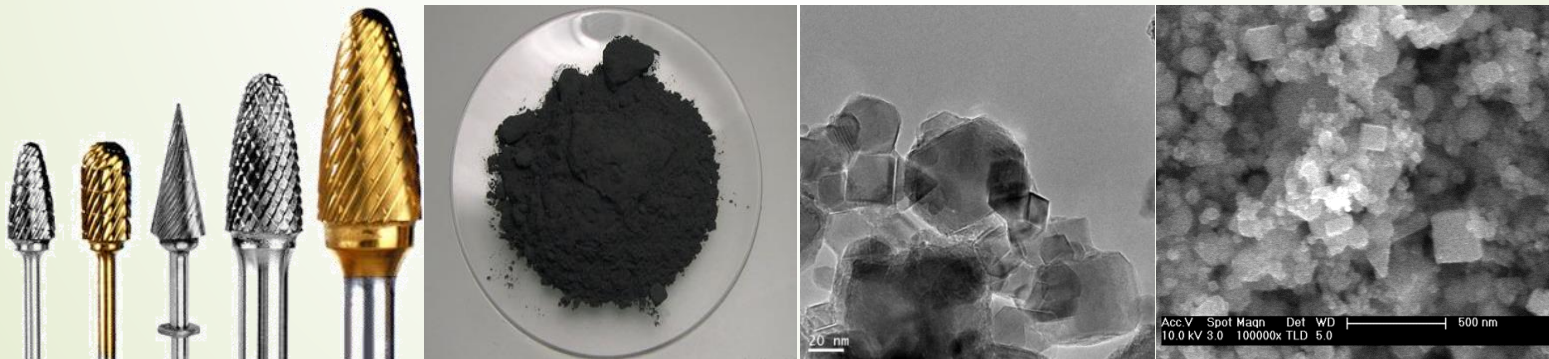
# TMCs as Catalyst and Catalyst Support





# What is the reason behind the explosion of interest in transition 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.

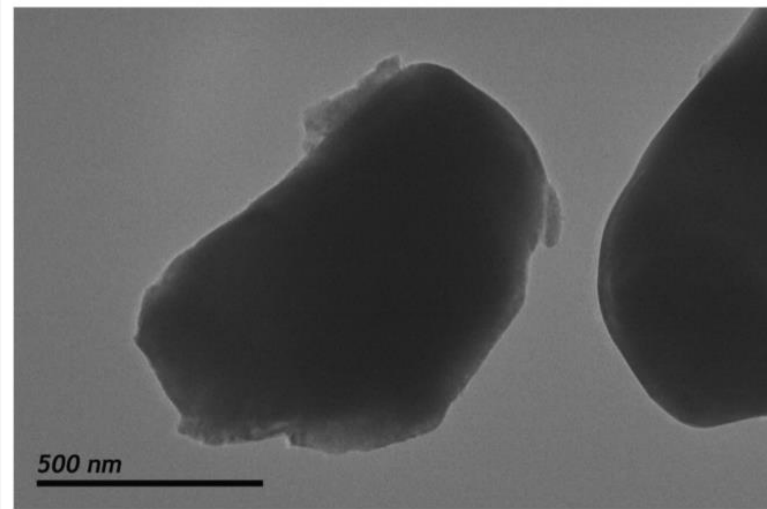




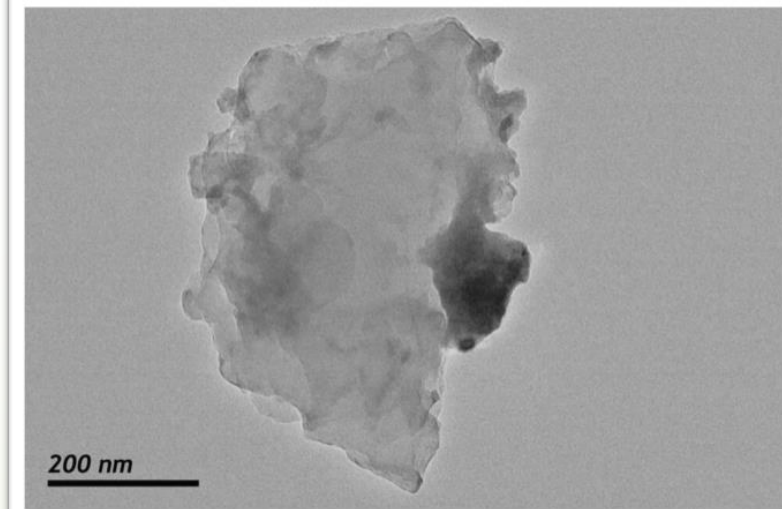
## Most common drawbacks of transition metal carbides

- Low surface area and **big particle size**
- Several phases (including oxides) are usually observed
- Crystalline structure is commonly lost under oxidative media

Commercial  $\text{Mo}_2\text{C}$

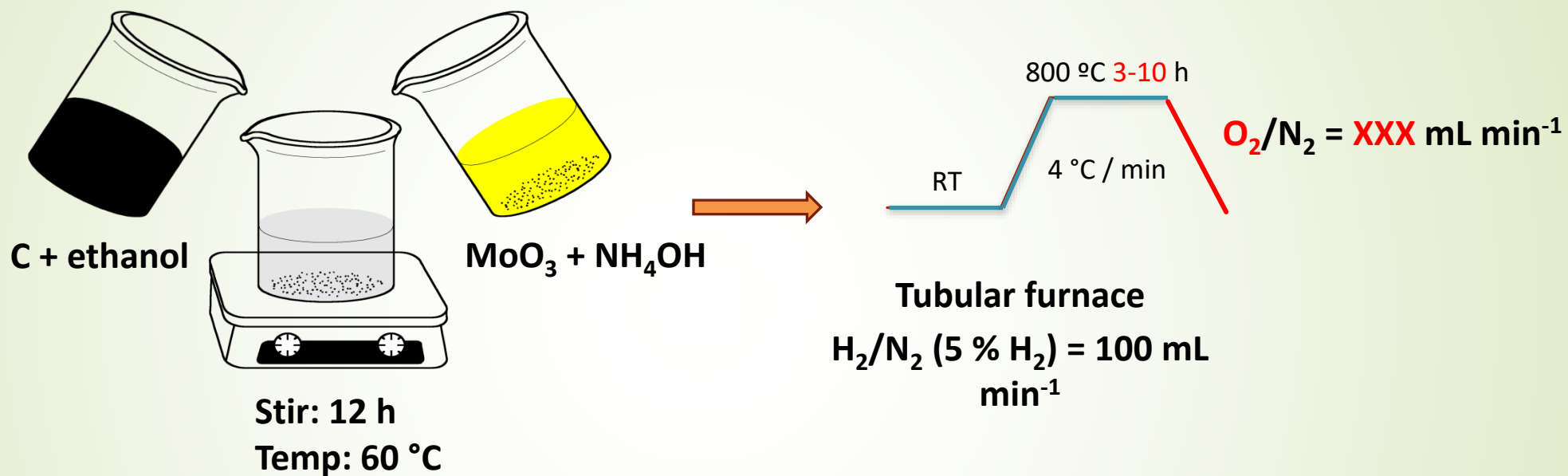


Commercial  $\text{W}_2\text{C}$





# I. Synthesis of $\text{Mo}_2\text{C}$ by the Carbothermal Route

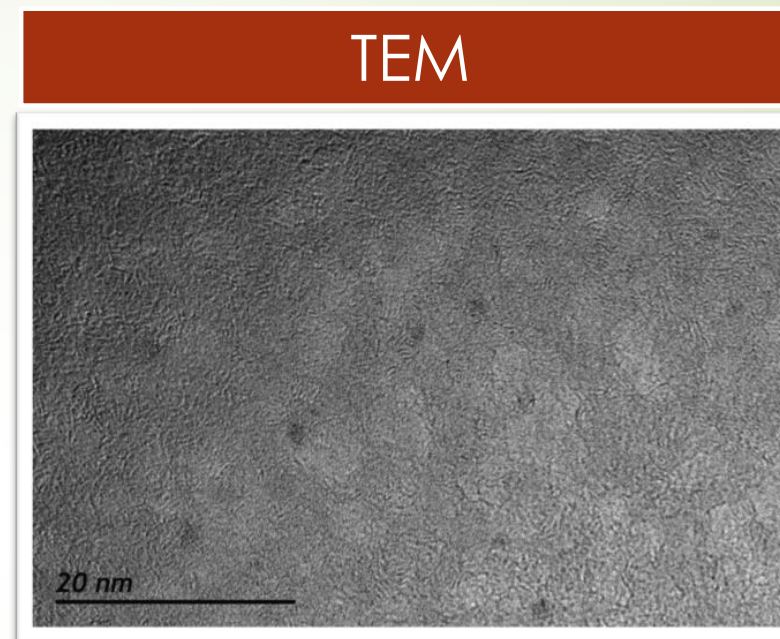
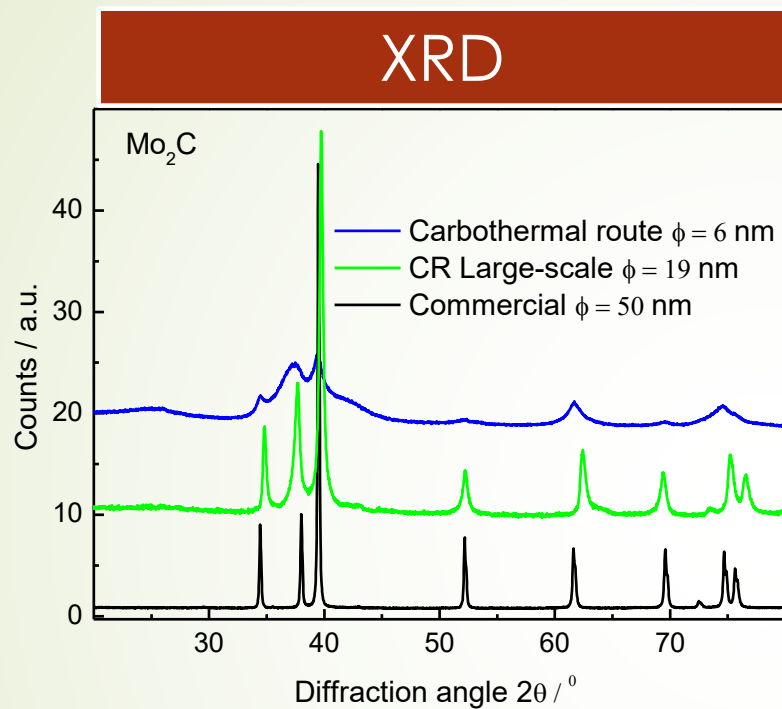


Low-cost and easy method





# I. Synthesis of $\text{Mo}_2\text{C}$ by the Carbothermal Route

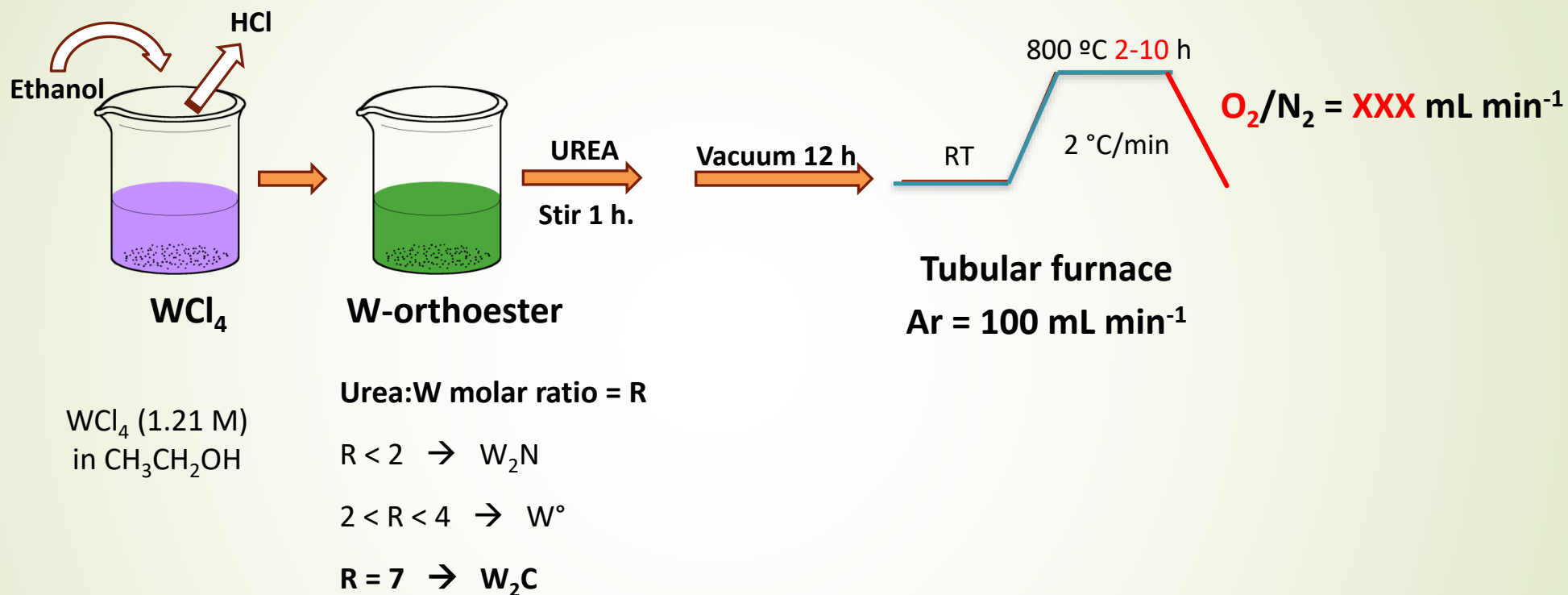


Catalyst	Main phase/s	Secondary phase/s	Crystallite Size (nm)
Mo <sub>2</sub> C (commercial)	Mo <sub>2</sub> C	---	> 50
Mo <sub>2</sub> C (carbothermal route)-0.1 gram	Mo <sub>2</sub> C	---	6
Mo <sub>2</sub> C (carbothermal route) Large-Scale	Mo <sub>2</sub> C	---	19





## II. Synthesis of $W_2C$ by the Urea-Glass Route



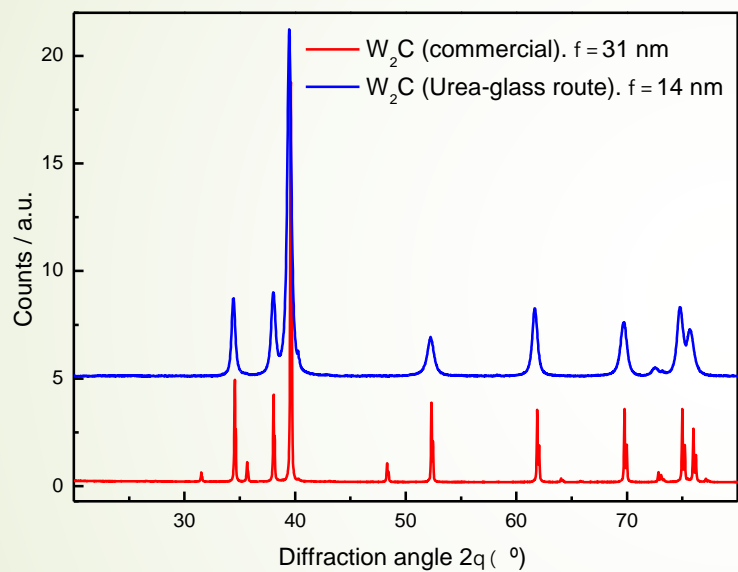
Low-cost. Numerous materials can be synthesized  
but is not facile method



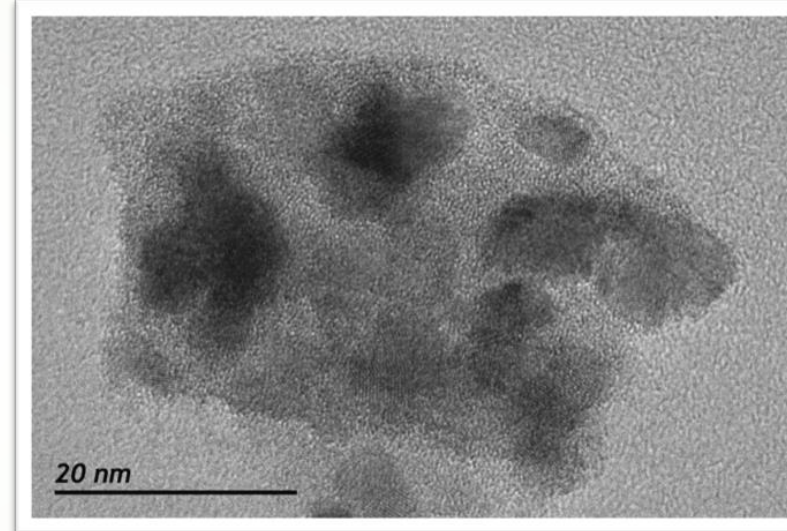


## II. Synthesis of $W_2C$ by the Urea-Glass Route

X-ray diffraction



Transmission electron microscopy



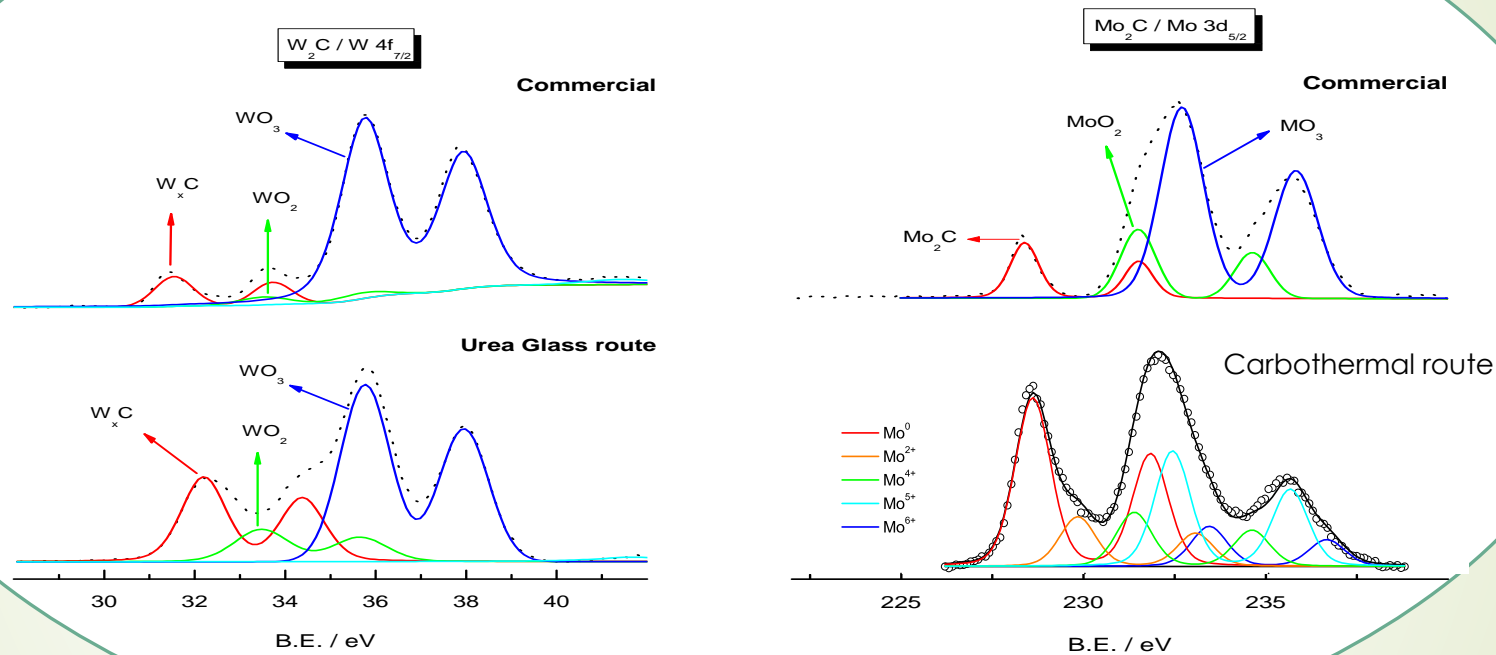
Catalyst	Main phase/s	Secondary phase/s	Crystallite Size (nm)
$W_2C$ (Commercial)	$W_2C$	WC	> 50
$W_2C$ (urea glass route)	$W_2C$	---	14





# What about TMC surface?

## X-ray photoelectron spectroscopy



Surface: metal carbide + transition metal oxides



# What are metal carbides?

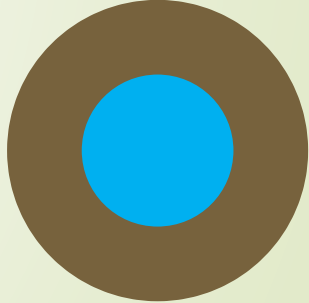
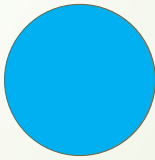
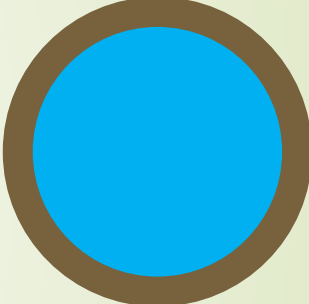
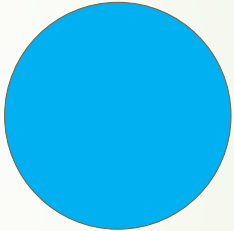
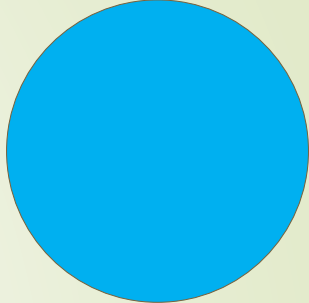
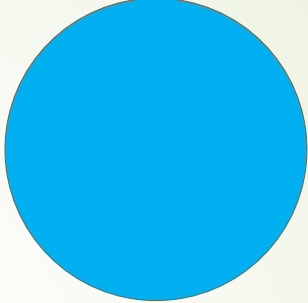
**TRANSITION  
METAL CARBIDE /  
NITRIDE**



**TRANSITION METAL OXIDES  
+  
TRANSITION METAL  
CARBIDES / NITRIDES**

**Time @ highest Temp**

**O<sub>2</sub> @ cooling step**



**Good conductivity – Small particle size –  
High surface area - Low-cost production ....**





# TMCs as Catalyst and Catalyst Support



- **TMC as Electrocatalyst:**

- *Hydrogen Evolution Reaction on  $\text{Mo}_2\text{C}$*

- **TMC as Electrocatalyst Support:**

- *Electrooxidation of Adsorbed CO on 20 wt.% Pt/ $\text{Mo}_2\text{C}$*

- *Electrooxidation of Adsorbed CO on 20 wt.% Pt/TiC, Pt/TiCN and Pt/TiN*

- *Electrooxidation of Methanol on 20 wt.% Pt/TiC, Pt/TiCN and Pt/TiN*

- *Electrooxidation of Ethanol on 16 wt.%  $\text{Pt}_{(3)}\text{Au}_{(3)}\text{Sn}_{(10)}/\text{W}_2\text{C}$  and  $\text{Pt}_{(3)}\text{Au}_{(3)}\text{Sn}_{(10)}/\text{C}$*

- **TMC as Photocatalyst:**

- *Photoreduction of  $\text{CO}_2$  on  $\text{Mo}_2\text{C}/\text{TiO}_2$*

- **TMC as Photoelectrocatalyst:**

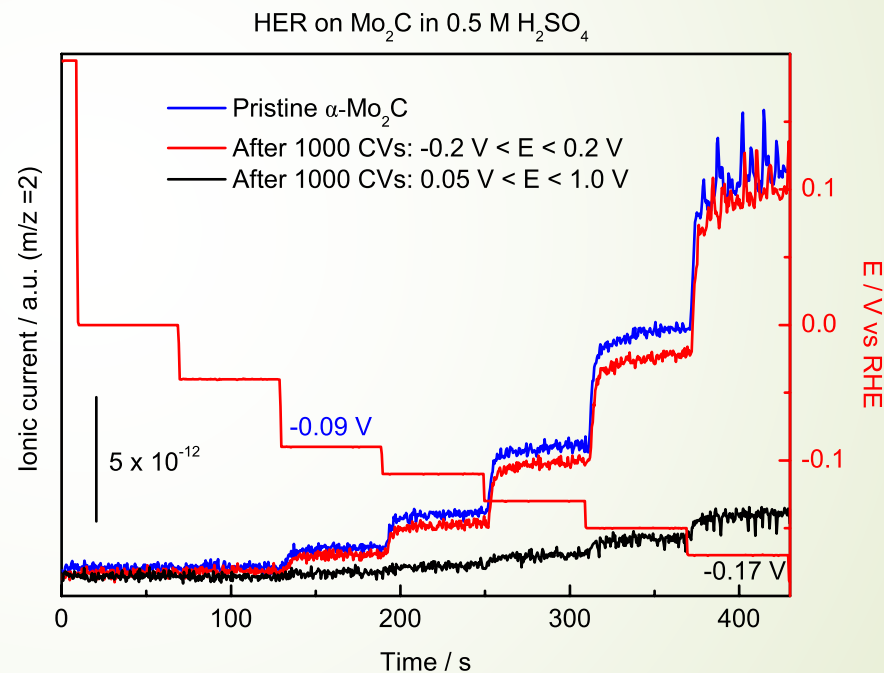
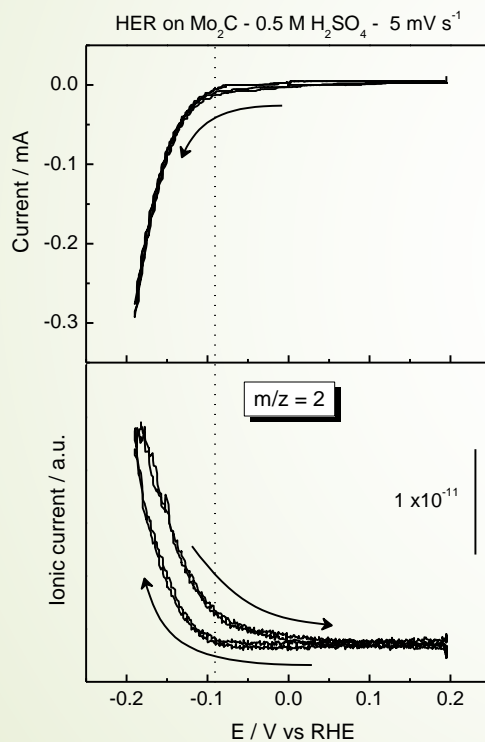
- *Patente: Uso de nanomateriales como catalizadores: P202000050*



# TMCs as Electrocatalyst: Hydrogen Evolution Reaction



## Electrochemical activity and stability of $\text{Mo}_2\text{C}$ in sulphuric acid medium

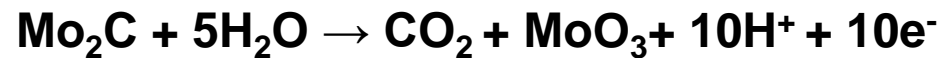
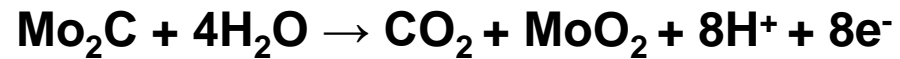
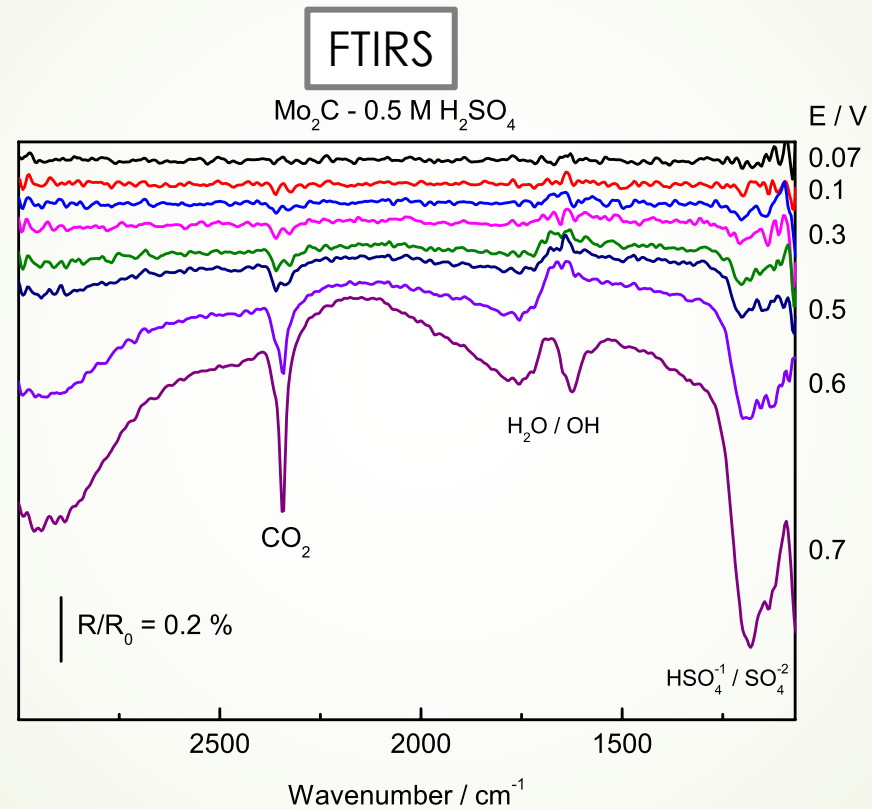
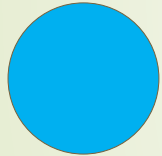


Onset potential strongly depends on the nature of the TMC



# TMCs as Electrocatalyst: Hydrogen Evolution Reaction (HER)

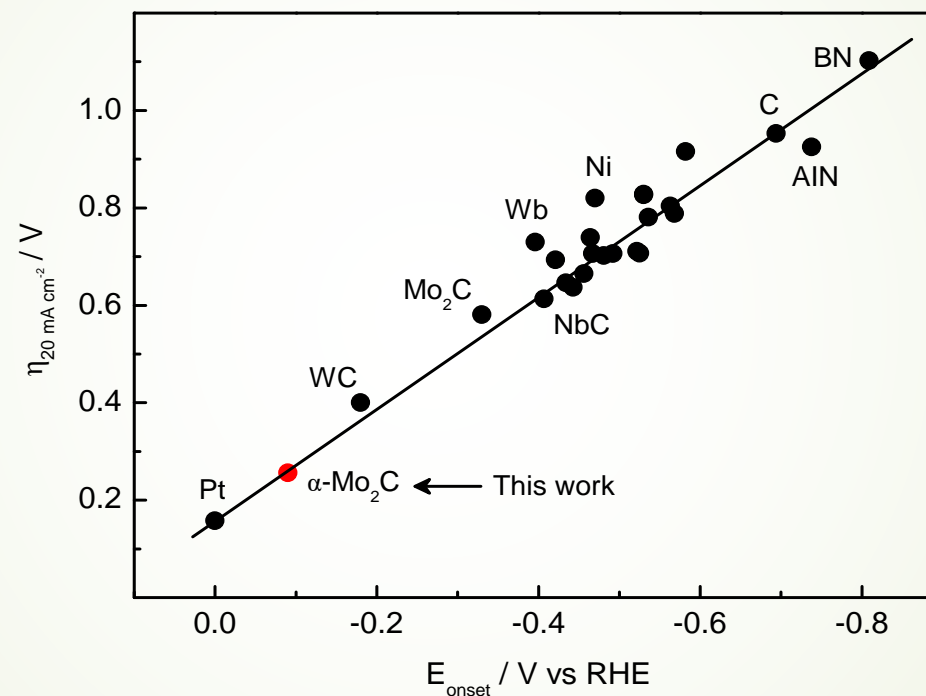
## Electrochemical stability of Mo<sub>2</sub>C in sulphuric acid medium





# TMCs as Catalyst: Hydrogen Evolution Reaction (HER)

$\eta$  @ -20 mA cm<sup>-2</sup> vs Onset Potential



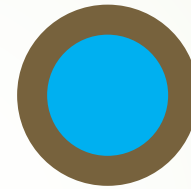
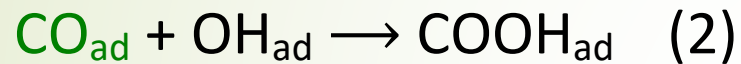
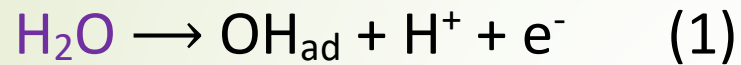
**$\text{Mo}_2\text{C}$**  synthesized by the carbothermal route appears as one of the most active **non-noble material** toward the **HER**





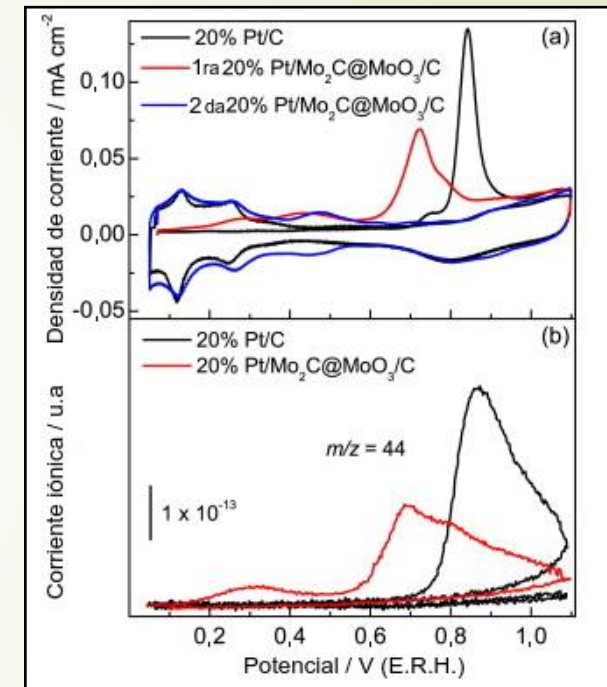
# TMCs as Electrocatalyst Support: Electrooxidation of Adsorbed CO on 20 wt.% Pt/Mo<sub>2</sub>C

## Reaction mechanism



- Bifunctional and electronic effects (CO bond strength) enhance the CO tolerance.
- It depends on the nature of the metal and on the surface structure.

## DEMS



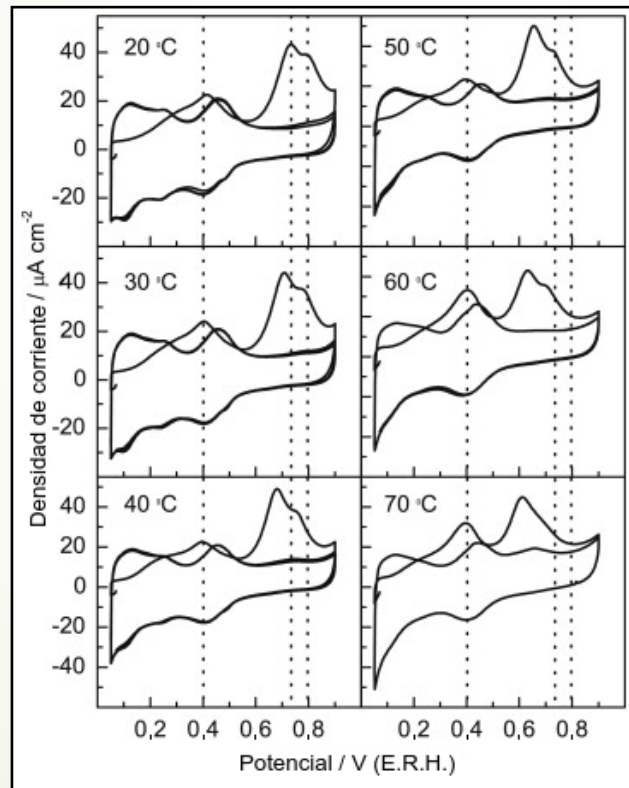
CO oxidation at 0.1 V @ Mo<sub>2</sub>C-based catalyst



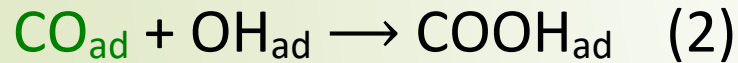
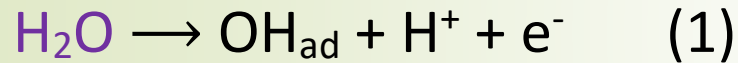


# TMCs as Electrocatalyst Support: Electrooxidation of Adsorbed CO on 20 wt.% Pt/Mo<sub>2</sub>C

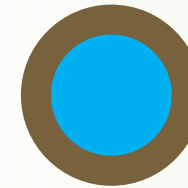
## Temperature Effect



## Reaction mechanism



Peak at low potentials increases while the peak at high potentials decreases



CO **diffuses** towards the most active site

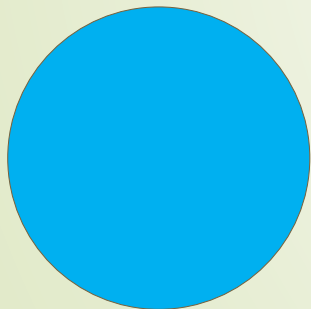
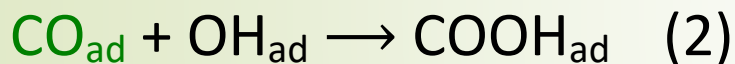
Several linked active sites





# TMCs as Electrocatalyst Support: Electrooxidation of Adsorbed CO on 20 wt.% Pt/TiC, Pt/TiCN and Pt/TiN

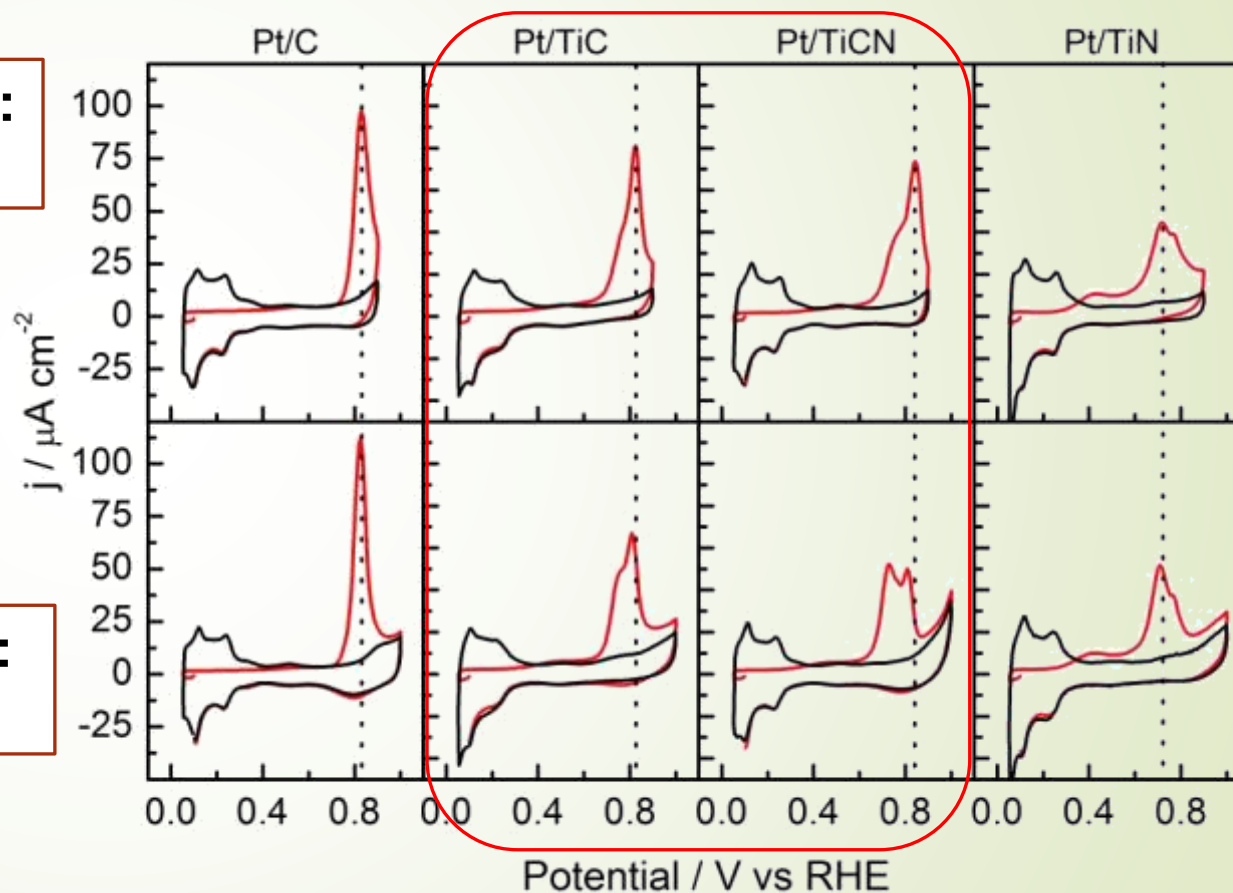
Reaction mechanism



Activation up to:  
**A) 0.9 V**

Activation up to:  
**A) 1.0 V**

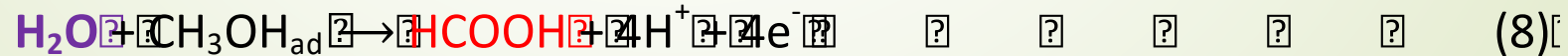
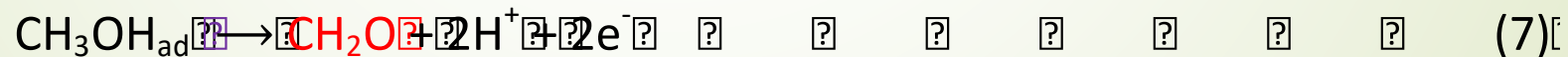
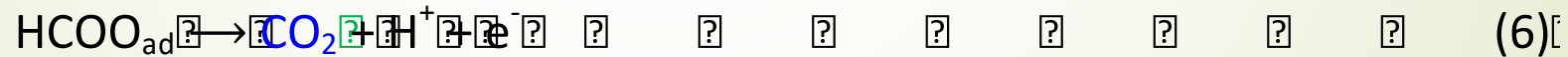
**CO tolerance increases at TiC and TiCN**





# TMCs as Electrocatalyst Support: Electrooxidation of Methanol on 20 wt.% Pt/TiC, Pt/TiCN and Pt/TiN

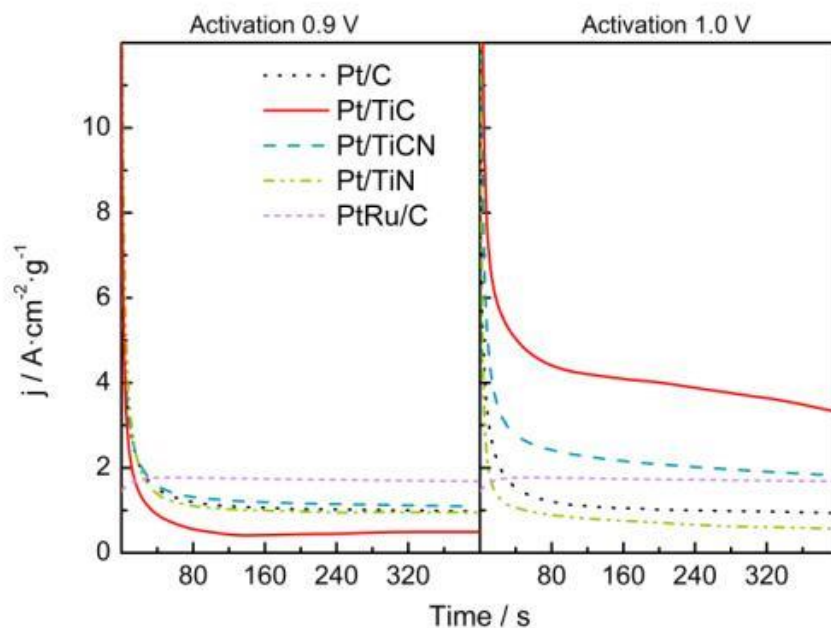
## Reaction mechanism



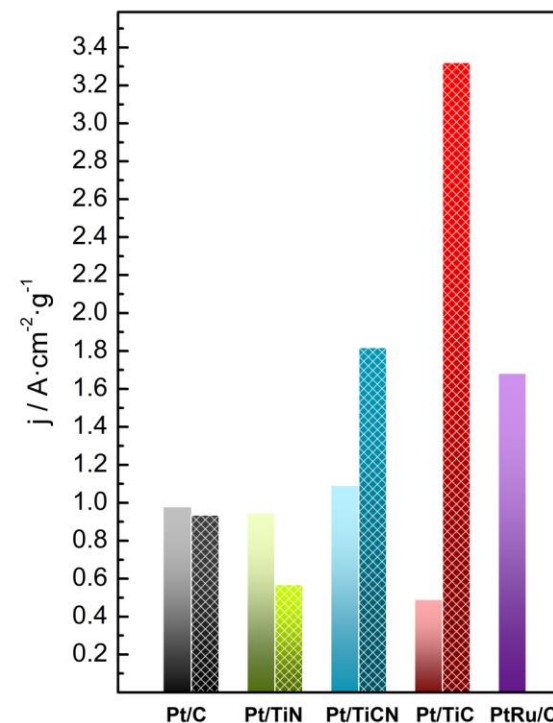


# TMCs as Electrocatalyst Support: Electrooxidation of Methanol on 20 wt.% Pt/TiC, Pt/TiCN and Pt/TiN

$E = 0.55 \text{ V}$ . 2 M Methanol + 0.5 M  $\text{H}_2\text{SO}_4$



Bifunctional and Electronic effects enhance the MOR.

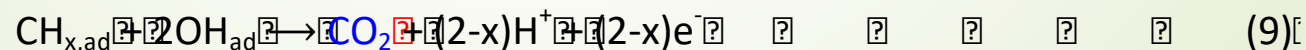
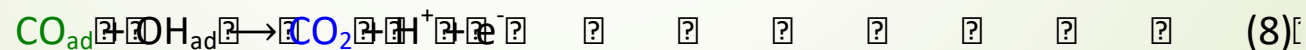
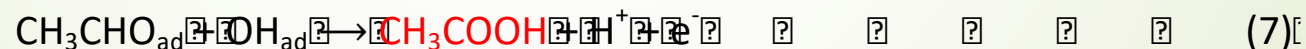
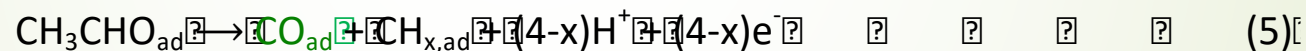
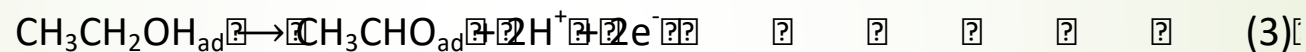


**Solid color: up to 0.9 V.**  
**Stripped color: up to 1.0 V.**  
**PtRu/C activated up to 0.8 V.**



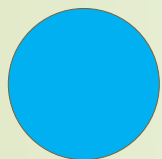
# TMCs as Electrocatalyst Support: Electrooxidation of Ethanol on 16 wt.% Pt(3)Au(3)Sn(10)/W<sub>2</sub>C and Pt(3)Au(3)Sn(10)/C

## Reaction mechanism

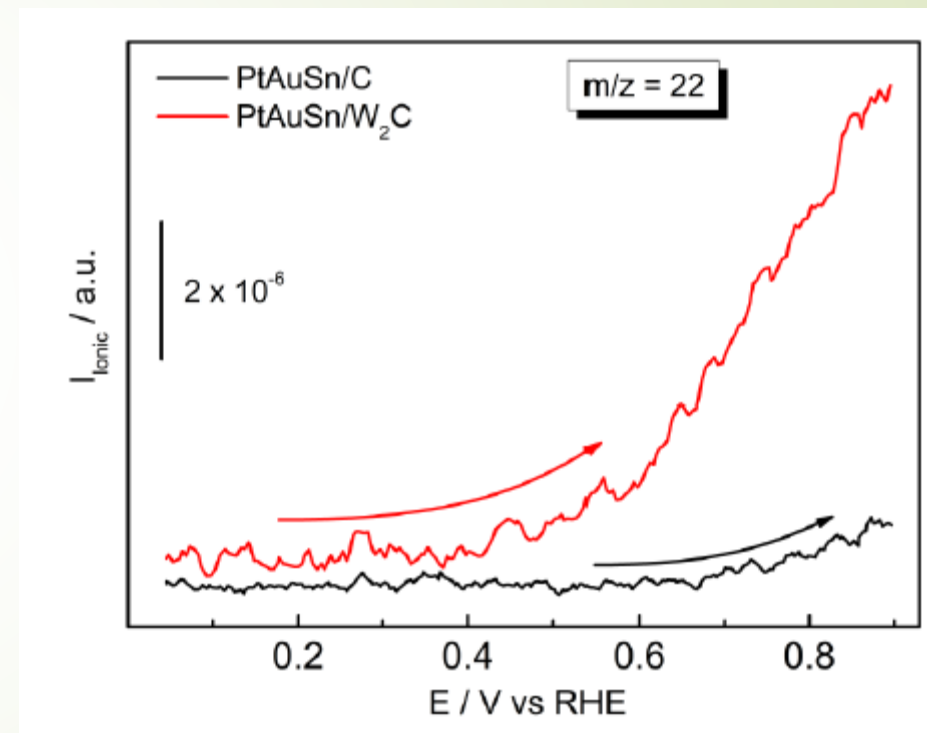
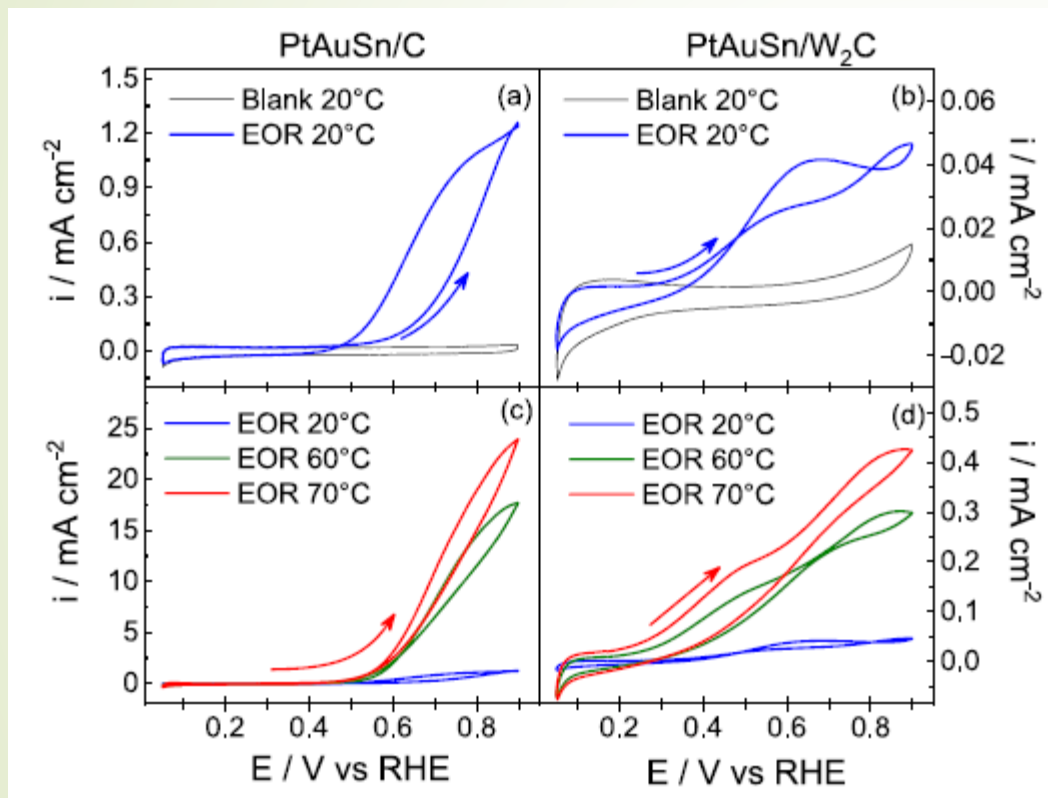




# TMCs as Electrocatalyst Support: Electrooxidation of Ethanol on 16 wt.% Pt(3)Au(3)Sn(10)/W<sub>2</sub>C and Pt(3)Au(3)Sn(10)/C



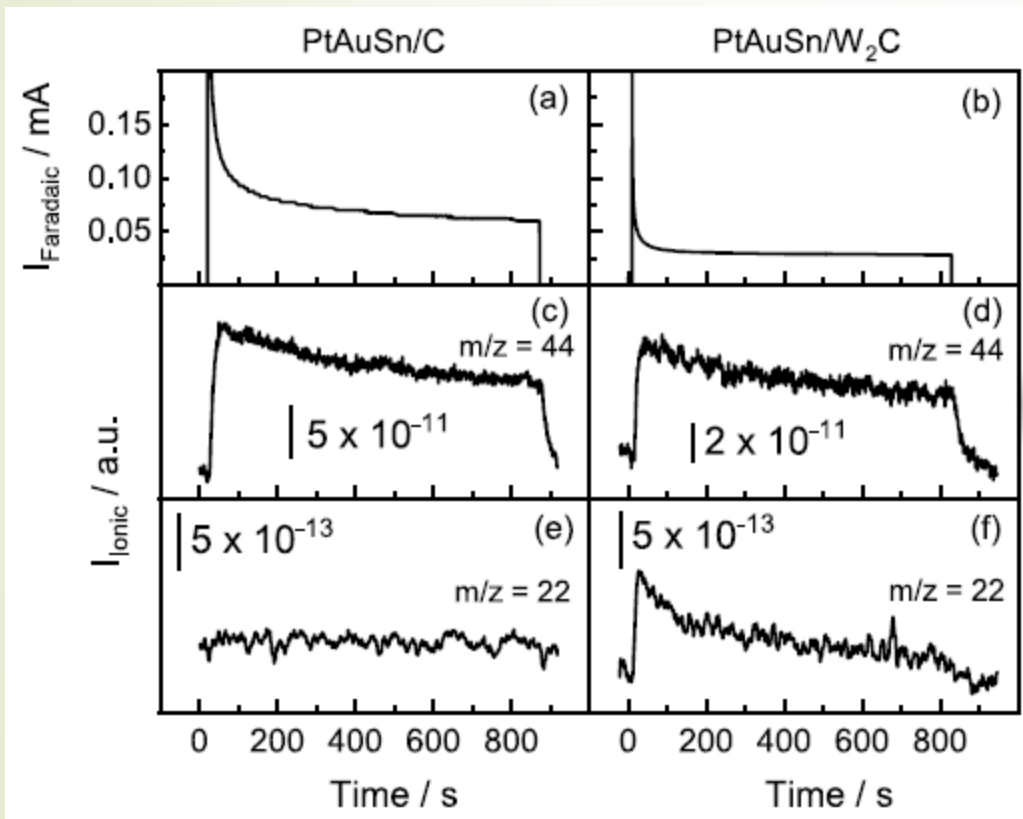
## DEMS





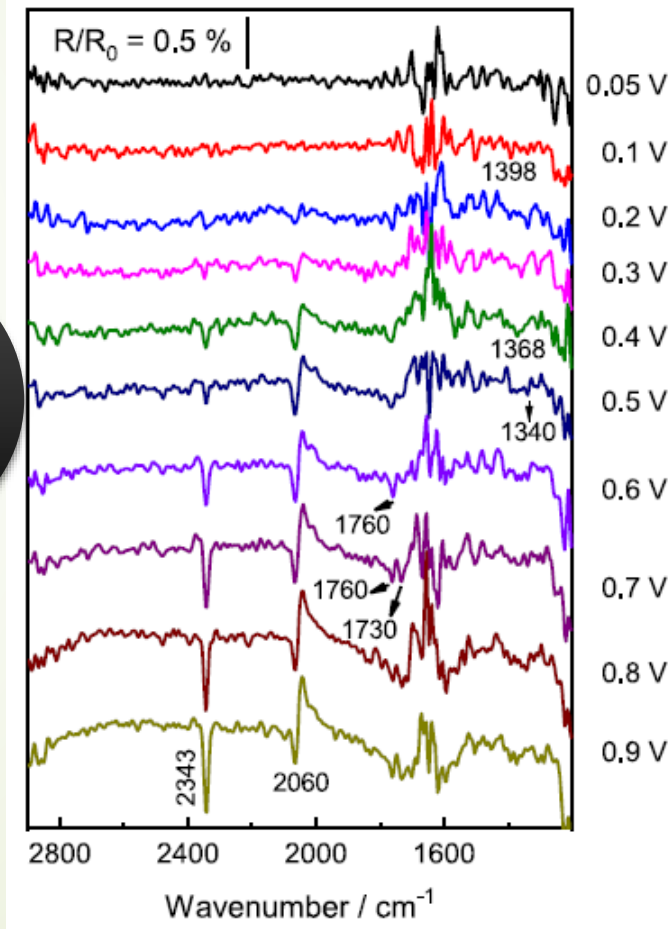
# TMCs as Electrocatalyst Support: Electrooxidation of Ethanol on 16 wt.% Pt(3)Au(3)Sn(10)/W<sub>2</sub>C and Pt(3)Au(3)Sn(10)/C

## Chronoamperometry @ 0.65 V



$E_f @ 0.65 \text{ V}$   
 $= 6.5 \%$

## FTIRS - PtAuSn/W<sub>2</sub>C

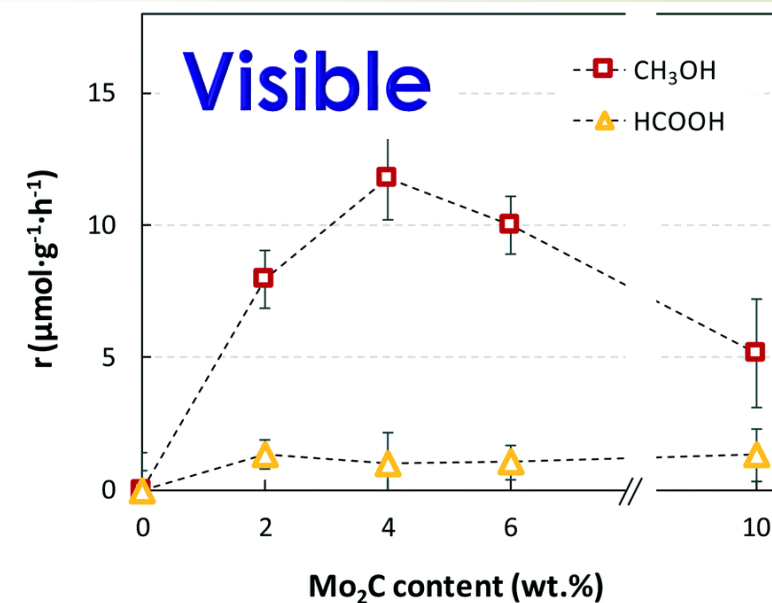
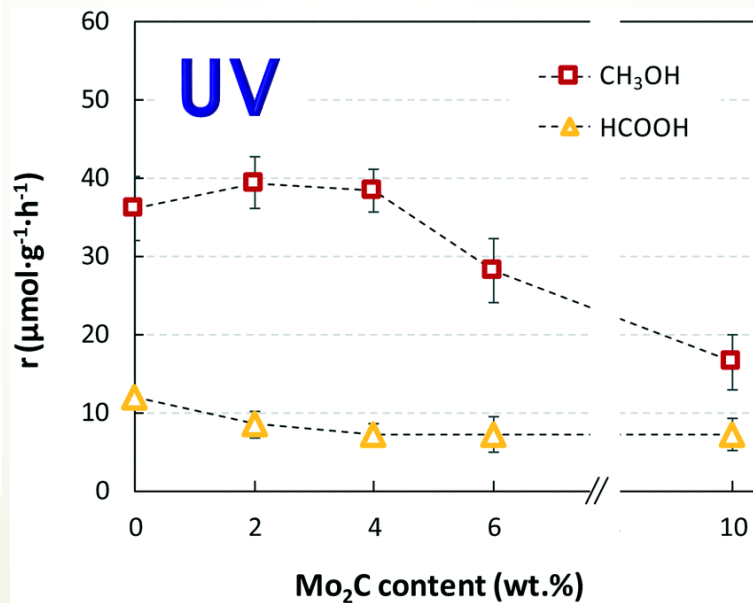
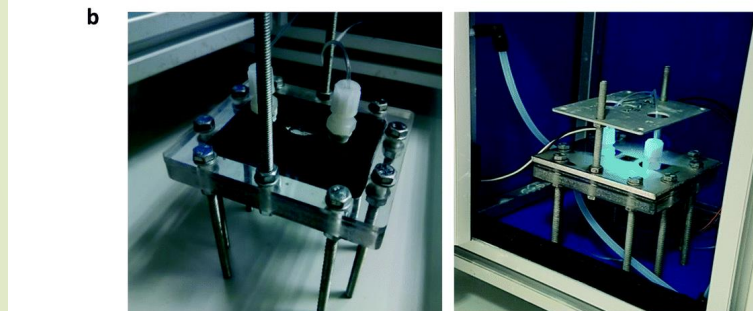
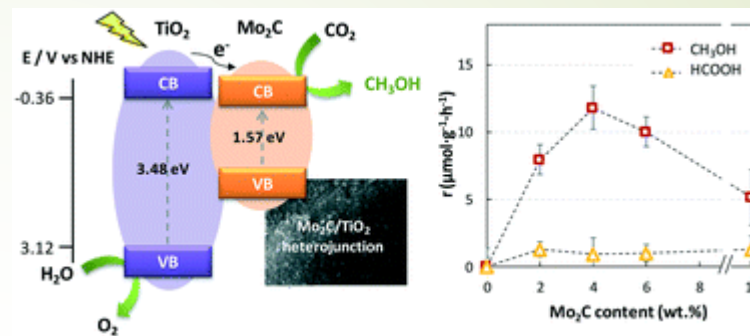
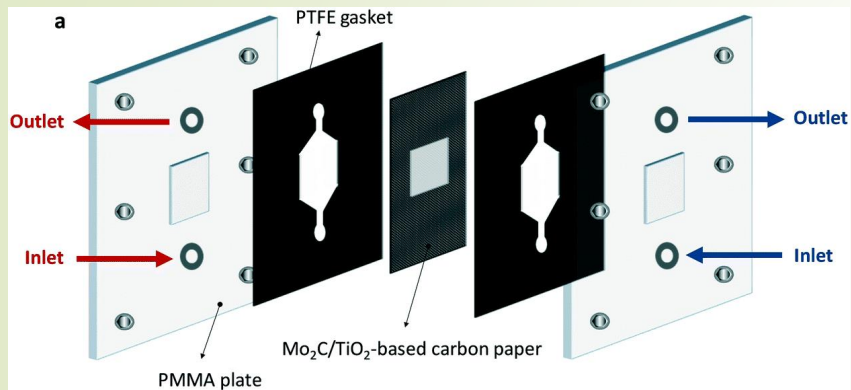
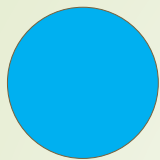


**W<sub>2</sub>C-Supported PtAuSn—A Catalyst with the Earliest Ethanol Oxidation Onset Potential and the Highest Ethanol Conversion Efficiency to CO<sub>2</sub> Known till Date**



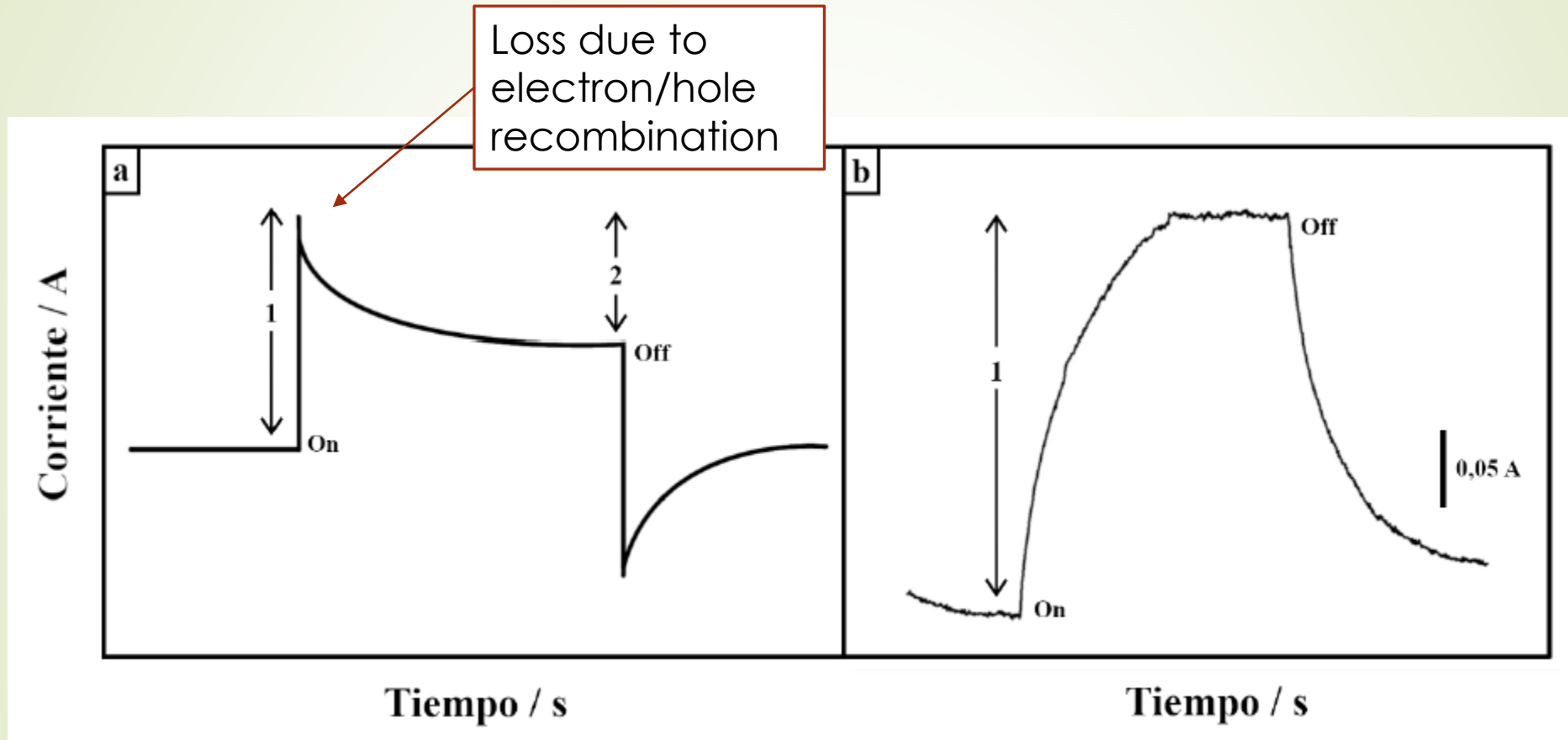


# TMCs as Photocatalyst: Photoreduction of CO<sub>2</sub> on Mo<sub>2</sub>C/TiO<sub>2</sub>





# TMCs as Photoelectrocatalyst

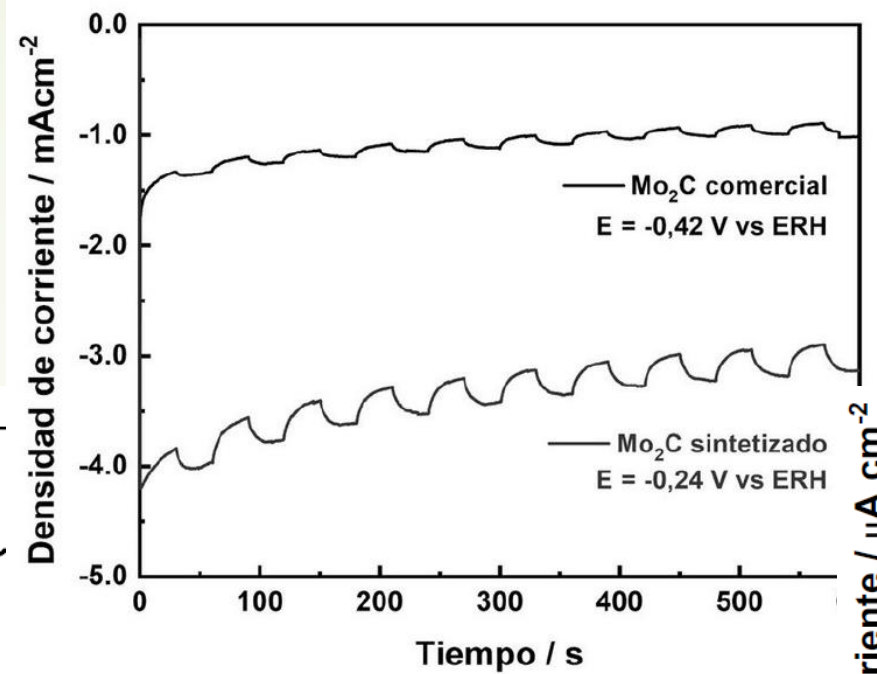




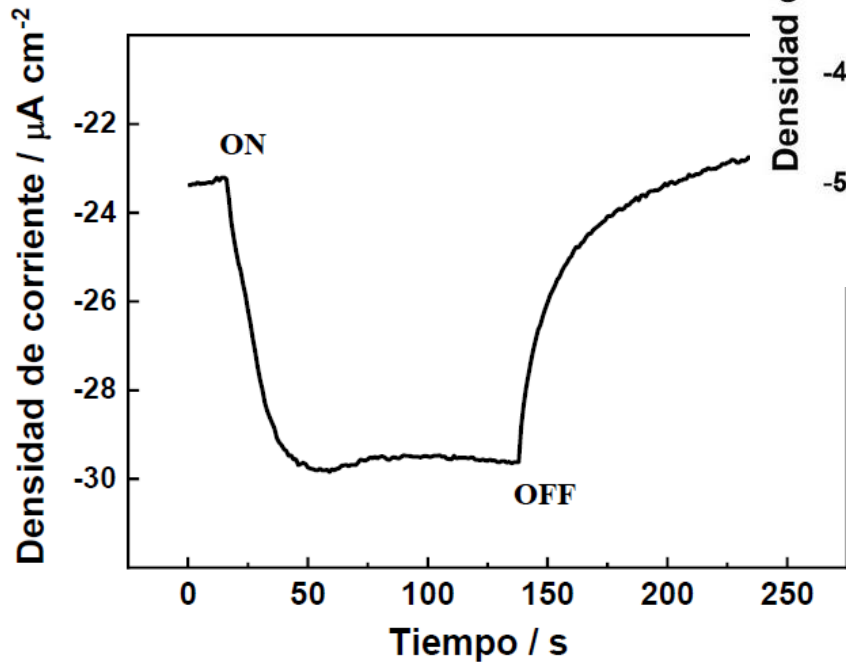
# TMCs as Photoelectrocatalyst



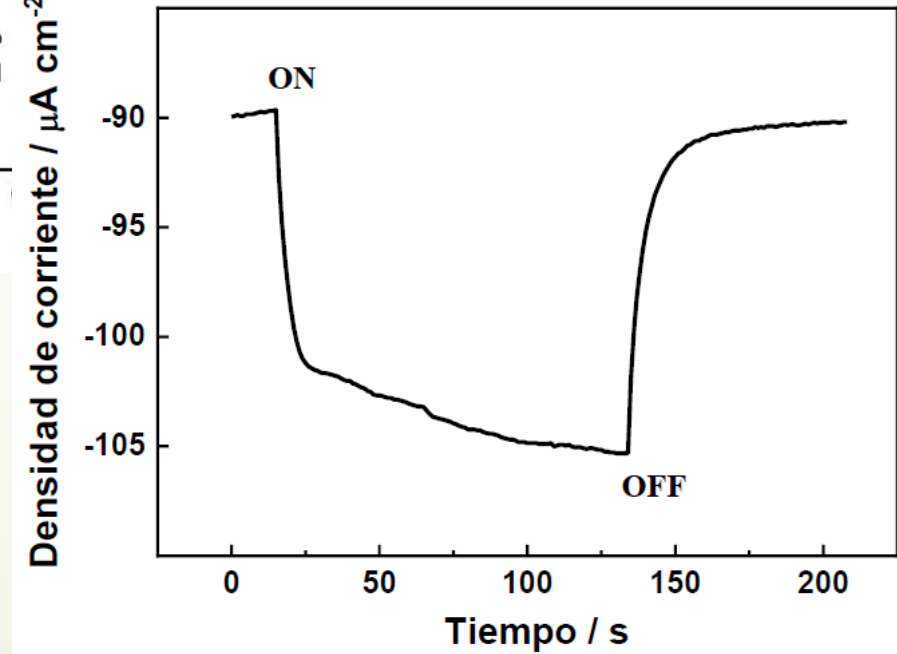
### HER / 0,1 M NaOH



### CO<sub>2</sub> / 0,5 M H<sub>2</sub>SO<sub>4</sub>



### KNO<sub>3</sub> / 0,5 M H<sub>2</sub>SO<sub>4</sub>

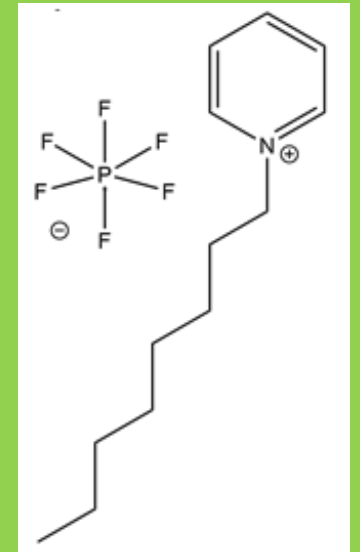
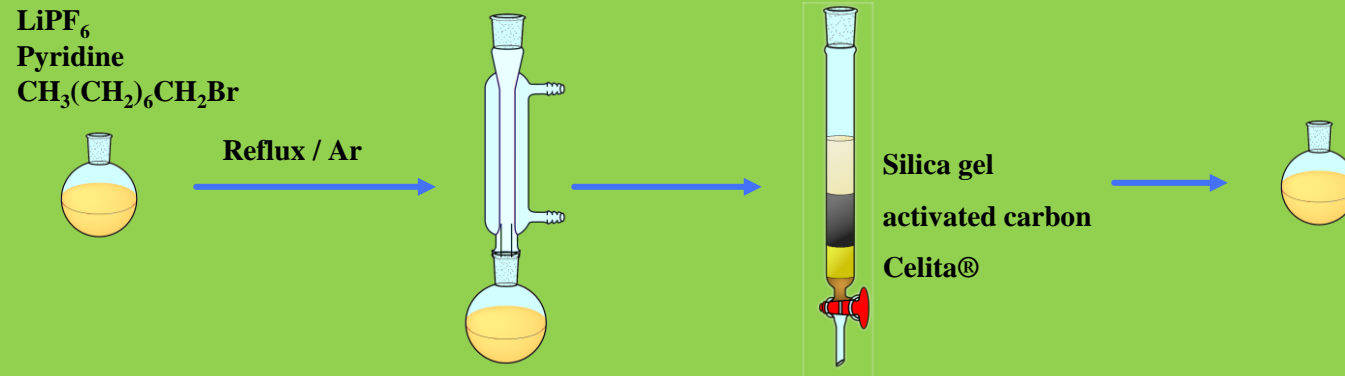




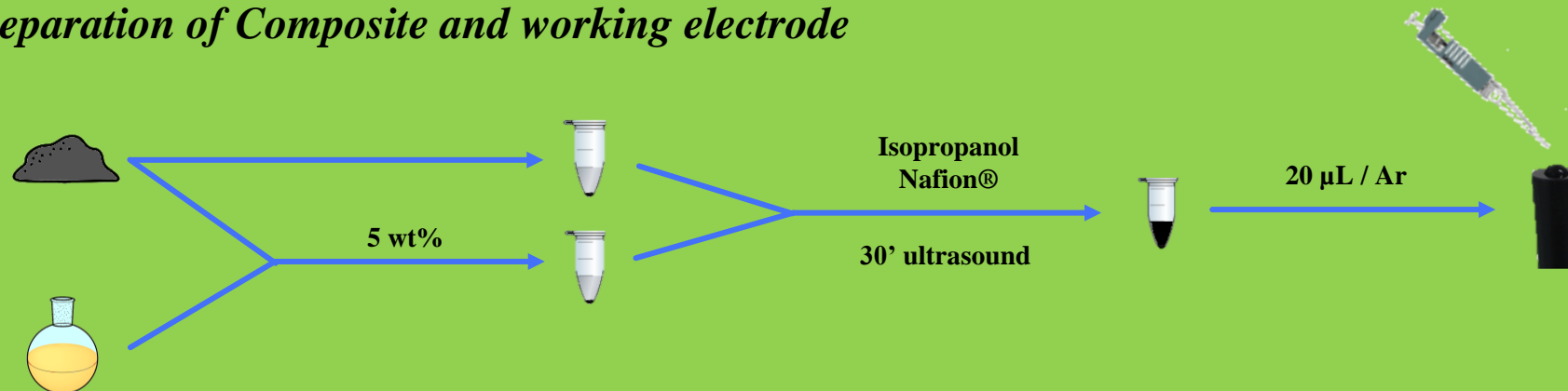
# Electrochemical Stability of TMC with ILs



## Synthesis of *N*-octylpyridinium hexafluorophosphate (OPy)

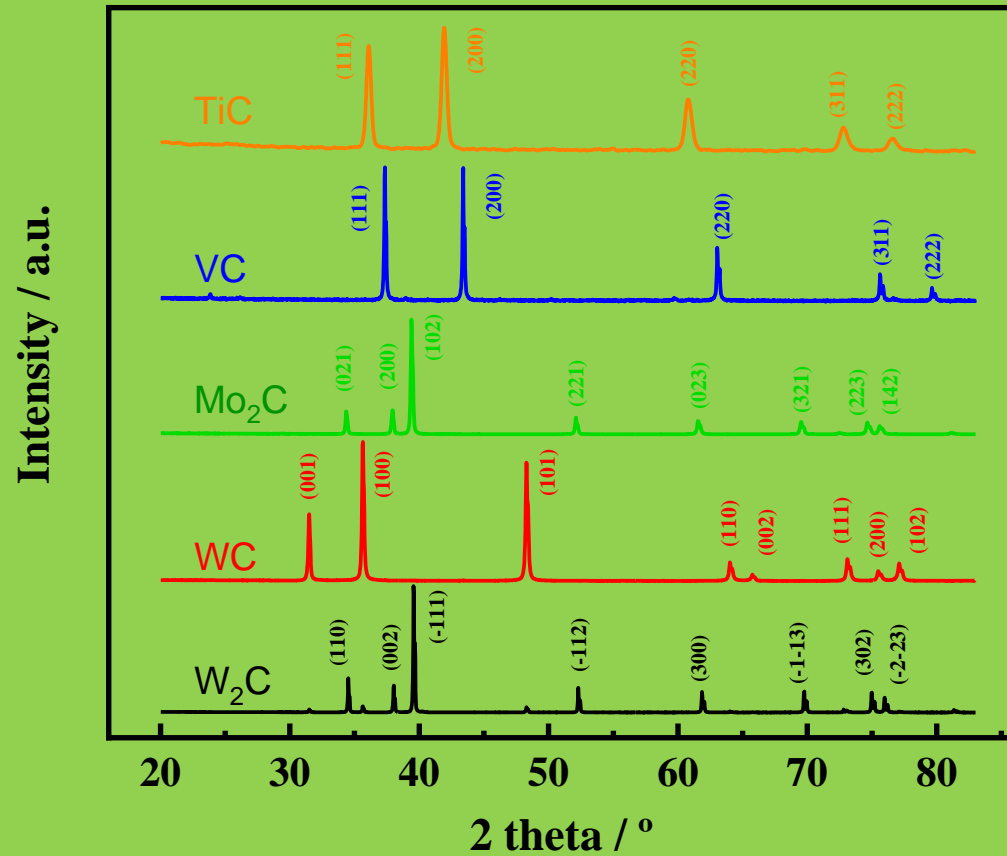


## Preparation of Composite and working electrode





# XRD and Raman





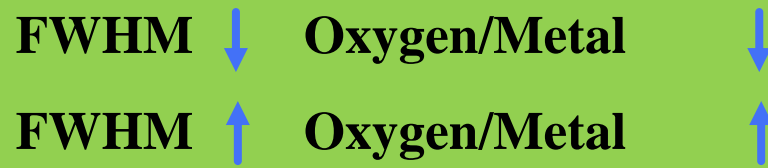
# XRD and Raman



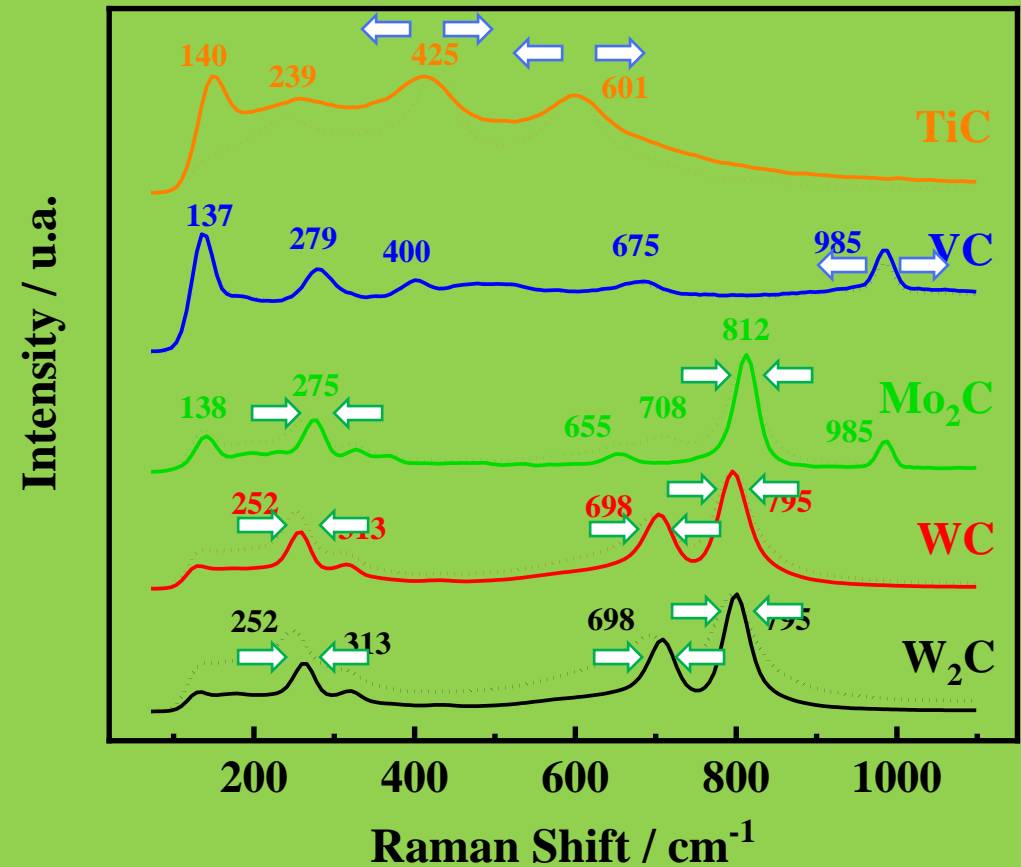
*Two different behaviors after OPY incorporation into the composite:*

**WC, W<sub>2</sub>C y Mo<sub>2</sub>C:** Raman shift towards higher values and Full Width at Half Maximum (FWHM) decreases.

**VC and TiC:** Raman shift towards lower values and FWHM rises.



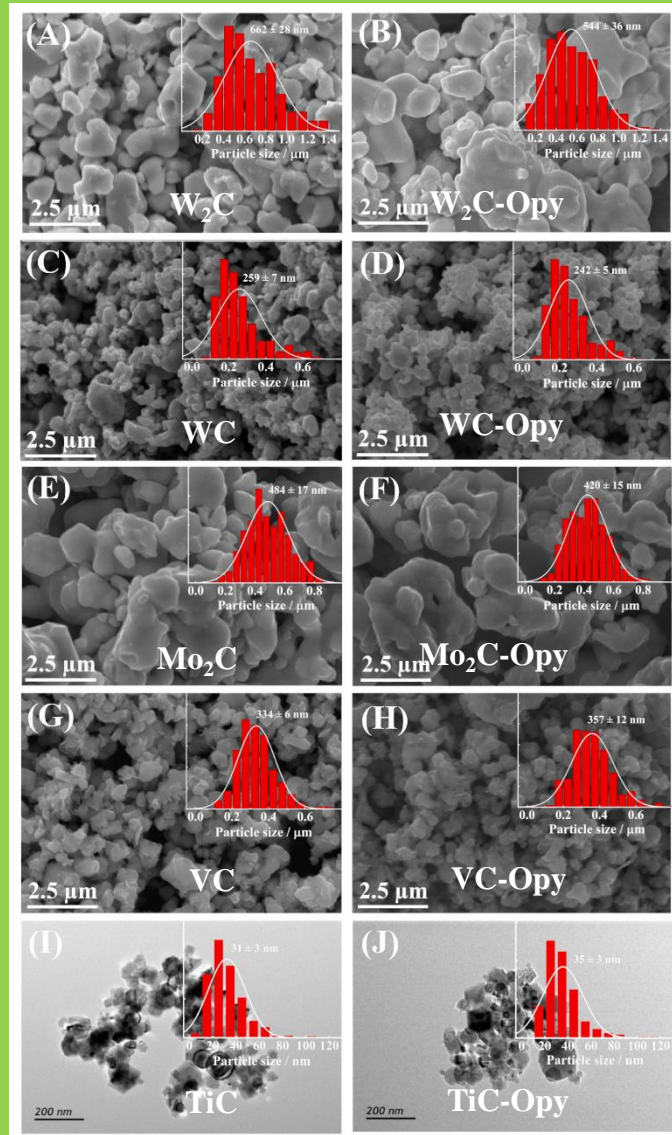
*Surface concentration of oxide species change with the OPY addition into the composite*



TMCs - - - -  
 Composites TM + OPy ————



# SEM



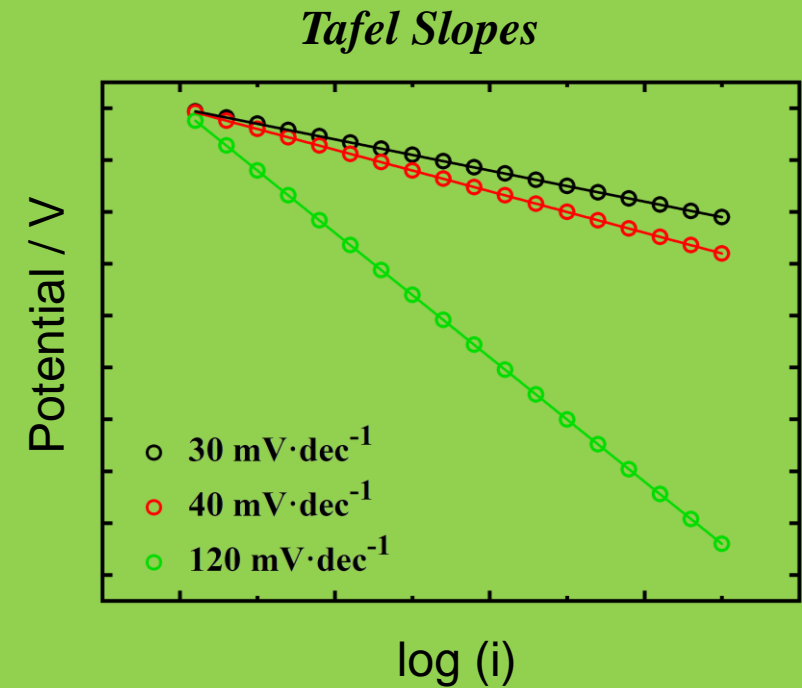
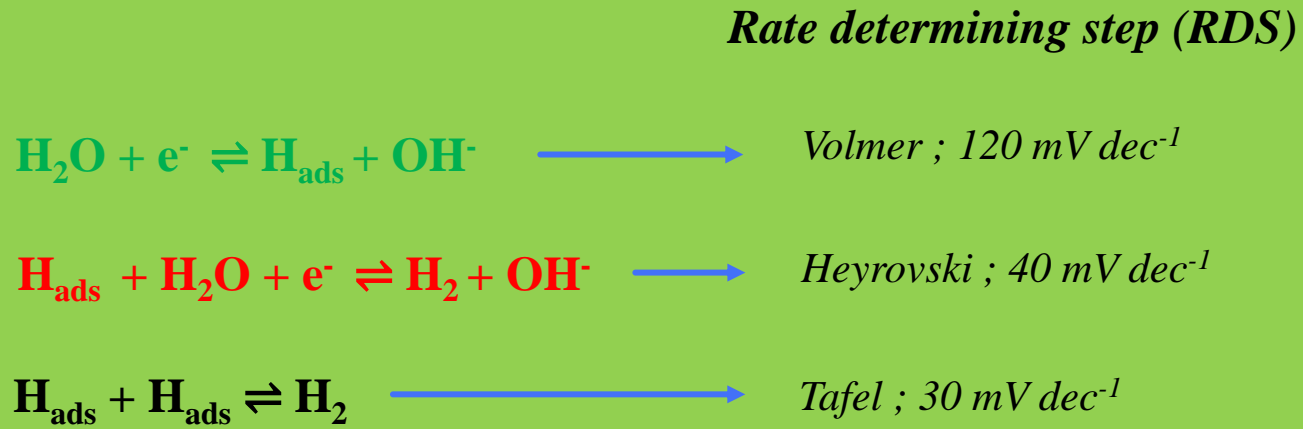
Sample	Particle size / nm	Variation of the Particle Size / nm
W <sub>2</sub> C	662	-118
W <sub>2</sub> C-OPy	544	
WC	259	-17
WC-OPy	242	
Mo <sub>2</sub> C	484	-64
Mo <sub>2</sub> C-OPy	420	
VC	334	23
VC-OPy	357	
TiC	31	4
TiC-OPy	35	

• WC, W<sub>2</sub>C y Mo<sub>2</sub>C. Particle size ↓

• VC y TiC. Particle size ↑



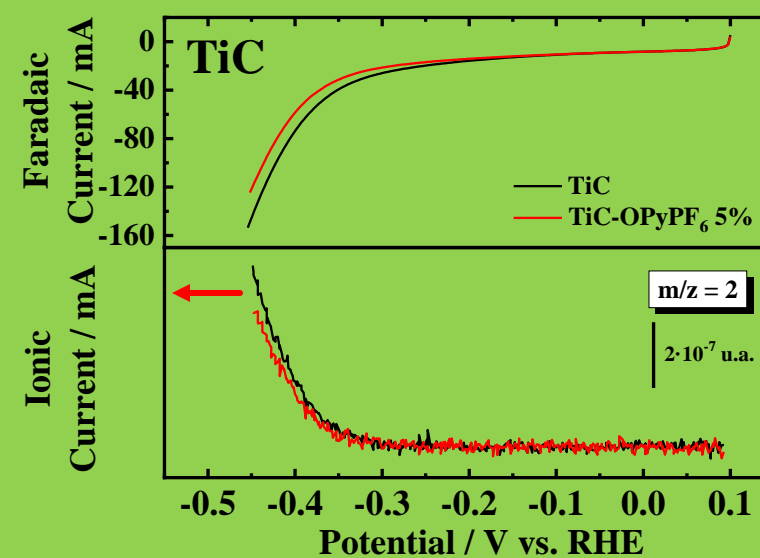
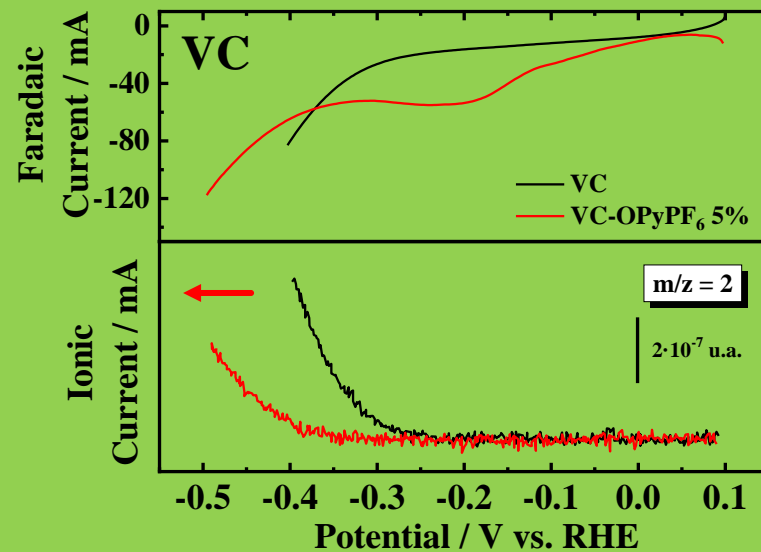
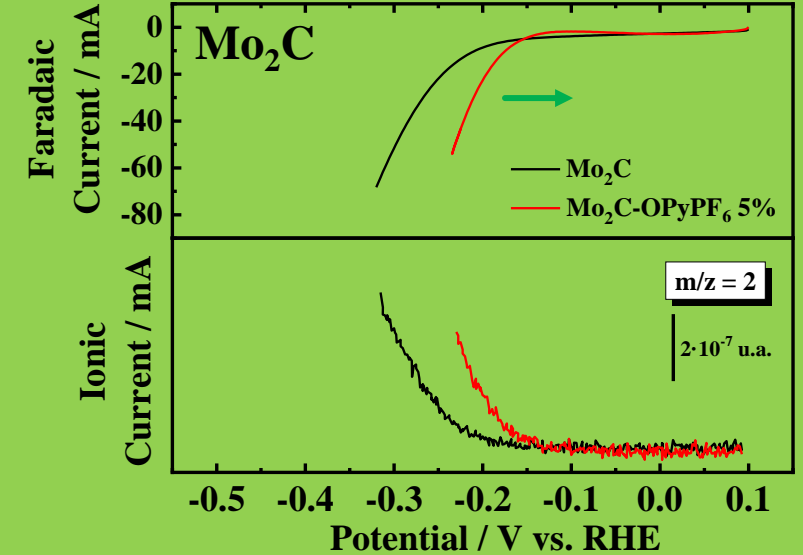
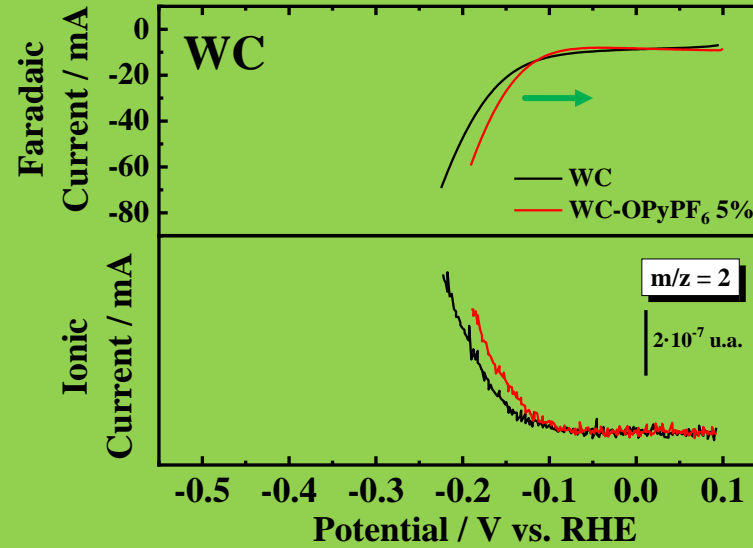
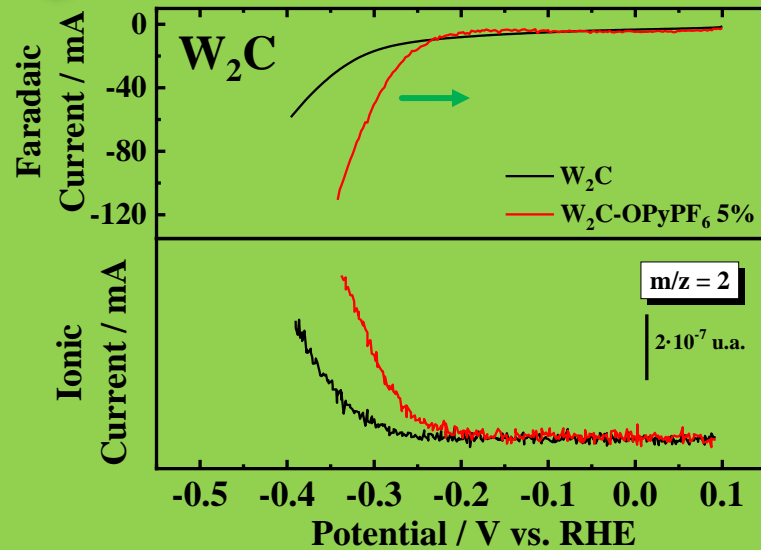
## Reaction mechanism of the HER





# DEMS

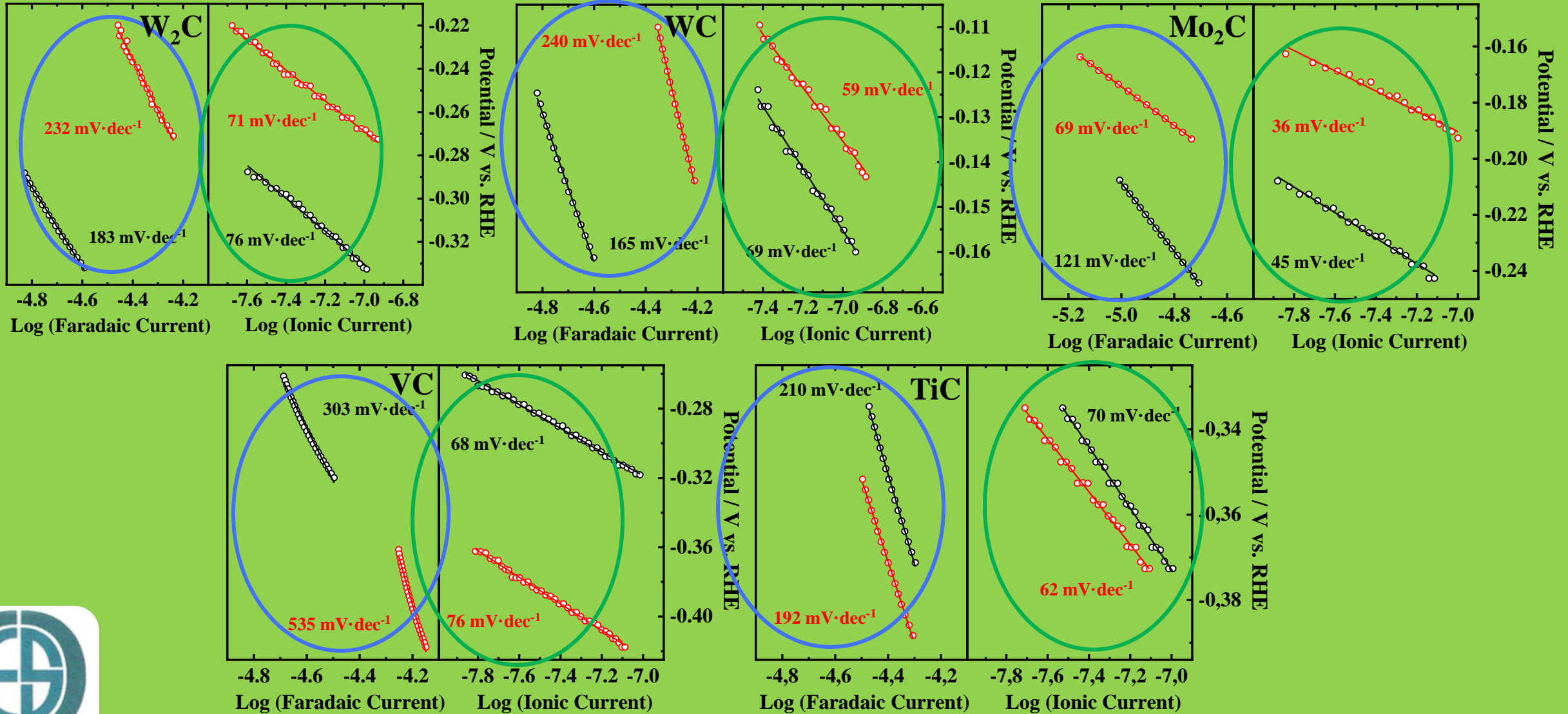
## TMCs – Composite (TMC + OPY)





# Tafel Slopes

TMCs – Composite (TMC + OPY)





# Summarizing

Sample	Particle size / nm	Variation of the Particle Size / nm	Onset Potential / mV	Shift of the Onset Potential/ mV	Tafel Slope / mV·dec <sup>-1</sup>
W <sub>2</sub> C	662	-118	-275	85	76
W <sub>2</sub> C-OPy	544		-190		71
WC	259		-17	-110	69
WC-OPy	242			-100	59
Mo <sub>2</sub> C	484		-64	-185	45
Mo <sub>2</sub> C-OPy	520			-145	36
VC	334	23	-260	-100	68
VC-OPy	357		-360		76
TiC	31	4	-334	0	70
TiC-OPy	35		-334		62

- Simple physical mixing of transition metal (Group VI) carbides and ionic liquids improves the HER.
- The ionic liquid acts as an inhibitor or promoter of oxide growth on the carbide surface.
- Raman spectroscopy and scanning electron microscopy reveal the effect of the ionic liquid on the surface oxide growth.



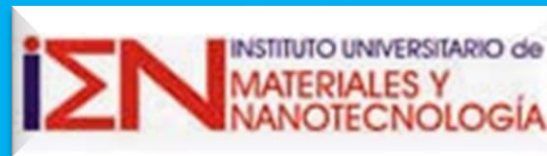


# Summarizing

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WC-OPy	242	-17	-100	10	69
Mo <sub>2</sub> C	484		-185	45	
Mo <sub>2</sub> C-OPy	520	-64	-145	40	36
VC	334		-260	68	
VC-OPy	357	23	-360	-100	76
TiC	31		-334	70	
TiC-OPy	35	4	-334	0	62

- Surface oxides inhibits the catalytic activity towards the HER.
- Differential Electrochemical Mass Spectrometry shows accurate onset potentials and Tafel slopes.





1<sup>ST</sup> LATAM ON GREEN AMMONIA  
POWER-to-X MEETING

# Acknowledgments

This work has been supported by the Ministerio de Ciencia e Innovación (MCIN) under projects PCI2020-112249 and PID2020-117586RB-I00 funded by MCIN/AEI/10.13039/501100011033, and by the Canarian Agency for Research, Innovation and Information Society (ACIISI, ProID2021010098). G.G. acknowledges NANOtec, INTech, Cabildo de Tenerife and ULL for laboratory facilities.

