Mesoporous Carbon-Based PGM-Free Catalyst Cathodes

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Project ID: FC303

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Overview

Timeline and Budget

- **Project Start Date**: October 1, 2018
- **Project End Date**: December 31, 2020
- **Percent complete**: 50%
- **Total Project Budget**: $1,430K
  - Total Recipient Share: $427K
  - Total Federal Share: $1,002K
  - Total DOE Funds Spent*: $474K

  * As of 3/01/2020

Barriers

A. Performance. Increase catalyst activity, improve the catalyst utilization, and facilitate the water dissipation to achieve the high-power density operation

B. Cost. Reduce the cost of PEM fuel cells using Precious Group Metal (PGM)-Free catalysts to replace PGM catalysts

C. Durability. Enhance the stability of PGM-free catalysts at relevant fuel cell operating conditions

Project lead

Indiana University Purdue University Indianapolis (IUPUI)

- PI: Jian Xie

IUPUI FULFILLING the PROMISE

Partners

University at Buffalo (UB) SUNY

- PI: Gang Wu

University at Buffalo The State University of New York

United Technologies Research Center (UTRC)

- PI: Zhiwei Yang

United Technologies Research Center

Electrocatalysis Consortium Members

ElectroCat Electrocatalysis Consortium; Argonne National Laboratory; NREL; Los Alamos National Laboratory; Oak Ridge National Laboratory

Relevance

- Technical Targets and Status

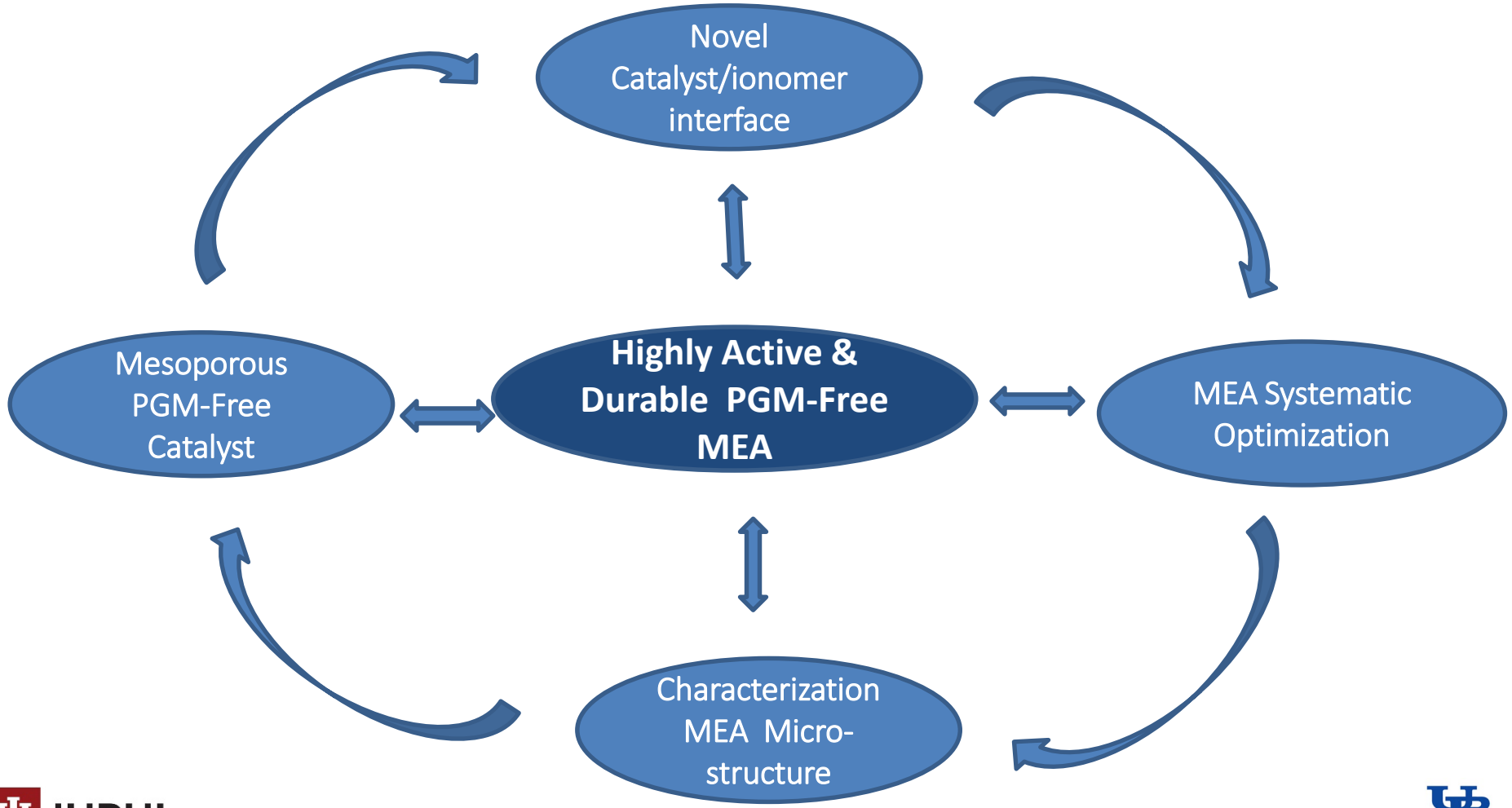
	Performance metrics	Unit	DOE Target (?)	Project Status	Y1 Milestone	Project Goal
Catalyst	Intrinsic activity ($E_{1/2}$)	V	>0.850	**0.830**	>0.820	0.850
	Stability (potential loss after 30K cycles(0.6-1.0V)), ($E_{1/2}$)	mV	< 30	**30**	< 30	20
MEA	Activity (H_2/O_2) @ 0.9 $V_{IR\text{-}free*}$	mA/cm^2	44	**33**	25	44
	Activity (H_2/air) @ 0.8 V*	mA/cm^2	150	**133**	100	150
	Peak Power density	mW/cm^2	500	**480**	300	500

* Backpressure: 150 Kpa abs

- Objectives
  - Design and develop hierarchically porous carbon sphere (HPCS)@M-N-C catalysts for PGM-free cathodes in PEMFCs through controllable synthesis to achieve
    - High density of accessible active sites
    - Hierarchy pore structure
  - Develop rationally designed ionomer/catalyst interface of PGM-Free catalyst membrane electrode assemblies (MEAs) to achieve
    - High catalyst utilization and high mass activity
    - High mass transport performance via systematically optimizing MEA structure
      - *Ink formulation*
      - *Ionomer effect*
      - *MEA fabrication process*

Approach (Overview)

- ❑ Develop hierarchically porous carbon sphere (HPCS) PGM-Free Catalysts.
- ❑ Rationally design ionomer/catalyst interfaces utilizing charge attraction.
- ❑ Systematically optimize MEA (ink formulation, ionomer, fabrication methods).



Approach/Milestone

Milestone 1. Catalyst Development (FY19-20) (Accomplished)

1.1 Scale up mesoporous synthesis > 5.0 g catalysts (100%)

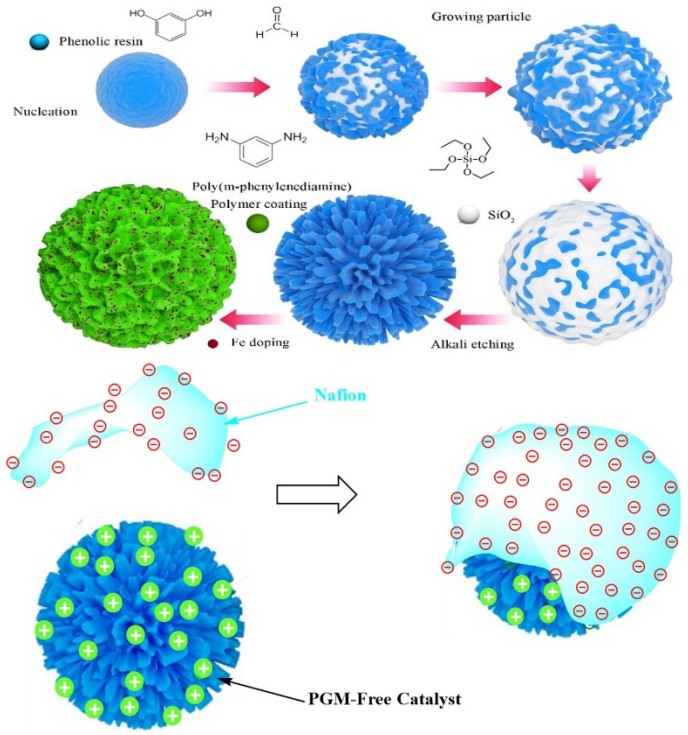
1.2 Achieve $E_{½}$ > 0.82 V and generate 0.25 mA/cm^2 at 0.90 V (100%)

1.3 Achieve $E_{½}$ > 0.85V, generate 0.50 mA/cm2 (at 0.90 $V_{IR\text{-}free}$) (100%)

(Go-No Go Decision Met)

Milestone 2. MEA Development (FY19-20) (Accomplished)

2.1 Achieve MEA : 25 mA/cm^2 (H_2/O_2, 0.90 $V_{IR\text{-}free}$, 150 KPa) (100%)



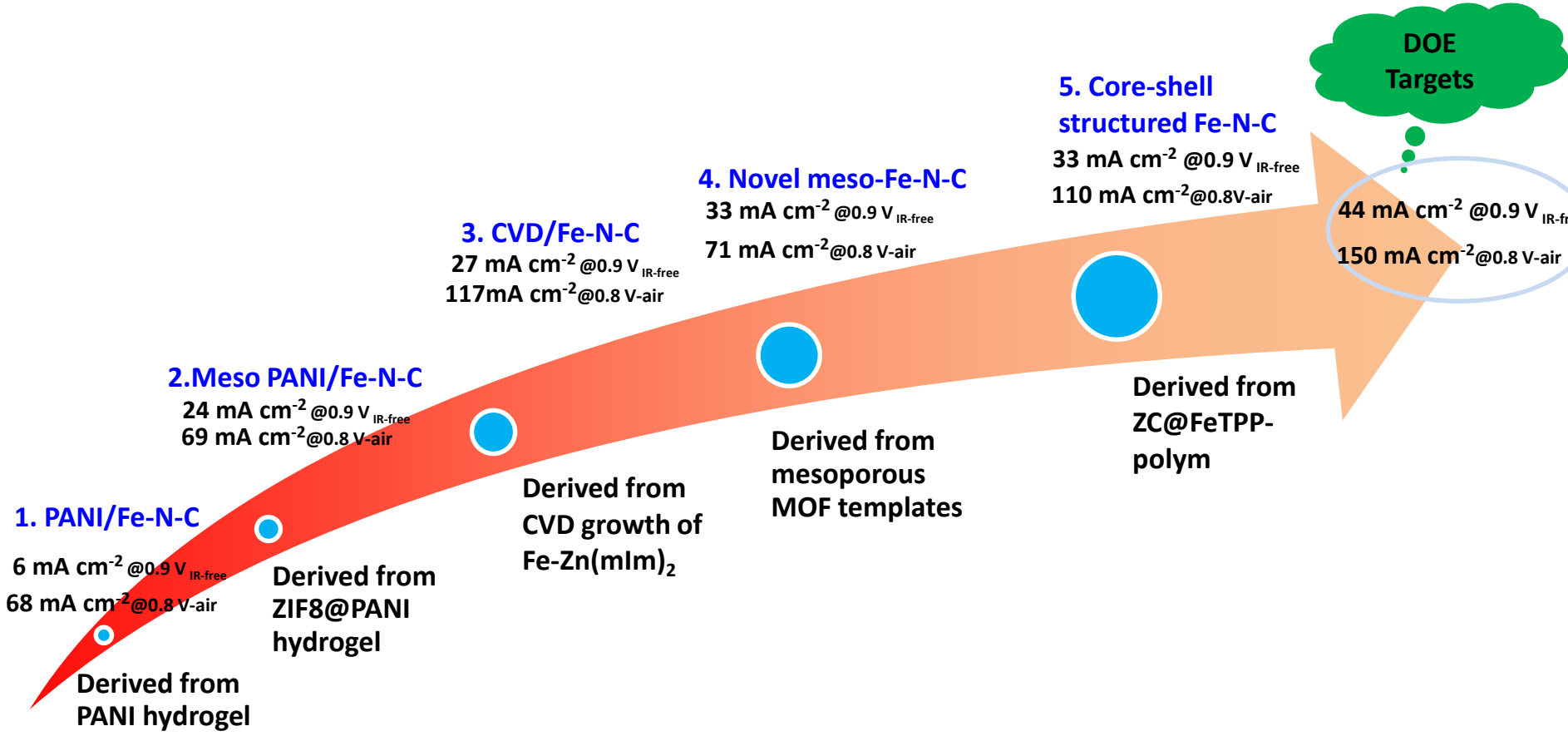
Synthesize HPCS Catalysts:

- ❑ Designing and synthesizing HPCSs *via* a template method and the further coating of the M-N-C electrocatalytic layer as the HPCS@M-N-C

Construct "ideal" ionomer/catalyst interface:

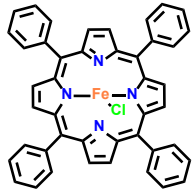
- ❑ Spontaneously forming ionomer/catalyst interface via charge attraction between catalyst ("+" charge} and ionomer ("-" charge) particles
- ❑ Controlling charge attraction resulting in **higher ionomer coverage (higher mass activity)** and **thinner ionomer film (lower O_2 diffusion barrier, higher high current density performance)**

Overview of Mesoporous Fe-N-C Catalyst Development



A variety of innovative Fe-N-C catalysts were developed by using template methods with continuously improving MEA performance, approaching DOE targets

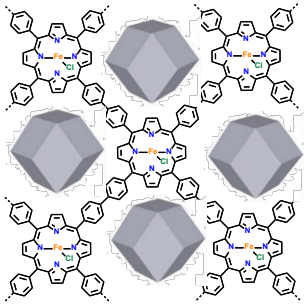
Approach 1: Synthesis of core-shell structured Fe-N-C Catalysts



FeTPP

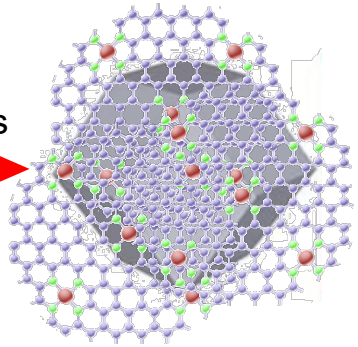
$AlCl_3$

ZIF-8-derived Carbon (ZC)

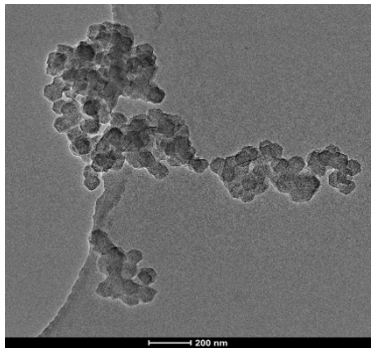


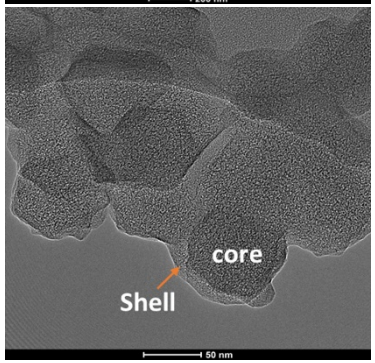
(CMP-Fe)@ZC

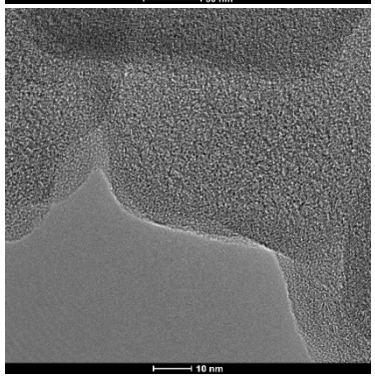
Pyrolysis



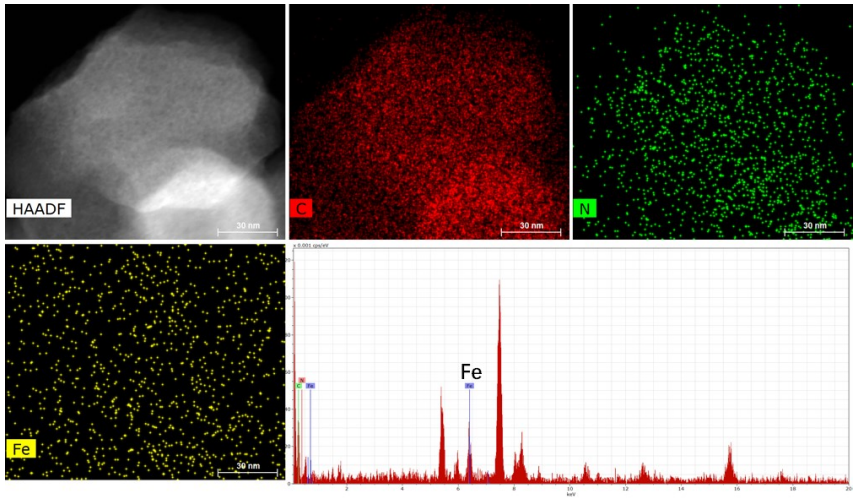
Core-shell Fe-N-C







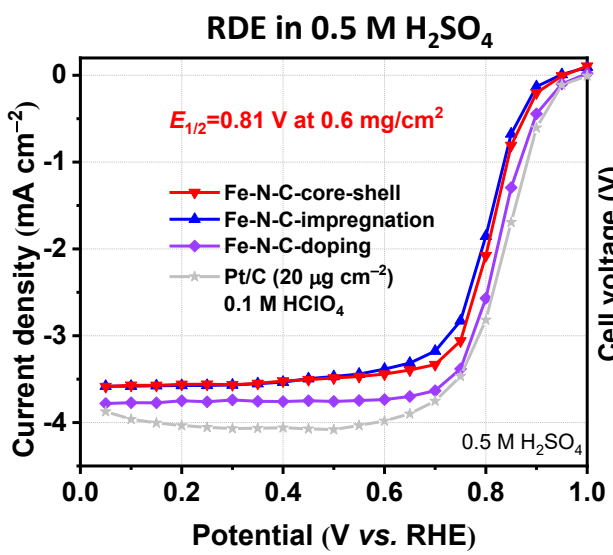
EDX Mapping and Spectrum

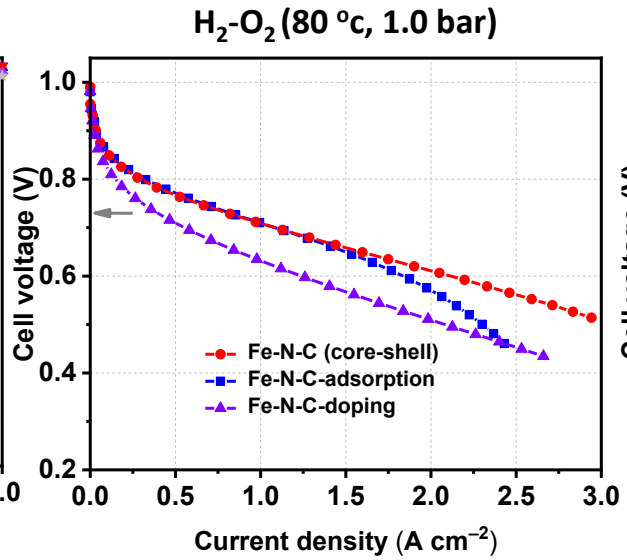


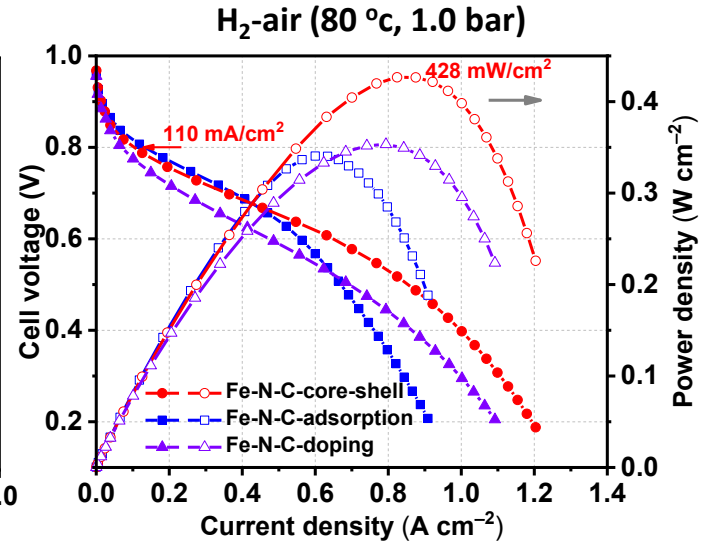
- **The polymer coating does not cause significant changes of morphology and size of carbon particles.**
- **As the core, the porous carbon framework derived from ZIF-8 can be encapsulated in the core-shell structure.**
- **After pyrolysis, the obtained core-shell structured Fe-N-C enhances the porosity of the carbon matrix, more defects and more exposed surface Fe active sites, which are all beneficial for MEA performane.**

High activity of active site; high surface area, micro/mesopores distribution

Innovative Core-shell structured Fe-N-C catalysts



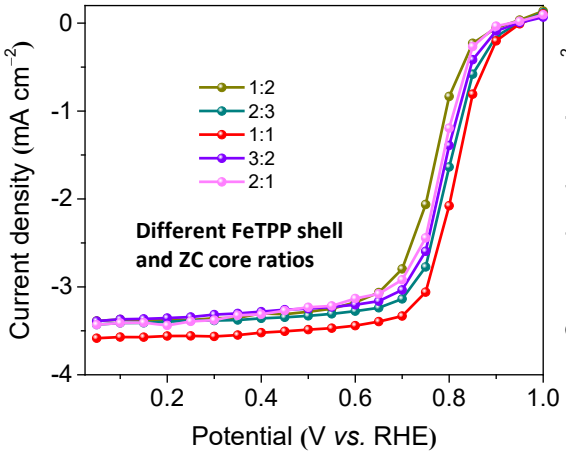


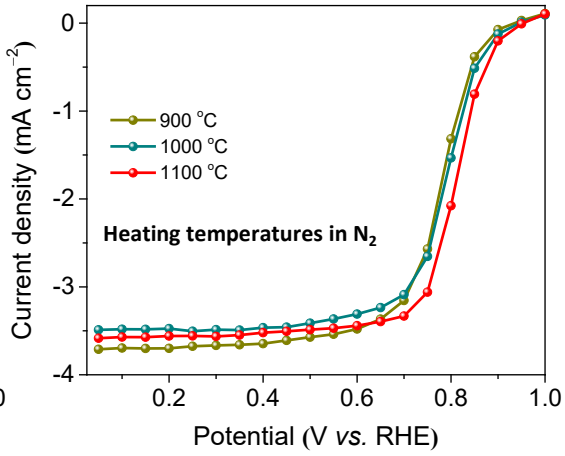


Catalysts	RDE ($E_{1/2,\ 0.6\ mg/cm^2}$)	H_2-O_2		H_2-air	
		j@0.9 V(mA cm^{-2})	Pmax (W cm^{-2})	*j*@0.8 V (mA cm^{-2})	Pmax (W cm^{-2})
Fe-N-C-core-shell	0.81	33	0.86	110	0.43
Fe-N-C-doping	0.83	21	0.70	72	0.35
Fe-N-C-adsorption	0.81	32	0.78	133	0.34

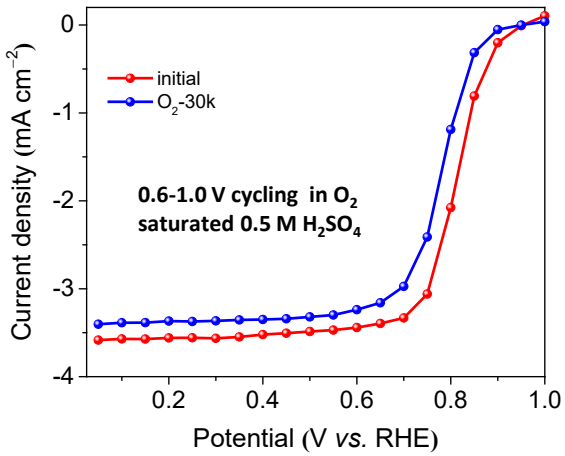
Core-shell structured Fe-N-C catalyst enriched Fe-N_4 sites at the surface enhanced mass transport and kinetic activity, related to other Fe-N-C catalysts from chemical doping and adsorption

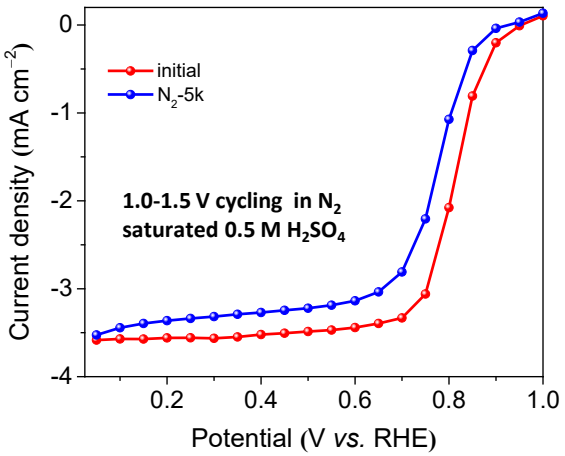
ORR activity and Stability of Core-Shell structured Catalysts

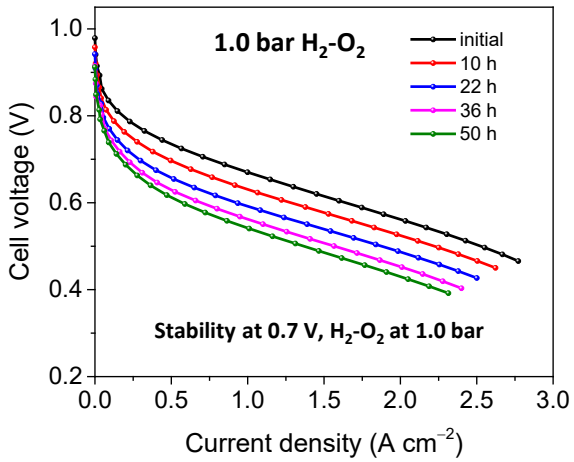




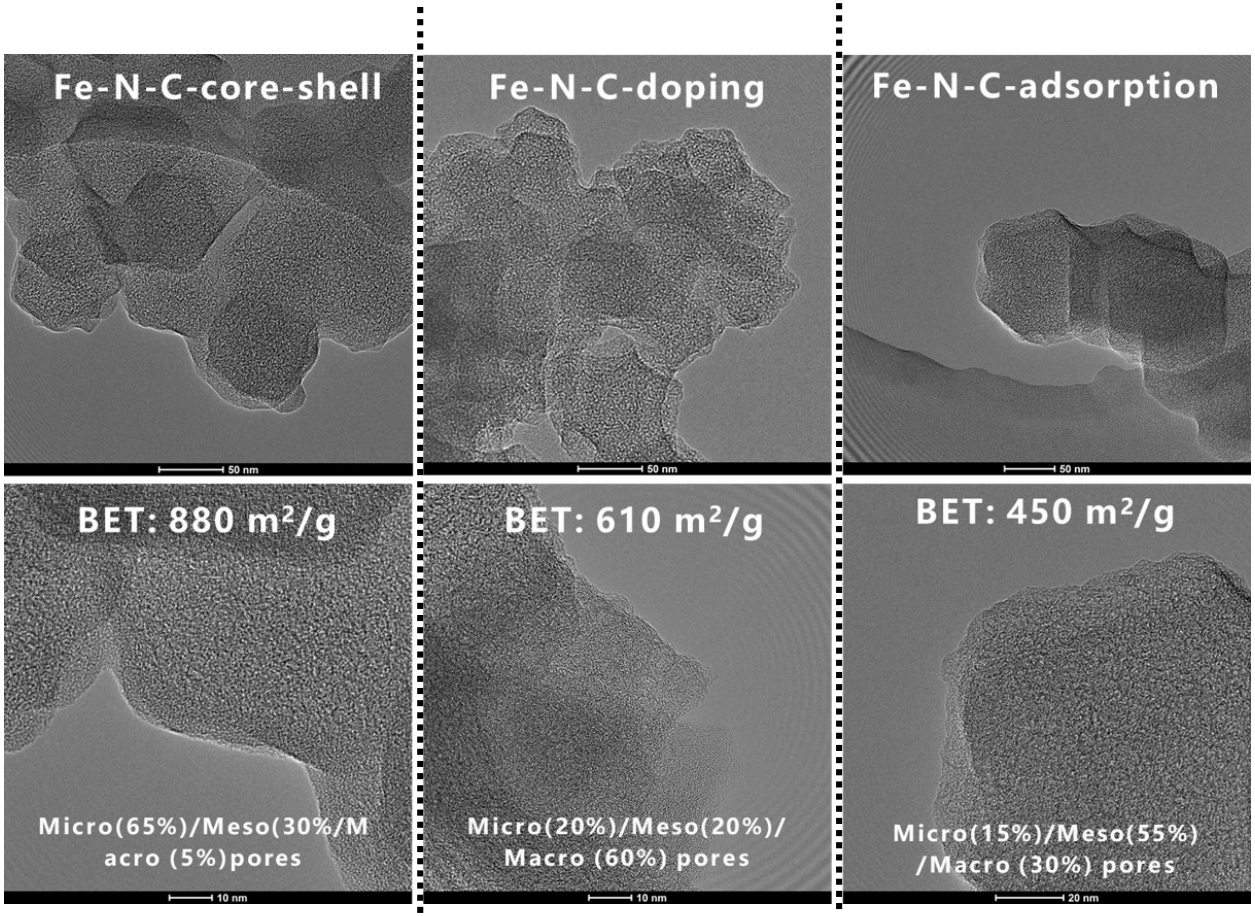
- During the synthesis, the ratios of core and shell and the heating temperatures are crucial for ORR activity.
- The core-shell structures exhibited reasonably good activity and carbon corrosion tolerance.
- Degradation occurs at 0.7 V under H_2-O_2

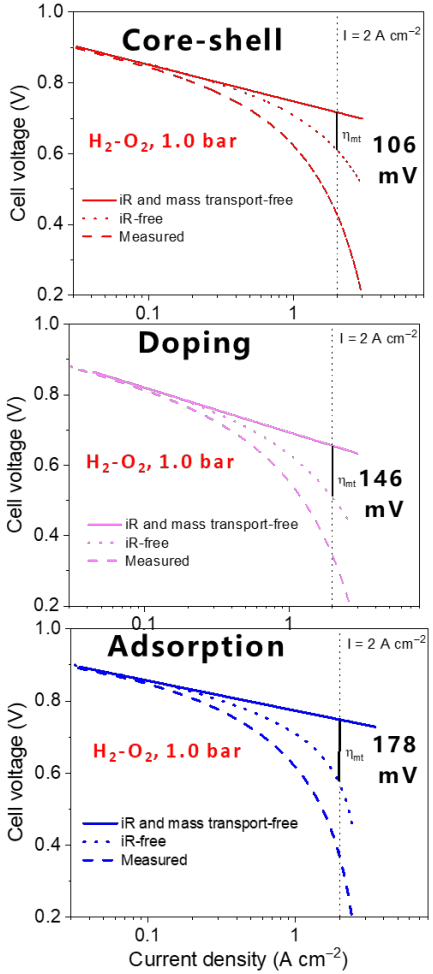






Comparison of Various Fe-N-C catalysts

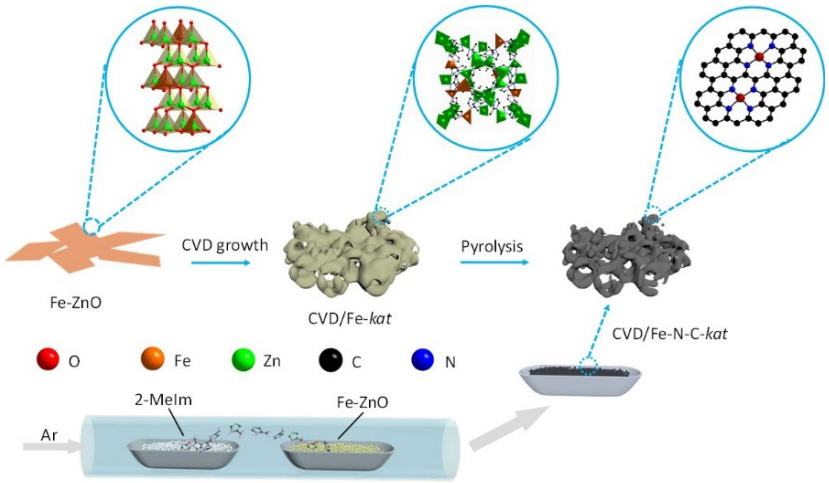




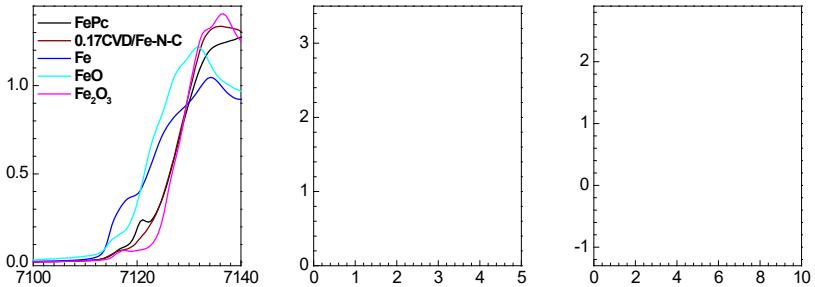
Core-shell structured Fe-N-C catalyst has optimal porosity to host dense active site and yield the most effective mass transport

Approach 2: Innovative CVD growth of mesoporous Fe-N-C catalyst

CVD synthesis methods

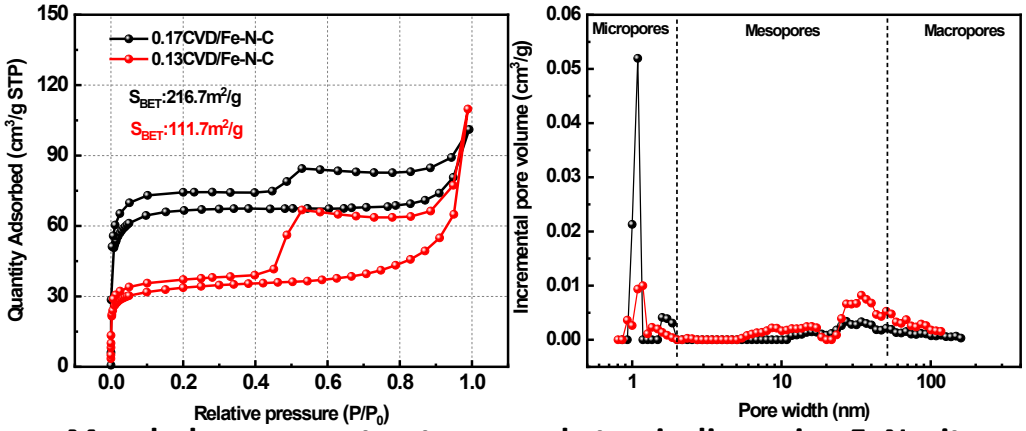


(1) Synthesis of Fe -doped ZnO nanosheets ; (2) CVD growth of Fe doped Zn(mim)$_2$ and pyrolysis to obtain atomically dispersed Fe-N-C catalyst

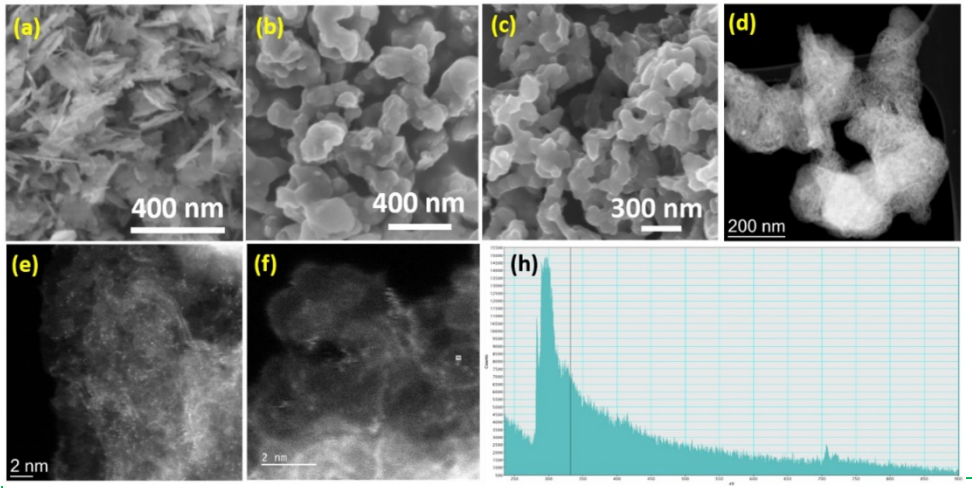


XAS analysis furtehr verify the atomically dispersed and nitrogen coordinated FeN$_4$ sites exclusively in the catalyst from CVD methods.

Mesopore feature



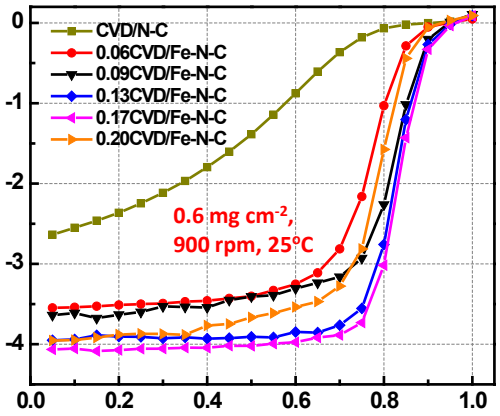
Morphology, mesostructures, and atomic dispersion FeN$_x$ sites



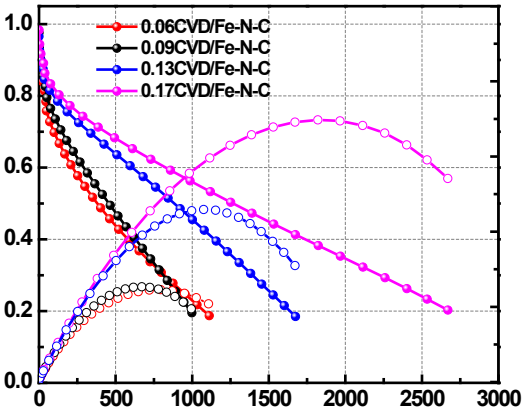
(a-c) Morphologies of (a) Fe-ZnO template, (b)Fe-Zn(mim)$_2$ precursors, and (c) the CVD/Fe-N-C catalyst;
(d-f) STEM images along with EEL point spectra confirm the single Fe site dispersion in the CVD/Fe-N-C catalyst

ORR activity and MEA performance of CVD Fe-N-C catalysts

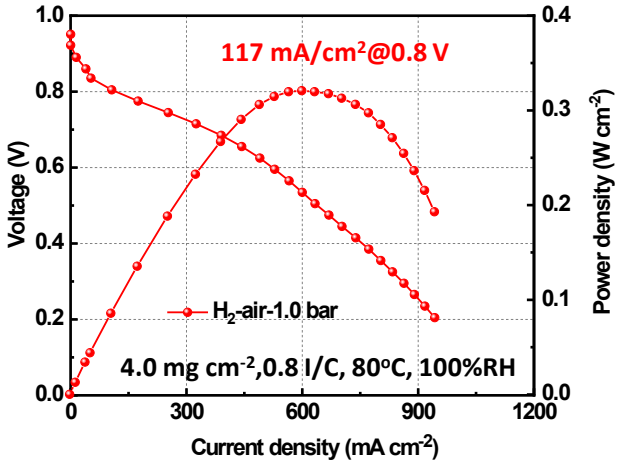
RDE in 0.5 M H_2SO_4

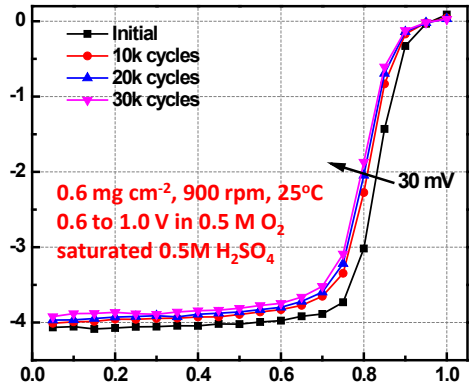


H_2-O_2, 1.0 bar



H_2-air 1.0 bar

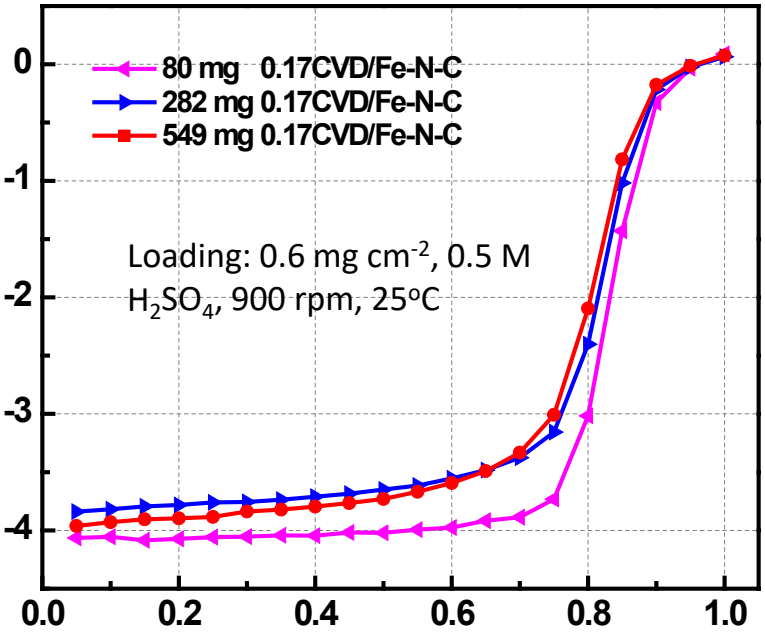


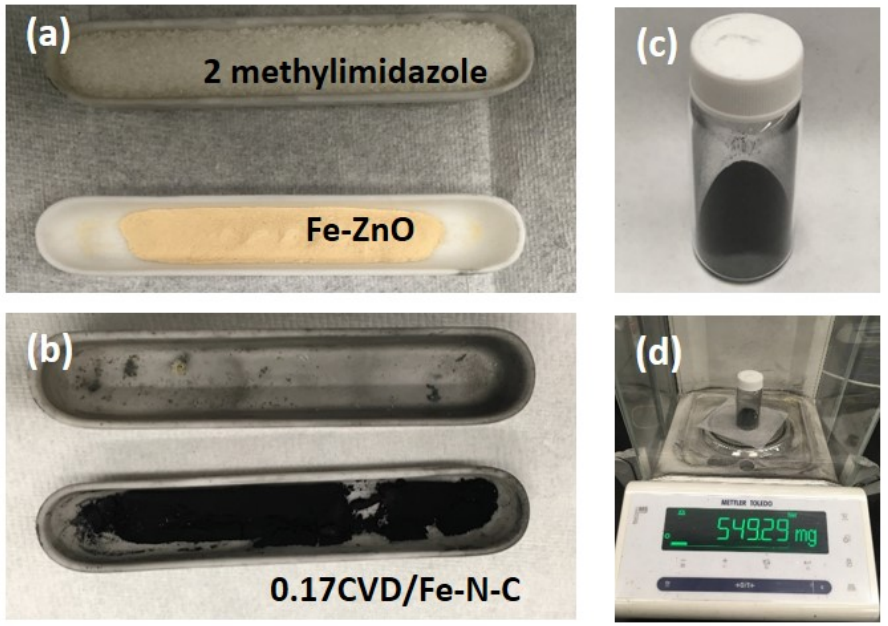


Catalysts with various Fe content	H_2-O_2	H_2-air
	j@0.9V,mA cm^{-2}	j@0.8V, mA cm^{-2}
0.17CVD/Fe-N-C	27	117
0.13CVD/Fe-N-C	13	50
0.09CVD/Fe-N-C	12	30
0.06CVD/Fe-N-C	6	20

high activity of active site; low surface area, micro/mesopores distribution

Scale-up Synthesis Study

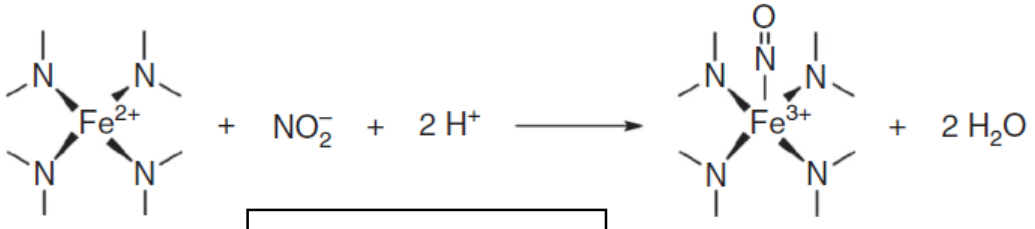




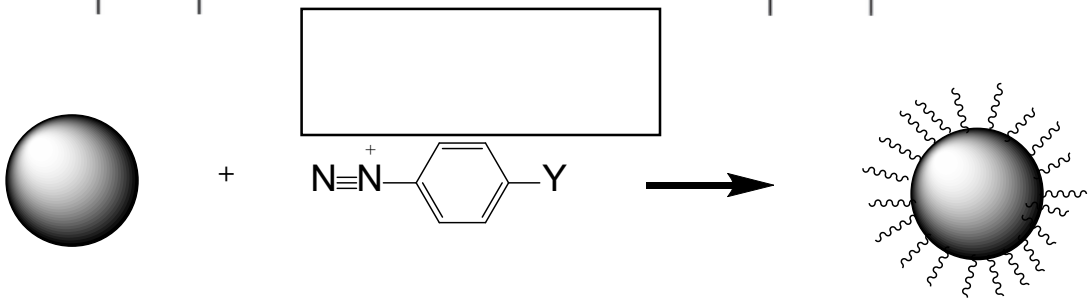
(a) Pictures of 2 methylimidazole and Fe-ZnO powder, **(b)** the obtained mesoporous CVD/Fe-N-C catalyst; **(c-d)** the vial loaded with ~ 0.55 g of the mesoporous 0.17 CVD/Fe-N-C catalyst

The newly developed CVD methods can synthesize atomically dispersed single Fe catalysts in a large scale with comparable ORR activity

Functionalize Catalyst with Positive Charge for Building Ionomer/catalyst Interface

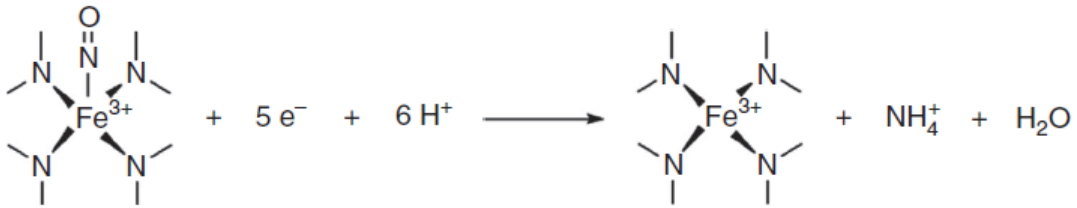


Masking the Fe-N_C active center via Chem Absorption with NO_2 (Oxidation)



Functionalization of catalyst via diazonium reaction

PGM-Free Catalyst | **Diazonium Salt** | **Functionalized Catalyst**

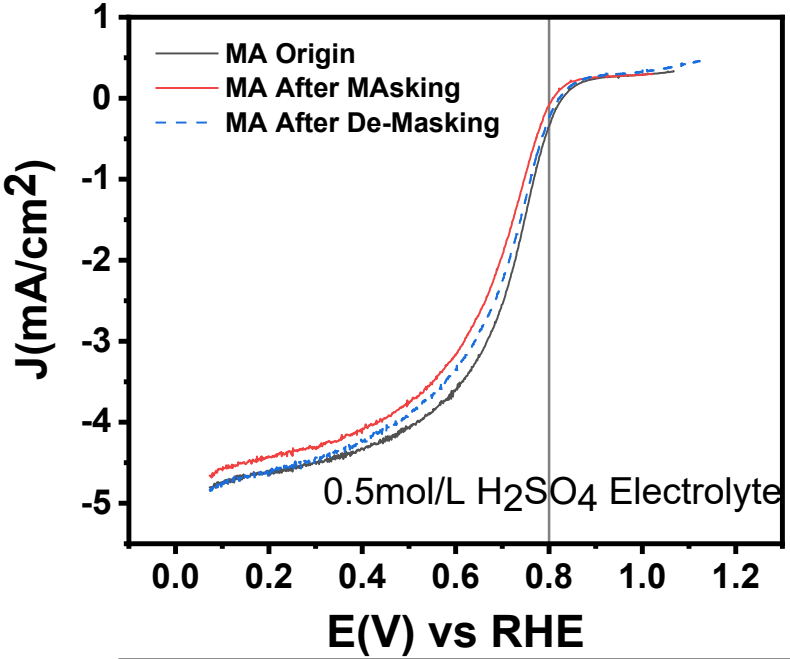


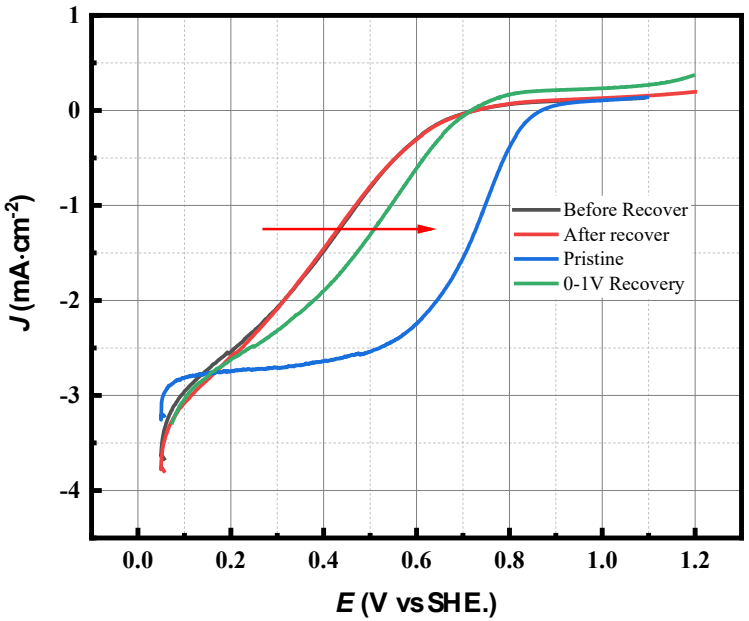
De-masking via Chem Desorption (Reduction)

Surface charge grafting:

- ❑ Covalently grafting -NH_2 group on catalyst surface for adding positive charges.
- ❑ Direct diazonium reaction leading to loss of ORR activity.
- ❑ Adopt new approach: (1) masking active center, (2) diazonium reaction, and (3) de-masking active center.

Masking Method For Protecting Active Sites of PGM-Free Catalysts





Black line?

Surface Masking/de-masking:

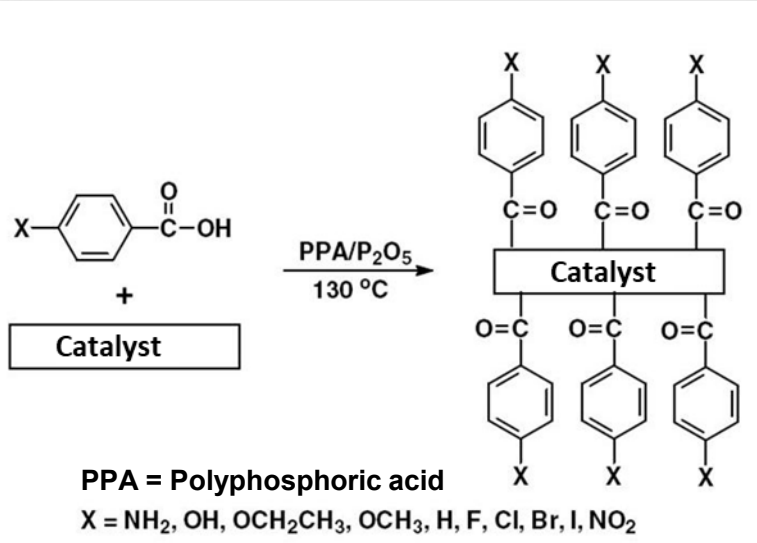
- ❑ Mass activity (MA) remains unchanged at 0.9 V after masking and de-masking.
- ❑ Slightly change of activity at higher current density.

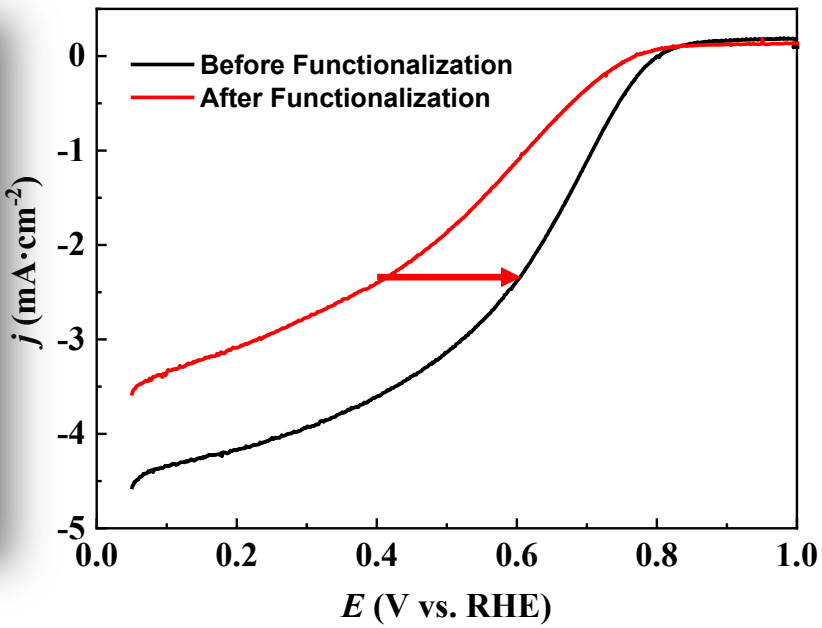
Surface Charge Grafting via Masking/Functionalizing/De-masking:

- ❑ Performance loss after the treatment
- ❑ De-masking recovers the performance
- ❑ Further de-masking by extending potential range of cycling recovers more performance

PGM-Free Catalyst from UB: 180515-HZ Fe-MOF-cat-100nm

2nd approach for functionalizing catalyst: **Mild** and **Nondestructive** Functionalization



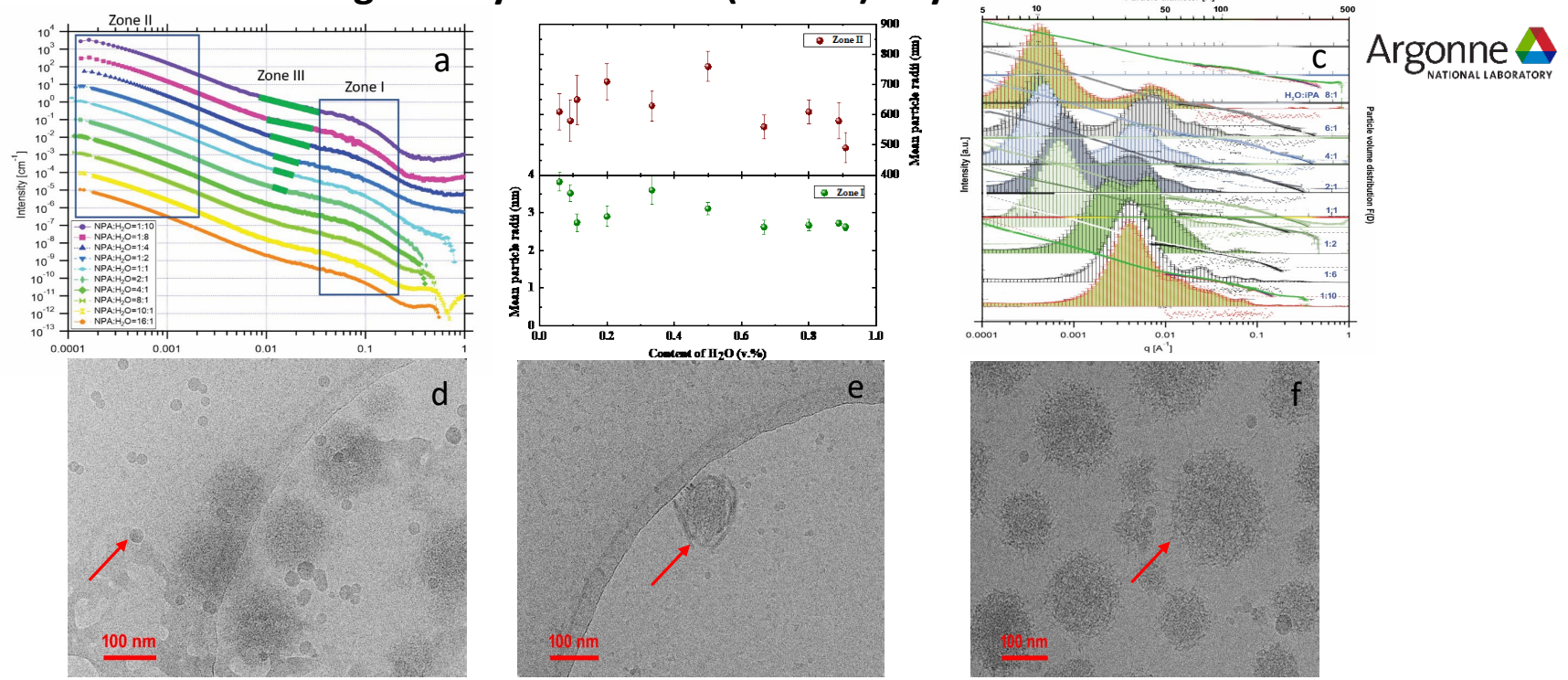


Surface charge addition:

- ❑ No masking needed.
- ❑ NH_2 group is covalently grafted on the surface of the PGM-free catalysts for positive charge.
- ❑ 70% performance achieved after functionalization.
- ❑ Need to further improve the performance recovery.

Rational Design MEA: from ink to the solid porous catalyst layer

Dispersion of catalyst and ionomer in H_2O/n-PA System studying with Ultra Small Angle X-ray Diffraction (USAXS)+ Cyro-TEM



USAXS data of ionomer and catalyst dispersion in H_2O/n-PA mixtures (a), USAXS fitting results of ionomer (lower) and catalyst particle (upper) size (b), ionomer particle size distribution in H_2O/n-PA mixtures (c), cyro-TEM images of ionomer and catalyst particles in H_2O/n-PA=1:4, (d), H_2O/n-PA=1:1, (e), H_2O/n-PA=4:1, (f), and all with I/C=1.0

Systematic Study of Catalyst and ionomer dispersion in H_2O/n-PA mixtures :

- ❑ Disperse both catalyst and ionomer in a solvent system.
- ❑ Morphology and geometry of catalyst, ionomer changing with different solvents.
- ❑ Ionomer particle size increasing with n-PA content, better dispersed in low n-PA content.
- ❑ Both catalyst and ionomer well dispersed at 70% H_2O content.
- ❑ Ionomer rods surrounding catalyst particle observed by cyro-TEM for 1:1 ratio solvent.

Rational Design MEA: from ink to the solid porous catalyst layer

Dispersion of catalyst and ionomer in H_2O/n-PA System studying with Ultra Small Angle X-ray Diffraction (USAXS)+ Cyro-TEM

H_2O: nPA volume ratio	Shape factor zone I	Mean particle size zone I (nm)	Shape factor zone II	Mean particle size (nm) zone II
10:1	4	2.62±0.10	3.124	490±50
8:1	4	2.72±0.09	3.207	580±60
4:1	4	2.67±0.15	3.291	610±40
2:1	4	2.62±0.18	3.335	560±39
1:1	4	3.11±0.17	3.275	760±50
1:2	4	3.60±0.27	3.289	630±60
1:4	4	2.90±0.23	3.355	710±82
1:8	4	2.74±0.38	3.265	650±50
1:10	4	3.52±0.24	3.353	580±60
1:16	4	3.82±0.22	3.32	610±69

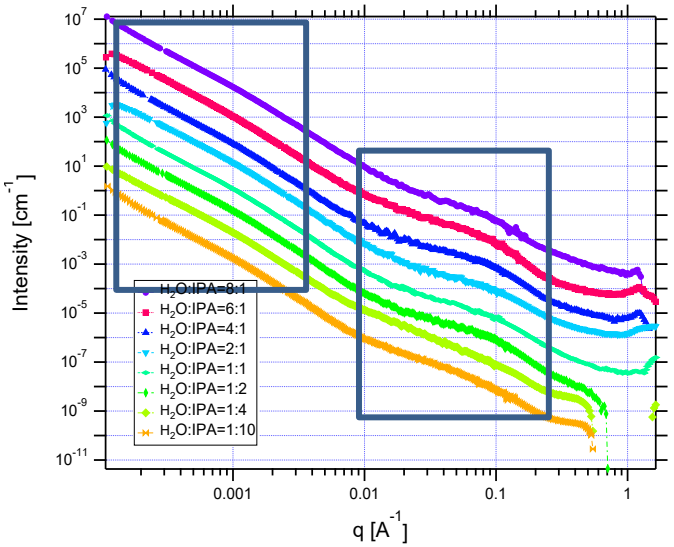
Systematic Study of Catalyst and ionomer dispersion in H_2O/n-PA mixtures :

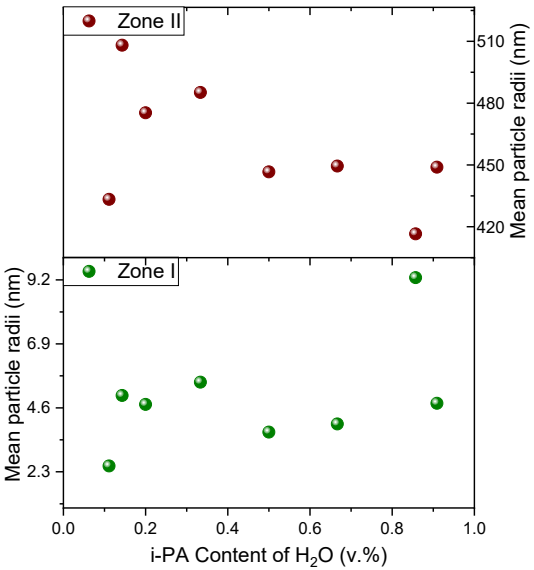
- ❑ Disperse both catalyst and ionomer in a solvent system.
- ❑ Morphology and geometry of catalyst, ionomer changing with different solvents.
- ❑ Ionomer particle size increasing with n-PA content, better dispersed in low n-PA content.
- ❑ Both catalyst and ionomer well dispersed at 70% H_2O content.
- ❑ Ionomer rods surrounding catalyst particle observed by cyro-TEM for 1:1 ratio solvent.

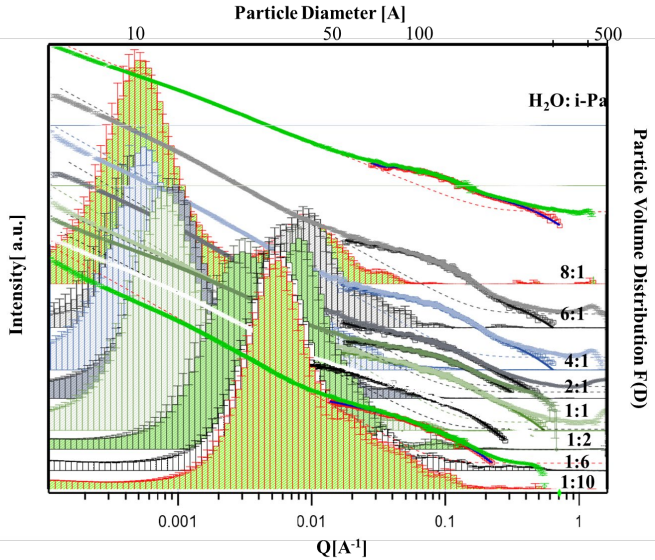
Rational Design MEA: from ink to the solid porous catalyst layer

Dispersion of catalyst and ionomer in H_2O/i-PA System studying with Ultra Small Angle X-ray Diffraction (USAXS)+ Cyro-TEM

Argonne NATIONAL LABORATORY





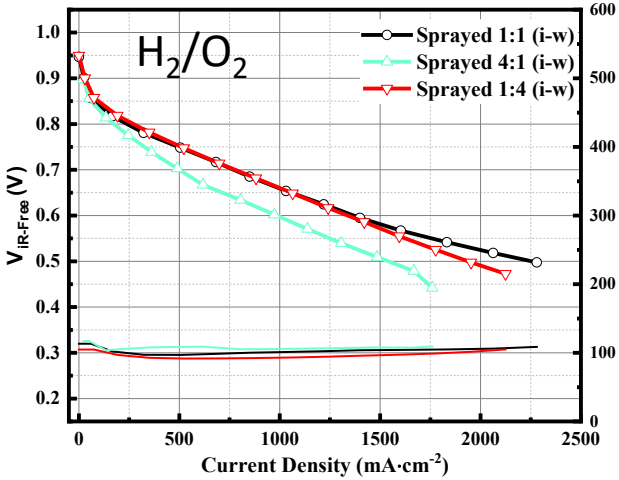


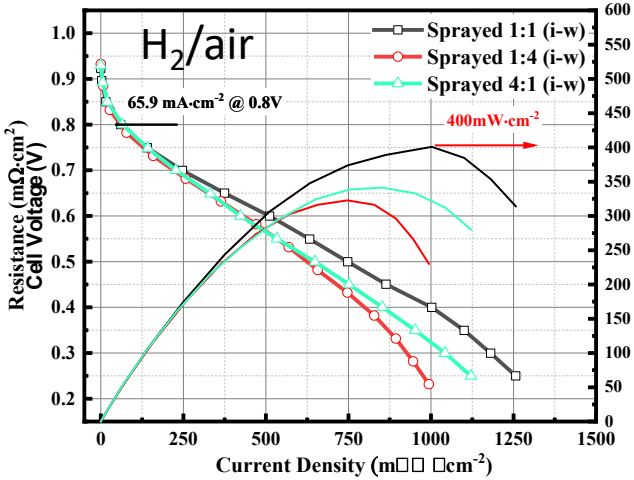
Systematic Study of Catalyst and ionomer dispersion in H_2O/i-PA mixtures:

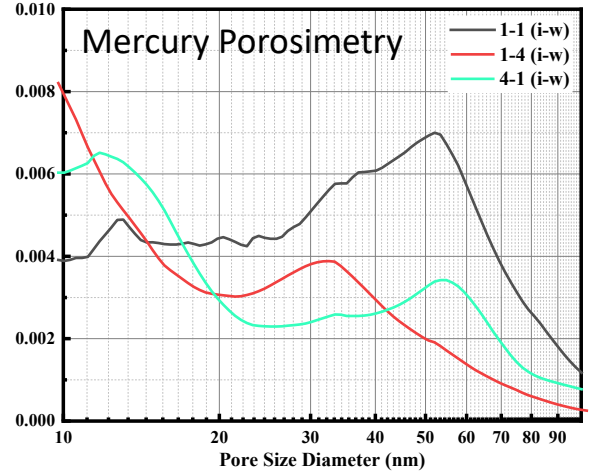
- ❑ Disperse both catalyst and ionomer in a solvent system.
- ❑ Morphology and geometry of catalyst, ionomer changing with different solvents.
- ❑ Ionomer particle smallest size at 50% H_2O.
- ❑ Catalyst particle smallest size at 90% H_2O.
- ❑ Combined ionomer and catalyst particle size at 50% H_2O content.

Rational Design MEA: from ink to the solid porous catalyst layer

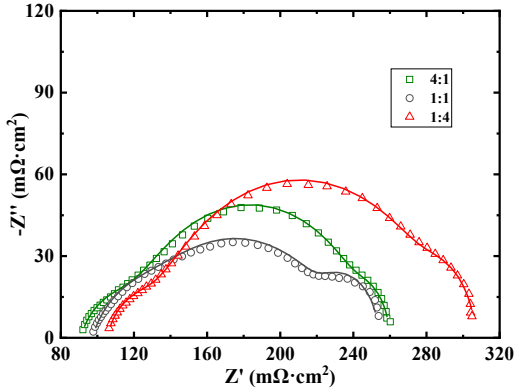
Solvent Effect on the Pore Structure of Catalyst Layer and MEA Performacne







MEA: 5 cm^2, anode: 0.2mg$_{Pt}$/cm^2 /Fe-N-C cathode: 4 mg/cm^2 , N212 80°C_100% RH 200:400 sccm H_2/O_2 ,500:1000 sccm H_2/air, 150KPaabs



w/n	R_Ω	R_{anode}	$R_{cathode}$	R_{mt}
4:1	91.0	30.4	127.3	11.7
1:1	97.3	37.6	**90.9**	28.6
1:4	105.5	24.5	159.7	18.1

	iPa-water (1-1)	iPa-water (1-4)	iPa-water (4-1)
Pore volume 0-20nm (mL/g)	**0.104**	0.128	0.107
Pore volume 20-200nm (mL/g)	**0.391**	0.147	0.199
Mesopore percentage (%)	**79.1**	53.3	65.0

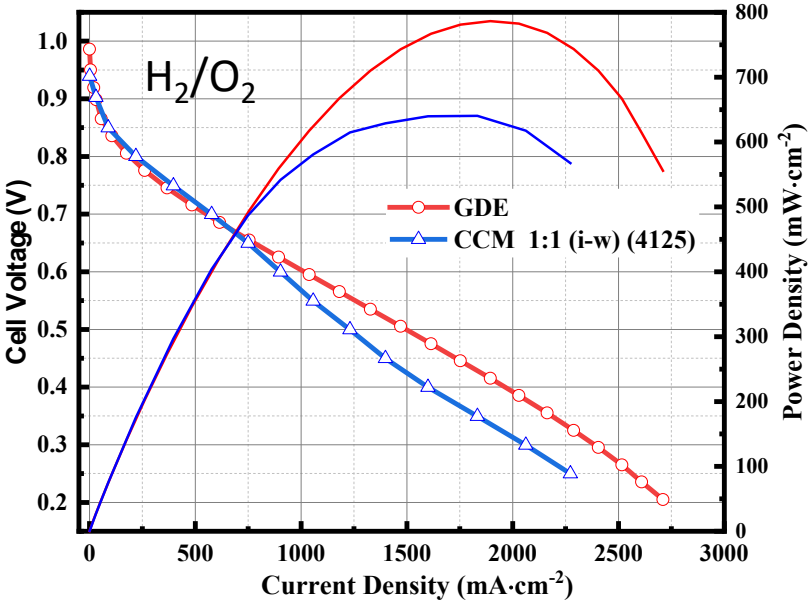
Ink combination	H_2-O_2		H_2-air	
	j@0.9 $V_{iR\text{-}Free}$(mA cm^{-2})	Pmax (W cm^{-2})	j@0.8 V (mA cm^{-2})	Pmax (W cm^{-2})
iPa-Water (1-1)	30	0.639	60.44	0.40
iPa-Water (1-4)	30.5	0.642	59.42	0.32
iPa-Water (4-1)	32	0.522	65.87	0.34

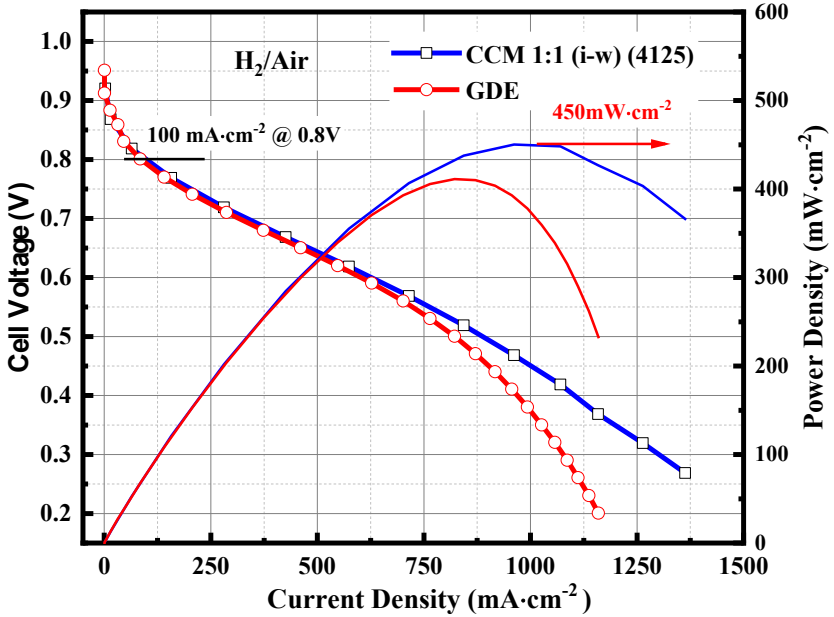
Solvent Effect on MEA (H_2O/i-PA mixtures) :

- Less effect on mass activity (high voltage).
- Strong effect on mass transport (H_2/Air).
- Solvent with 1:1 (i-PA:H_2O) ratio has the highest pore volume, mesopores percentage and $R_{cathode}$ (AC impedance), well correlated with mass transport.

MEA Fabrication Methods

Gas Diffusion Electrode (GDE) vs. Catalyst Coated Membrane (CCM)





MEA: 5 cm^2, anode: 0.2mg_{Pt}/cm^2 /Fe-N-C PGM-Free cathode: 3.6-4 mg/cm^2 , N212 80°C_100% RH 200:400 sccm H_2/O_2 ,500:1000 sccm H_2/Air, 150KPaabs

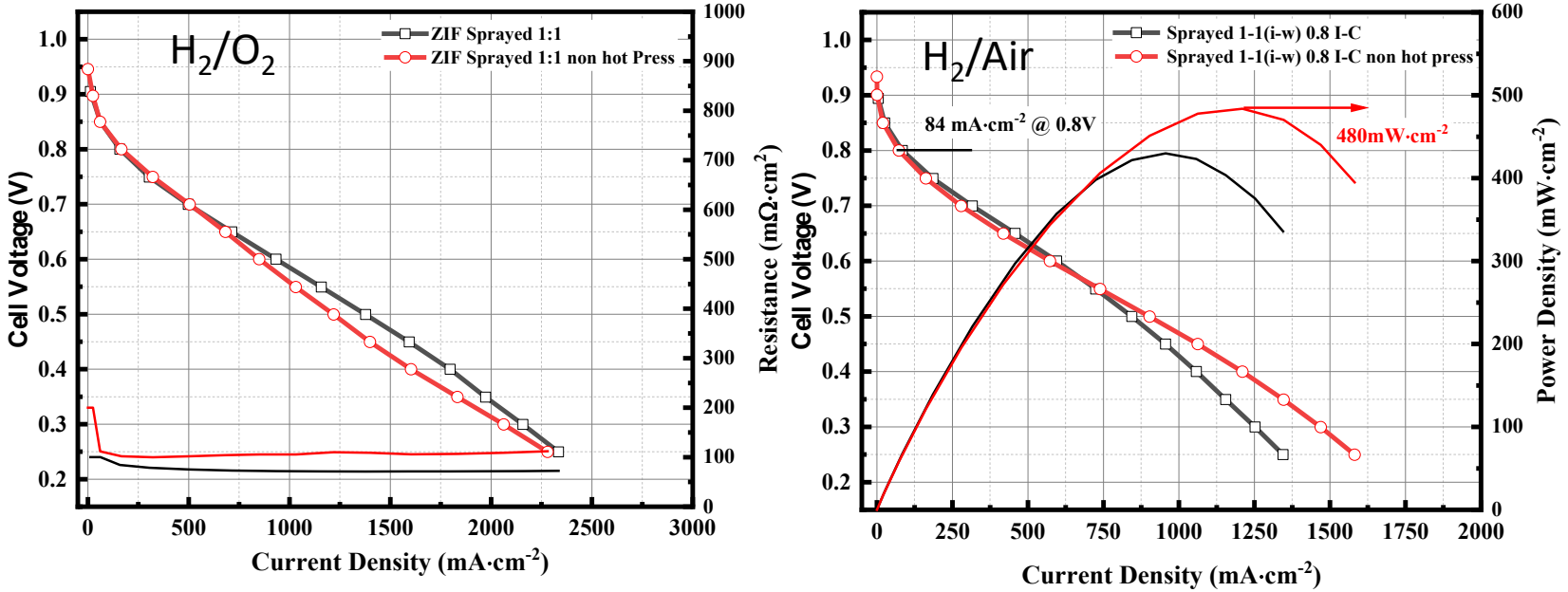
GDE	31	0.78	86	0.41
Sprayed 1:1 (4125)	35	0.64	100	0.45

Fabrication Method Comparison:

- ❑ Machine sprayed CCM with similar performance with that of GDE for mass activity.
- ❑ Machine sprayed CCMs with high reproducibility.

MEA Fabrication Methods

Hot Press *vs*. Non Hot Press



MEA: 5 cm^2, anode: 0.2mg_{Pt}/cm^2 /Fe-N-C cathode: 3.6-4 mg/cm^2 , N212 80°C_100% RH 200:400 sccm H_2/O_2 ,500:1000 sccm H_2/Air, 150KPaabs

Hot Press				
Hot Press	25	0.72	84	0.43
Non hot press	32	0.64	73	0.48

Fabrication Method-Hot Press Effect for Sprayed CCM:

- ❑ Hot press leading to lower mass activity.
- ❑ Hot press resulting in higher peak power for O_2.
- ❑ Non hot press making higher resistance but higher mass activity.
- ❑ Non hot press leading to better air performance.

Response to Previous Year Reviewers' Comment

Approach

Comment: The approach involves functionalizing only the surface of this large particle and not the mesopores.

Response: functionalization utilizes the wet chemistry, e.g. diazonium reaction or other reactions in liquids (e.g. H_2O). The UB's PGM-free catalyst is somewhat hydrophilic (can be dispersed in H_2O). To ensure the functionalization, sonication will be used to enhance the liquids penetrating into mesopores. Micropore should not be affected as ionomer particles can't go into them.

Comment: It is not clear how the ionomer thickness and distribution are being controlled.

Response: The ideal is to increase the ionomer coverage by charge attraction while reducing the thickness of ionomer film. This has been proved by our and other's publications.

Comment: Adding mesoporosity, ideal ionomer interface and ink formulations……

Response: The goal is indeed to increase the accessible density of active sites. Ink formulation has a large portion in the project as reported. Surface groups includes –NH2, polybenzimidiazole (PBI), and polyaniline (PANI).

Response to Previous Year Reviewers' Comment

Collaboration

Comment: Working with National Lab. ***Response***: currently, closely working with ANL on USAXS, XAS and modeling and ORNL on TEM.

Relevance/potential impact

Comment: PGM-free catalyst scale-up. ***Response***: up to 5 g synthesis has been achieved.

Future work

Comment: Little attention to ionomer effect…… ***Response***: Ionomer loading in catalyst layer and ionomer dispersion in ink are heavily investigating.

Project weakness

Comment: How functionalization affects ionomer coverage and thickness need to be added. ***Response***: This is being actively studied.

Comment: Project management and communication. ***Response***: bi-weekly meeting and PI meeting.

Recommendation

Comment: Modeling effort is needed. ***Response***: Working closely with ANL (Rajesh's group)

Comment: Durability testing. ***Response***: It is undertaking.

Collaboration and Coordination

Institution	Project Role
IUPUI-Leading PI (J. Xie, Y. Liu, C. Li and G. Qing)	Project lead, management and cordination, construct ionomer/catalyst interface vai charge attraction, MEA design, ink formulation, MEA testing and characterization
UB- Co-PI(G. Wu, Shengwei Liu)	Design and develop advanced ***hierarchically porous carbon sphere*** (HPCS)@M-N-C catalysts for PGM-free cathodes in PEMFCs through controllable synthesis
UTRC (JV. Yang)	Test, diagnose sub-scale MEAs (25-50 cm^2) and carry out the economic analysis

ElectroCat Consortium	
Institution	**Capability**
ANL	Ultra-small Angle X-ray Scattering (USAXS), in situ and Operando Atomic, Nano-, and Micro-structure Characterization (X-ray adsorption, including ex-
ORNL	Electron microscopy (TEM, ELLS, etc.)
NREL	Kinetics and Transport (Operando differential cell measurements of electrochemical kinetics and transport)

Remaining Challenges and Barriers

- Increase activity of PGM-Free catalyst while improving stability.
- Construct ideal ionomer/catalyst interface via charge attraction.
- Functionalize PGM-Free catalyst with desired charge density while maintain high performance.
- Increase MEA mass activity while improve power performance with air.

Proposed Future Work

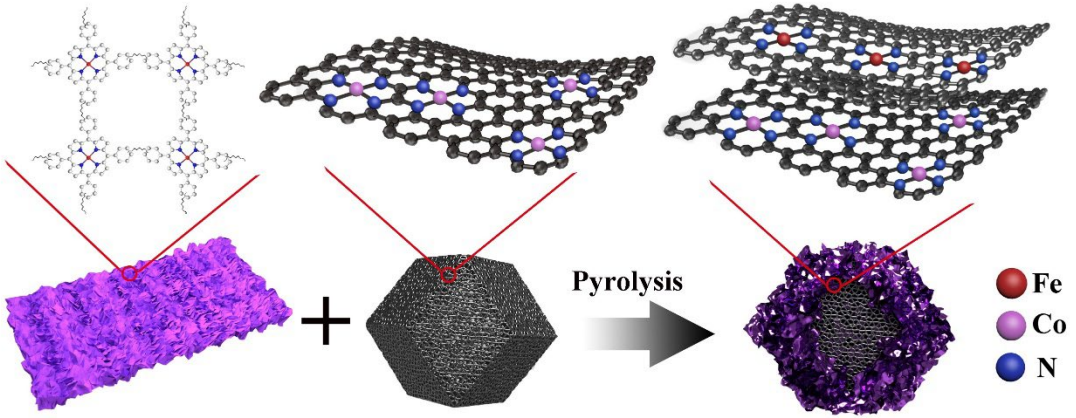
- Synthesize core-shell structured Fe/Co-N-C catalyst with high activity and high stability.
- Optimize the functionalization methods for controlling the surface charge of PGM-Free catalyst while maintain high activity and stability.
- Develop machine sprayed CCM with both high mass activity and high-power performance.
- Optimize ink formulation on ionomer/carbon ratio and different solvent systems.

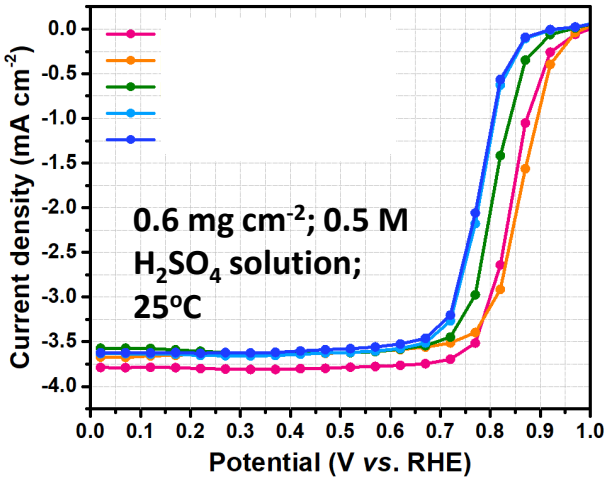
Summary

- The developed core-shell structured Fe/Co-N-C catalyst exceeds year 1 milestone (33 mA/cm^2 at 0.90 $V_{IR\text{-}free}$ in H_2/O_2, 133 mA/cm^2 at 0.80 V, 480 mW/cm^2, H_2/Air).
- Developed machine sprayed MEAs with the same performance of GDE.
- Achieved simultaneously dispersion of ionomer and catalyst particles aiding by USAXS+cyro-TEM.
- Rationally designed MEA from ink to solid porous layer using USAXS+cyro-TEM, and Hg porosimetry.
- Developed new methods for PGM-Free catalyst functionalization.

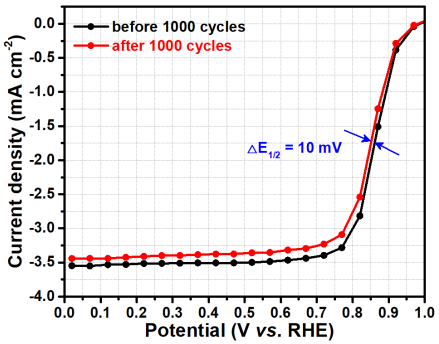
Technical Back-up Slides

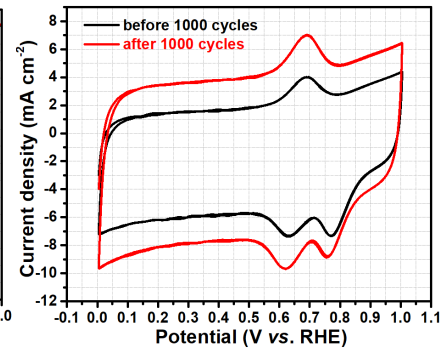
Future direction: Core-shell structured Fe/Co-N-C Catalysts



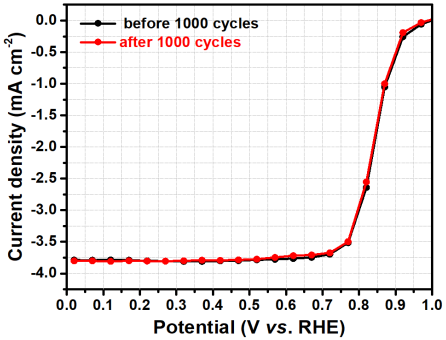


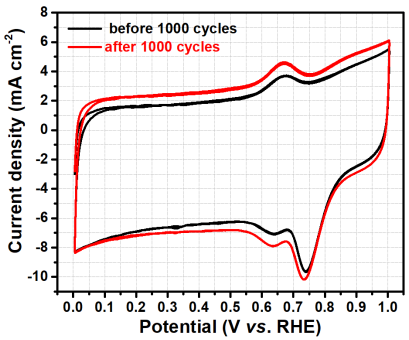
Fe@Fe-N-C Catalysts





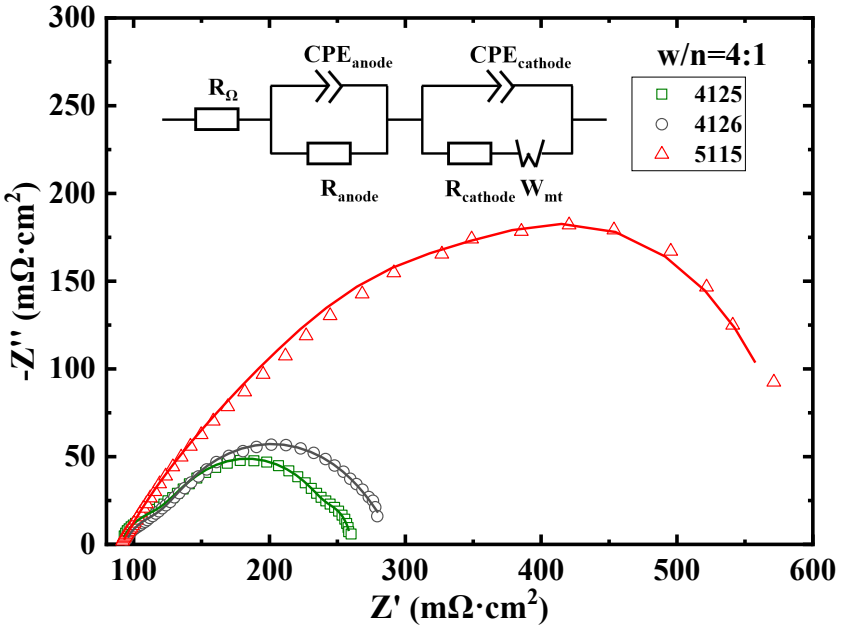
Co@Fe-N-C Catalysts

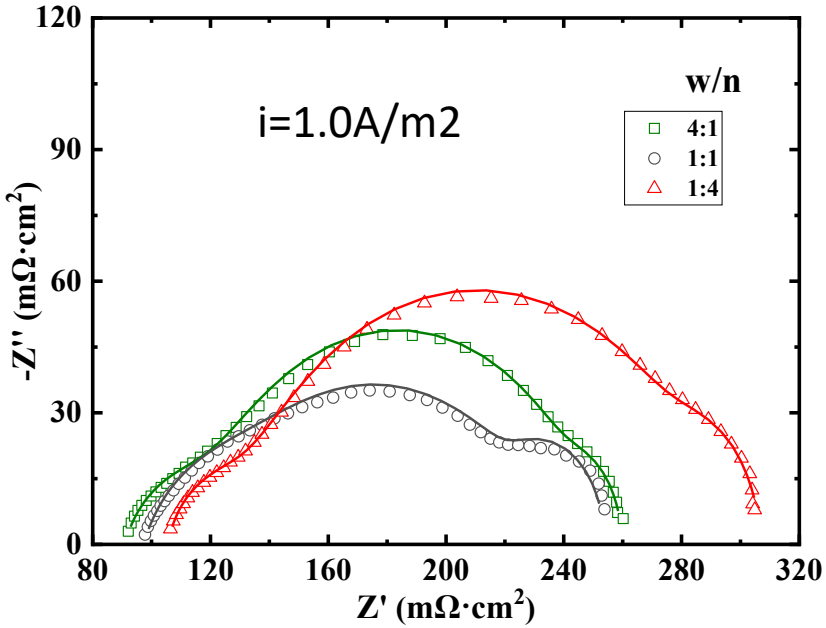




Core-shell Co@Fe catalyst exhibited encouraging RDE activity $E_{1/2}$=0.85 V and enhanced stability relative to Fe@Fe catalyst due to the more stable carbon structures

AC Impedance of MEA





Pinch	R_Ω	R_{anode}	$R_{cathode}$	R_{mt}
4125	91.0	30.4	**127.3**	11.7
4126	93.6	26.0	165.1	26.3
5115	91.1	232.5	230.4	48.1

w/n	R_Ω	R_{anode}	$R_{cathode}$	R_{mt}
4:1	91.0	30.4	127.3	11.7
1:1	97.3	37.6	**90.9**	28.6
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Rational Design MEA: from ink to the solid porous catalyst layer

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1:16	4	3.82±0.22	3.32	610±69

4= sphere
3=disc

Systematic Study of Catalyst and ionomer dispersion in H_2O/n-PA mixtures :

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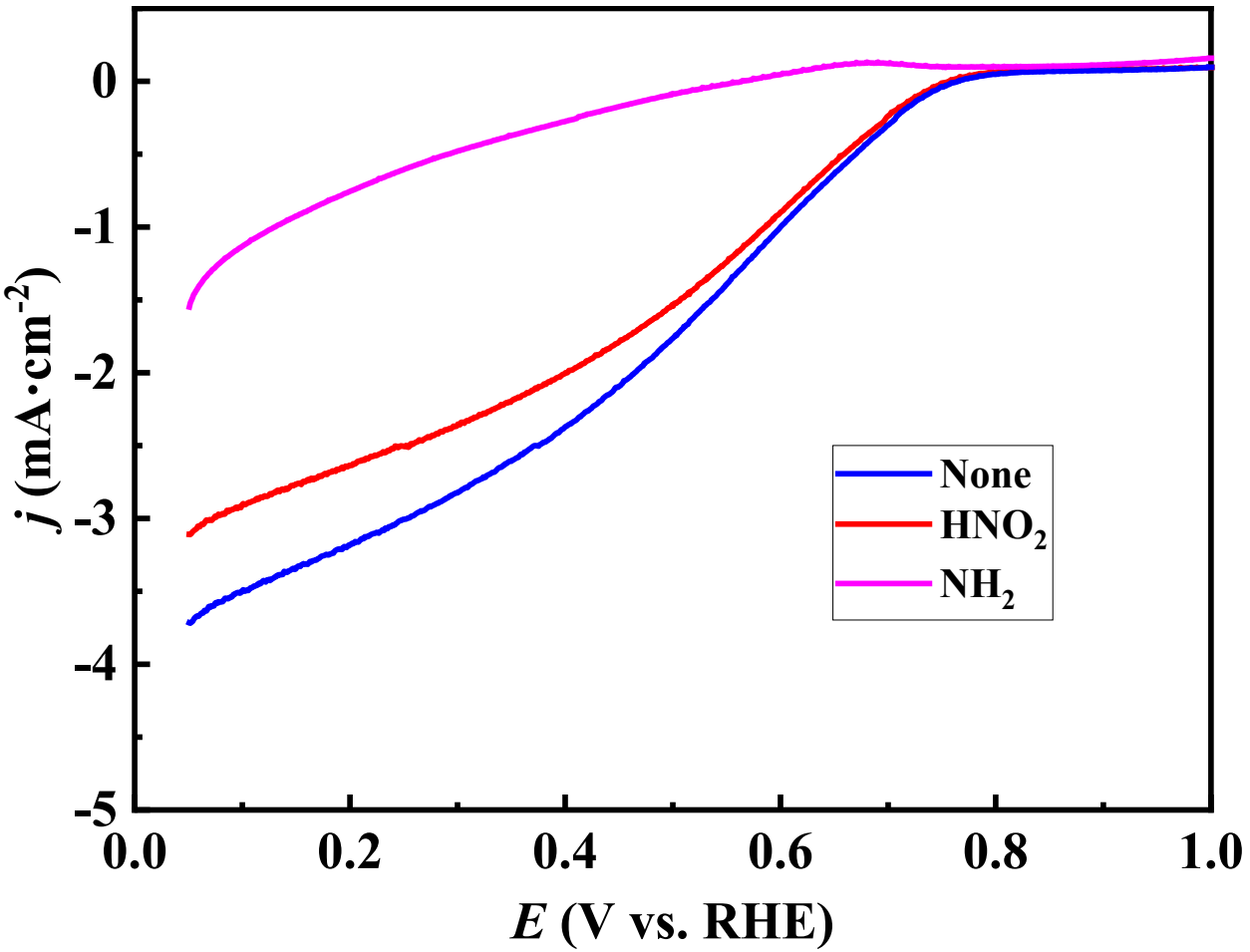
Rational Design MEA: from ink to the solid porous catalyst layer

Dispersion of catalyst and ionomer in H_2O/i-PA System studying with Ultra Small Angle X-ray Diffraction (USAXS)+ Cyro-TEM

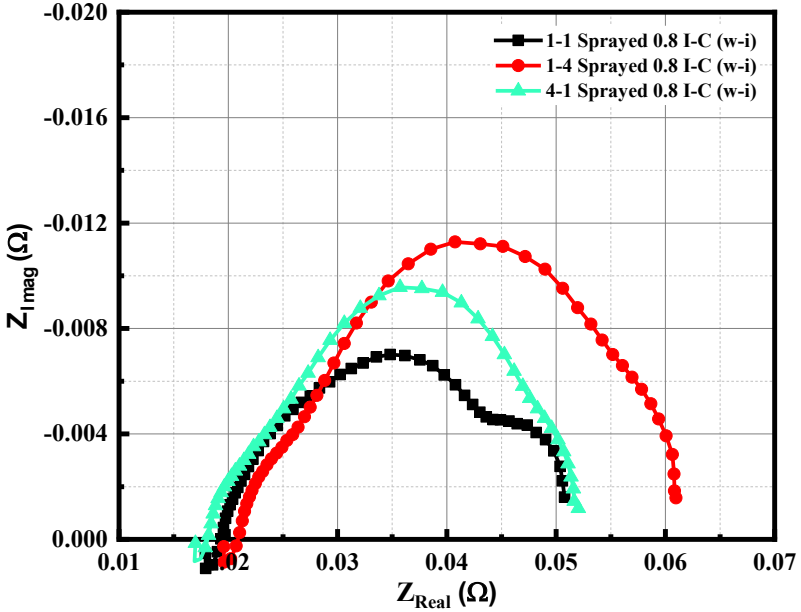
Table 2 Particle size and shape information in different catalyst inks i-PA and water system

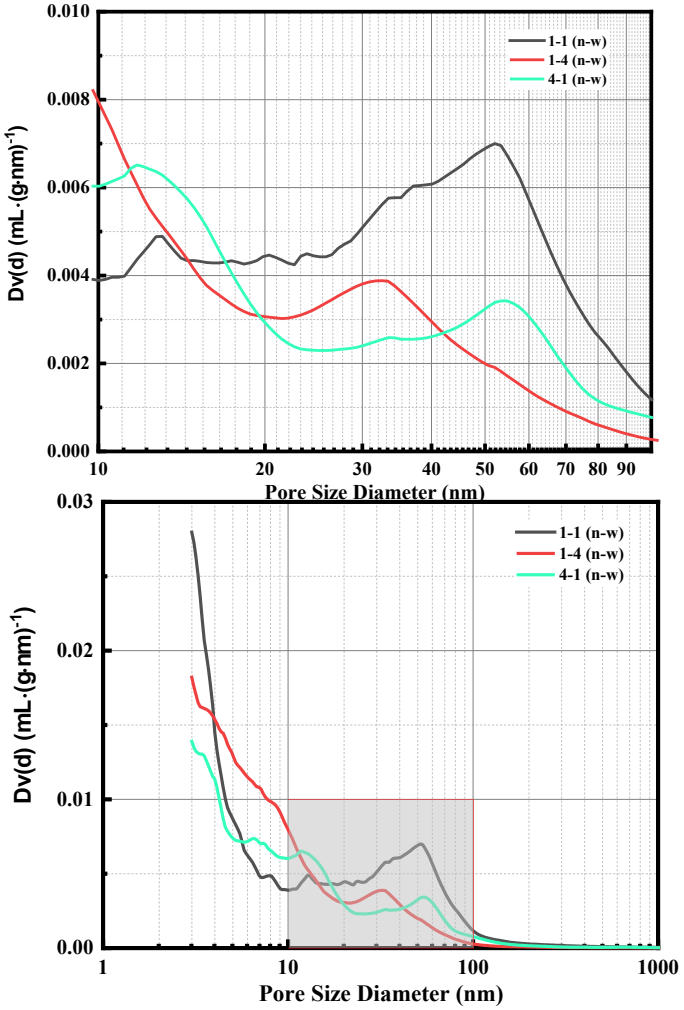
H_2O: iPA volume ratio	Shape factor zone I	Mean particle size zone I (nm)	Size range Zone I (nm)	Shape factor zone II	Mean particle size (nm) zone II
8:1	4	2.34±0.17	0.5-10	3.358	500±25
6:1	3.275	3.68±0.45	0.5-11	3.421	530±46
4:1	3.323	2.40±0.25	0.5-9	3.372	590±62
2:1	3.518	4.03±0.43	0.5-11	3.362	580±57
1:1	3.836	4.60±0.43	1-8	3.302	630±91
1:2	4	3.88±0.27	1-30	3.461	520±52
1:6	4	4.22±0.33	1-15	3.243	700±71
1:10	4	4.18±0.21	1-30	3.364	580±89

Root Cause Analysis



PGM-Free MEA fabrication optimization





Anode: 20wt. % Pt/XC72 ETEK 0.2mg$_{Pt}$/cm^2
Cathode: 20mg Fe_2O_3 ZIF PGM-Free CAT 4mg$_{cat}$/cm^2
EIS test under 5A @ 300-500 sccm (H_2-O_2)

	iPa-water (1-1)	iPa-water (1-4)	iPa-water (4-1)
Pore volume 0-20nm (mL/g)	0.103517	0.128467	0.107344
Pore volume 0-200nm (mL/g)	0.391498	0.146661	0.198963
Mesopore percentage (%)	0.790881084	0.53306461	0.649554205