

Evolution of Ultrathin CoFe-Nanomesh for Oxygen Evolution Reaction: From Slit Pores to Ink-Bottle Pores

6th Commonwealth Chemistry Posters

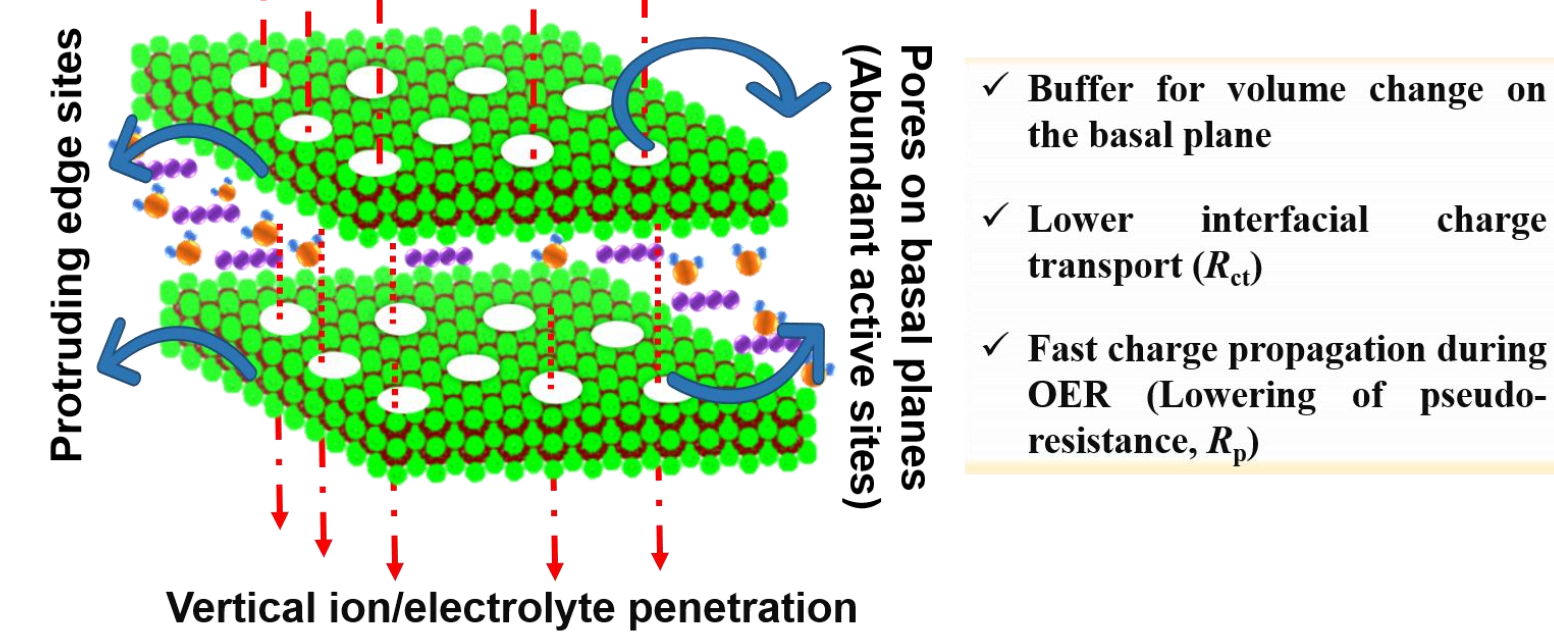
Shashank Sharma, and Amit Paul* Email: apaul@iiserb.ac.in
Indian Institute of Science Education and Research, Bhopal (M.P) India

Research Background

❖ Layered double hydroxides (LDHs)

- ✓ Heteroatom doping
- ✓ Interlayer anion exchange
- ✓ Exfoliation and reducing lateral size
- ✓ Abundant defects generation

- Nanomaterials having micropores usually lead to structural collapse ✗
- Narrow mesopores (3–4 nm pore size) provided mechanical strength under continuous OER operation ✓

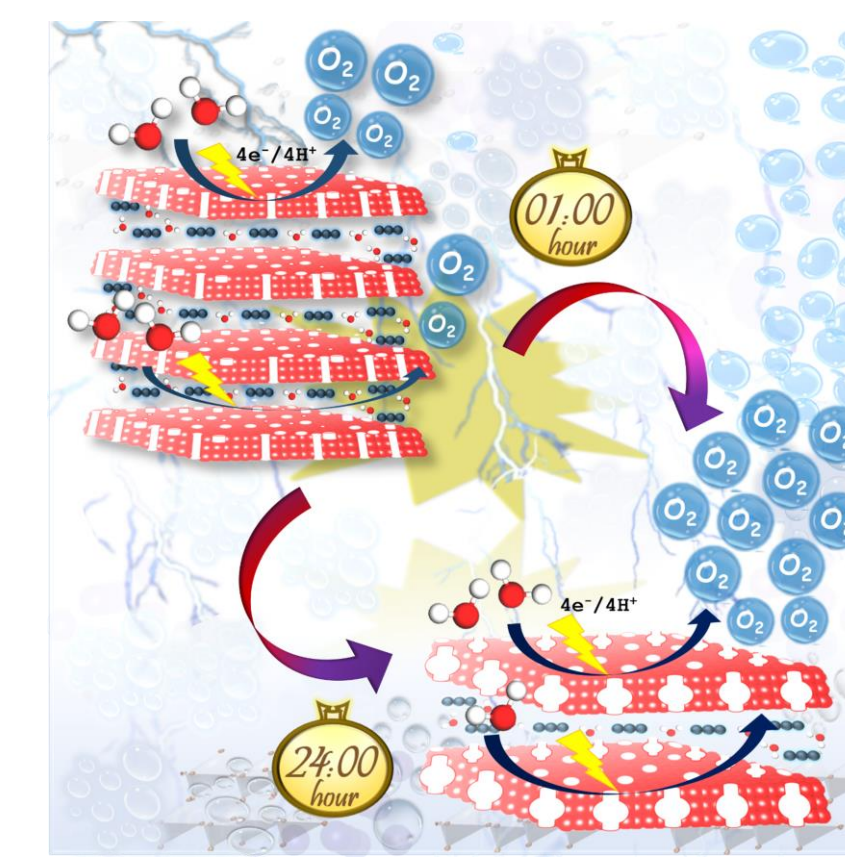


❖ Aim

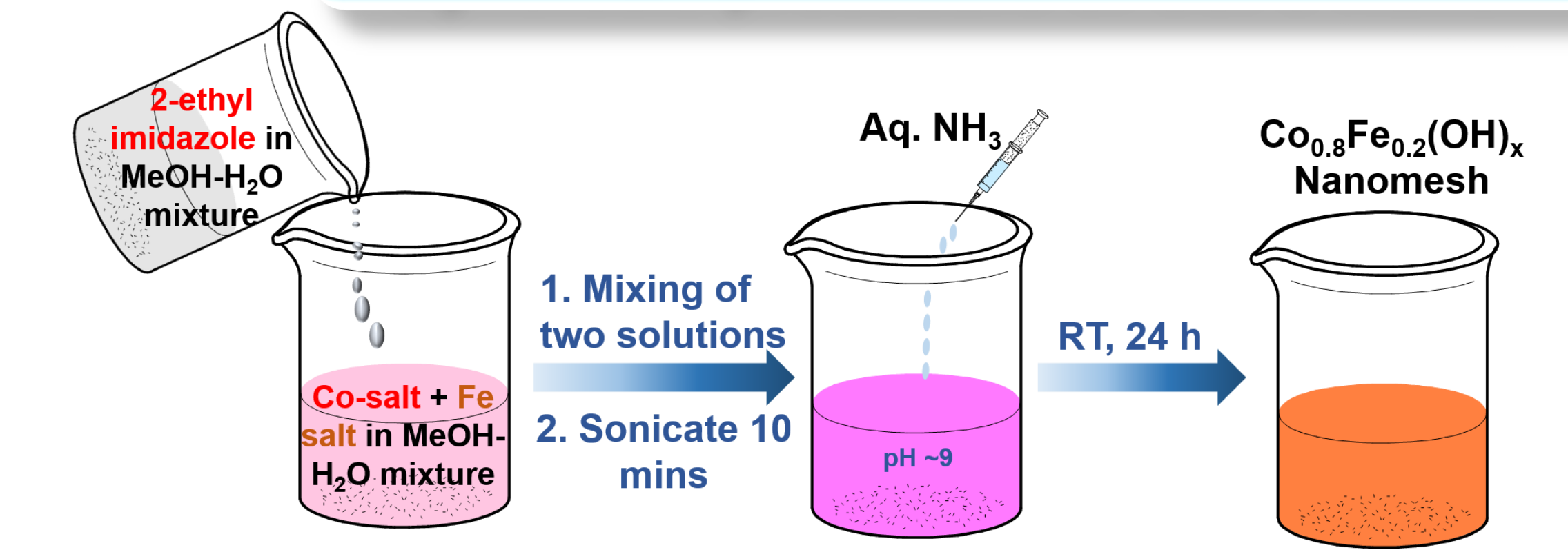
To study the electrocatalysis by generating abundant narrow mesopores on the basal planes

Abstract

- ✓ The time-dependent mechanism underlying the formation of $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -t nanomesh (nanomesh having 80% Co and 20% Fe, “t” represents materials synthesis time) has been identified towards the development of a highly effective catalyst for the oxygen evolution reaction (OER)
- ✓ Utilizing 2-ethyl imidazole (2-HEIM) as an etching reagent and the Ostwald ripening process enabled the evolution of nanomesh formation with a precise pore size of ink-bottle shape
- ✓ This research underscores the critical role of pore morphology in nanomeshes

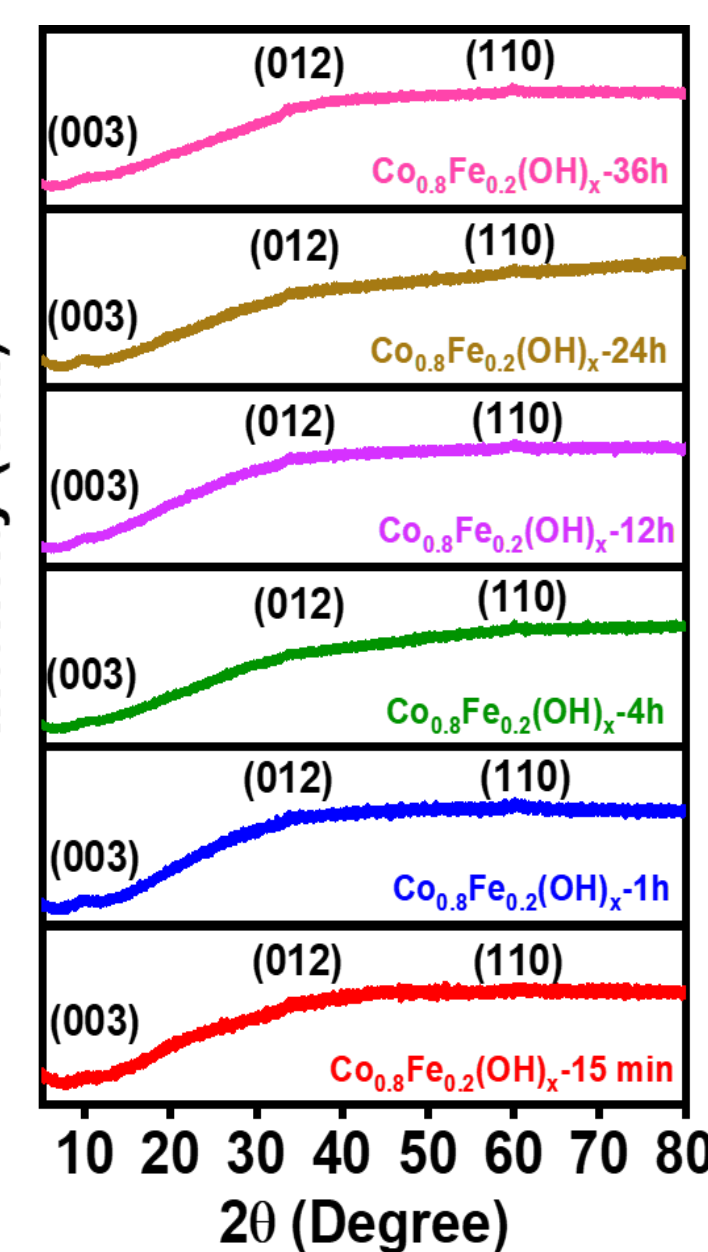


Synthesis procedure of CoFe-Nanomesh

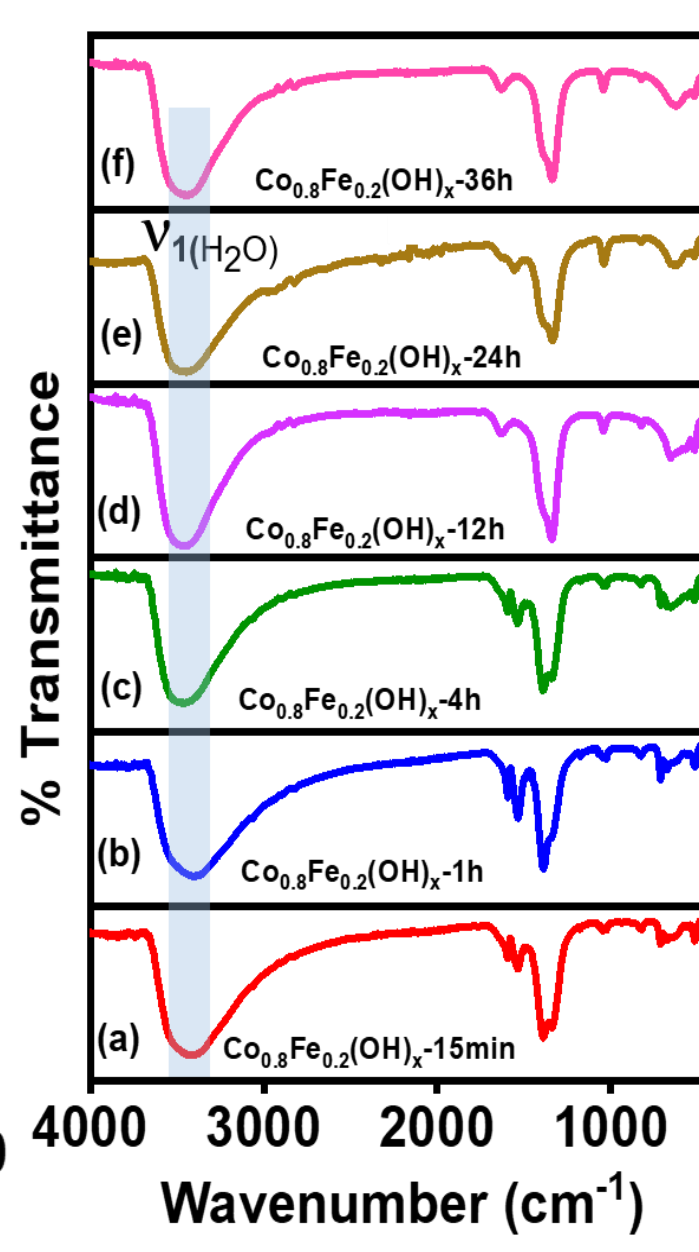


- $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -t-nanomeshes were synthesized at different reaction time intervals (t=15 min; 1, 4, 12, 24, and 36 h)
- Named as $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -15 min, $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -1h, $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -4h, $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -12h, $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -24 h, and $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -36 h

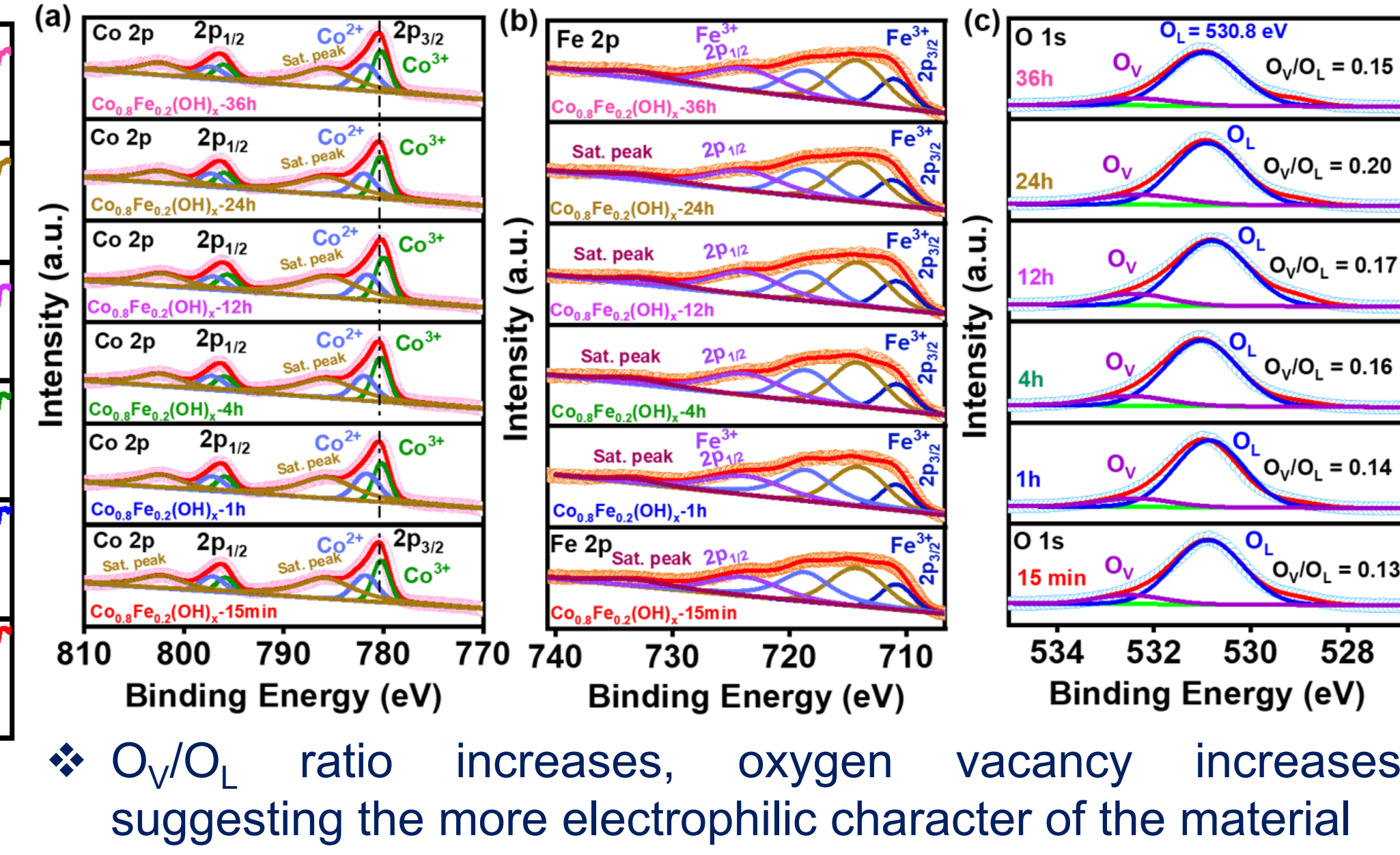
PXRD



FTIR

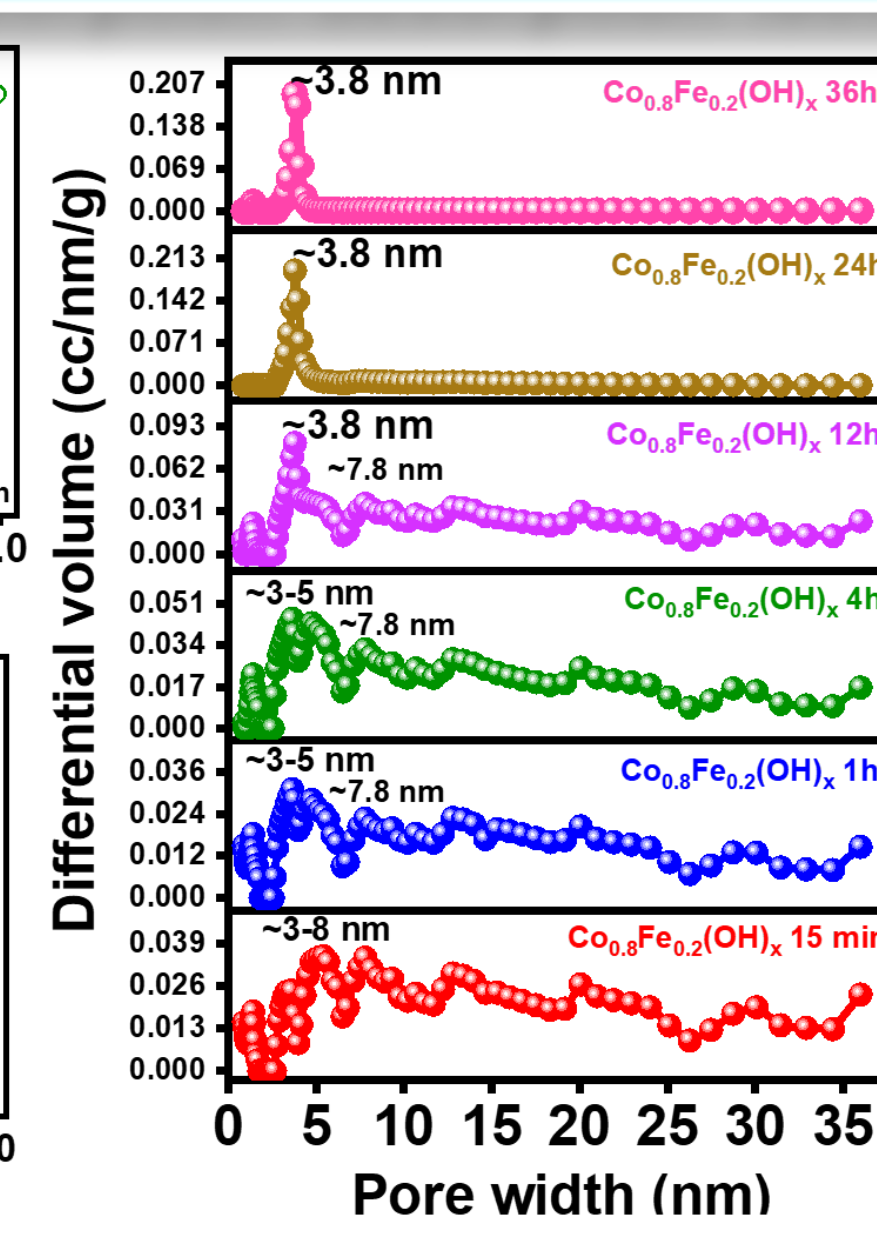
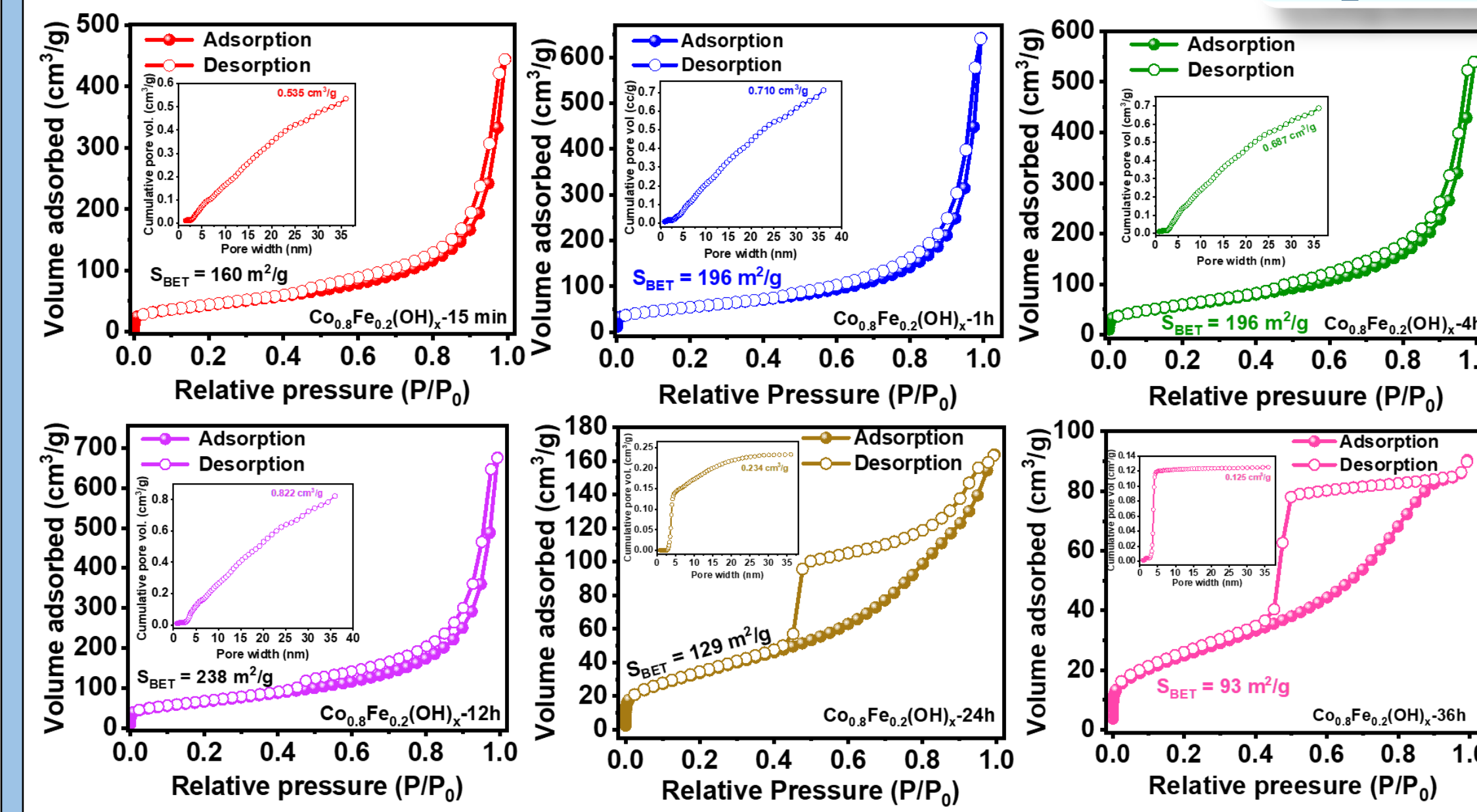


XPS analysis



❖ O_v/O_t ratio increases, oxygen vacancy increases, suggesting the more electrophilic character of the material

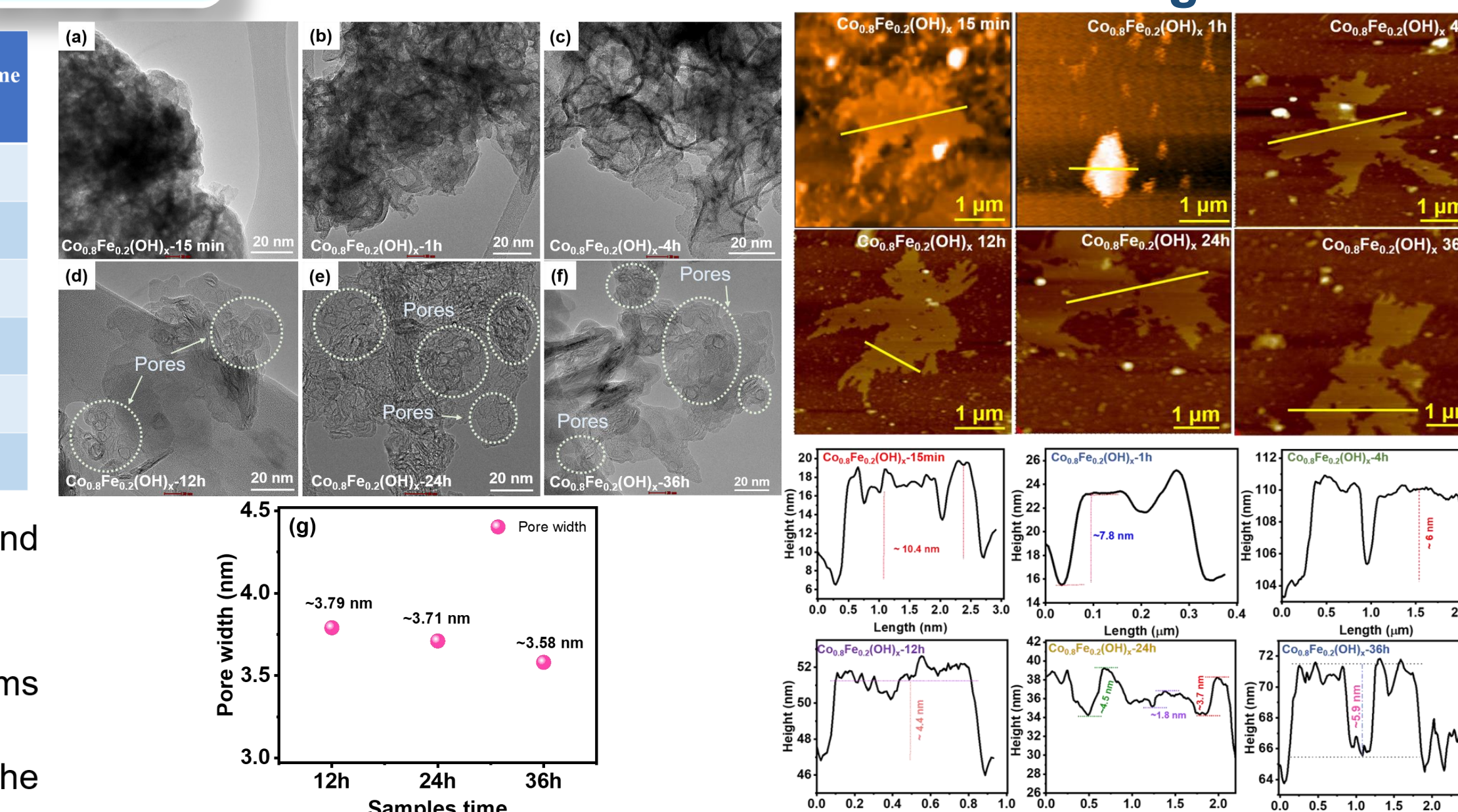
N₂ adsorption-desorption isotherms, pore size distribution, and TEM analysis



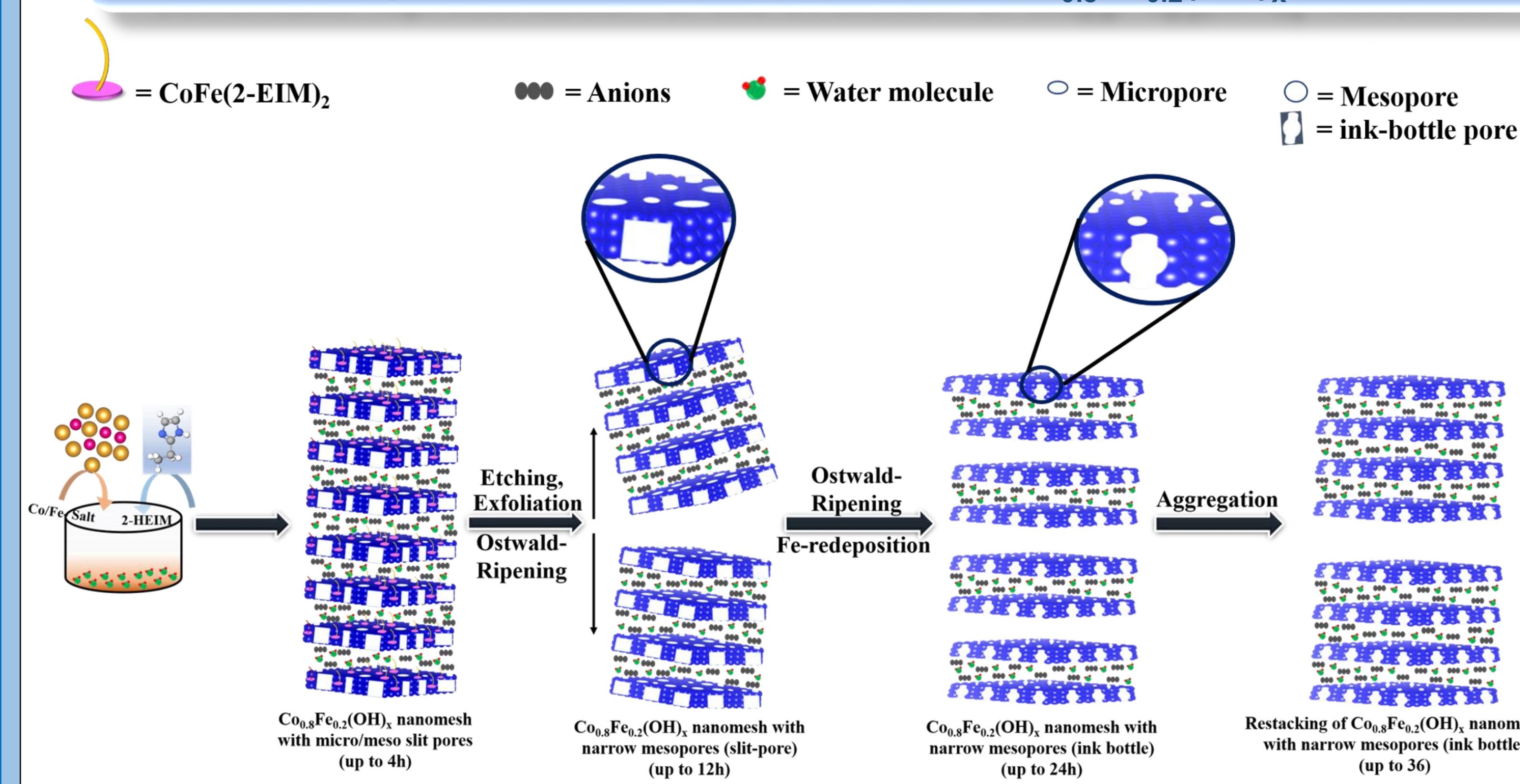
Sample	BET-SA (m ² /g)	Pore width (nm)	Micropore volume (cm ³ /g)	Narrow mesopore volume (cm ³ /g)	Large mesopore volume (cm ³ /g)	Pore volume (cm ³ /g)
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -15 min	160	3.8	0.013	0.058	0.464	0.535
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -1 h	196	3.8	0.016	0.052	0.643	0.711
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -4 h	179	3.8	0.016	0.088	0.583	0.687
CoFe20-LDH 12 h	238	3.8	0.017	0.103	0.702	0.822
CoFe20-LDH 24 h	129	3.8	0.000	0.143	0.091	0.234
CoFe20-LDH 36 h	93	3.8	0.004	0.111	0.010	0.125

$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -t (t=15 min, 1, 4, and 12 h) $\Rightarrow$ type-I and type-IV isotherms $\Rightarrow$ Microporous and mesoporous
 $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -t (t= 24, 36 h) $\Rightarrow$ type-IV sorption isotherms with H2-type hysteresis $\Rightarrow$ Ink-bottle pores
 $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -24 h $\Rightarrow$ uniform pore width ~3.8 nm with the highest narrow mesopore volume

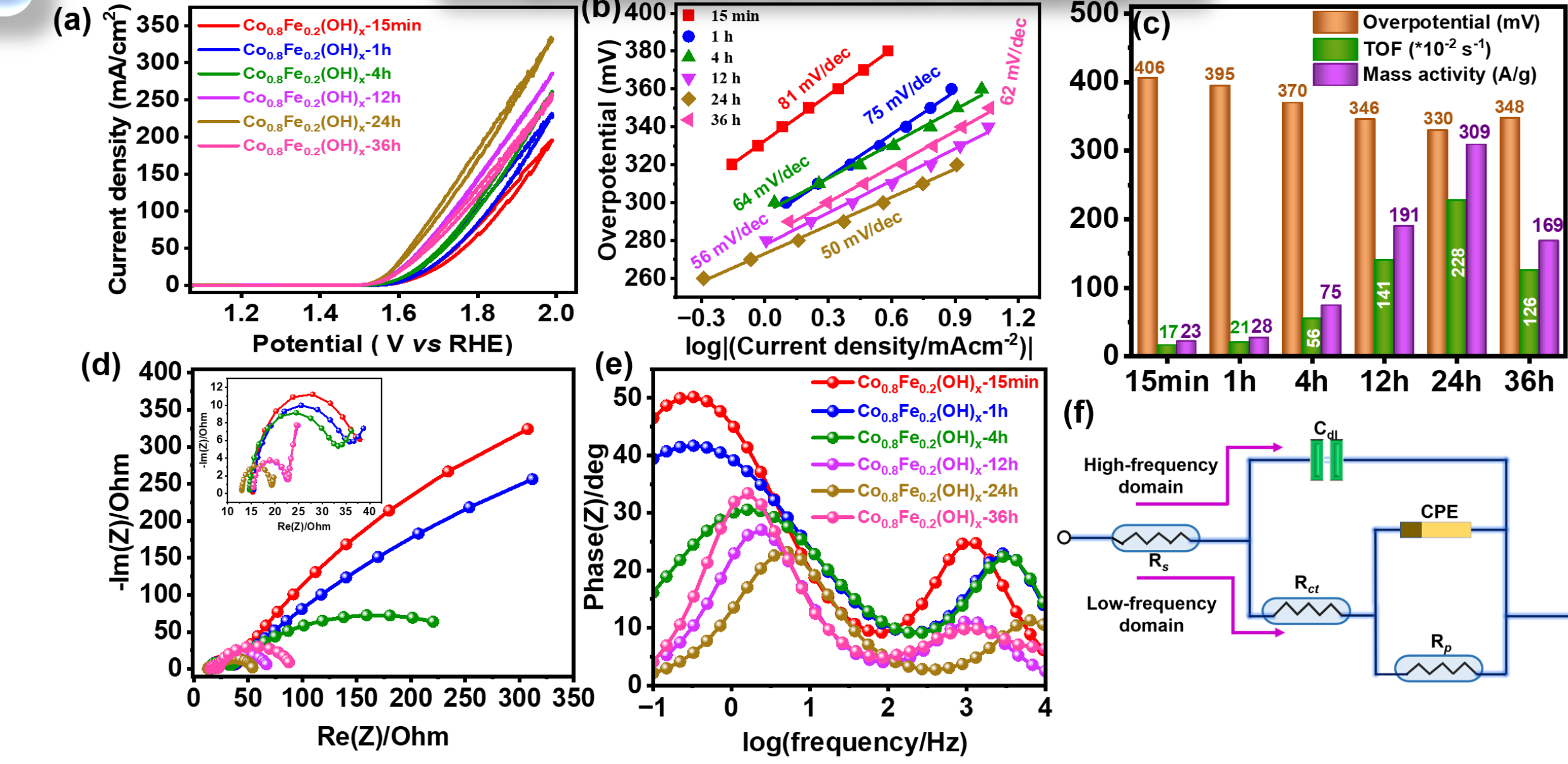
AFM and Height Profile



Schematic representation of the evolution of $\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ nanomesh

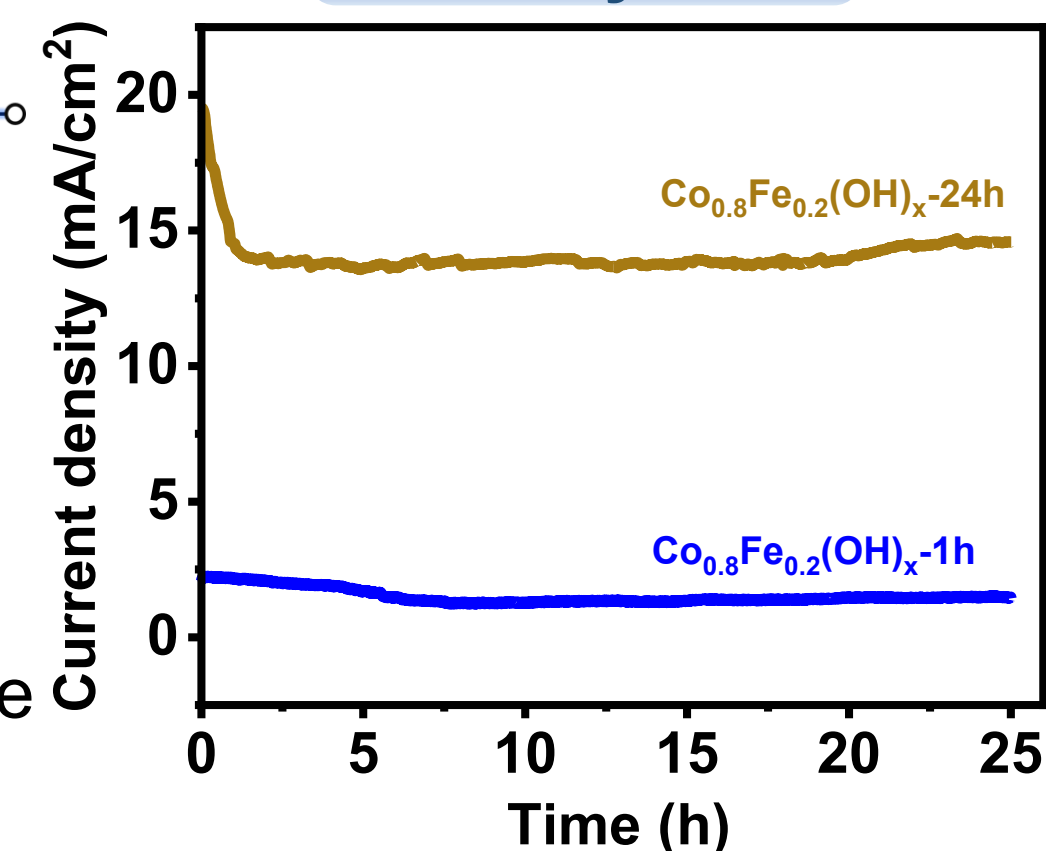


Electrochemical results

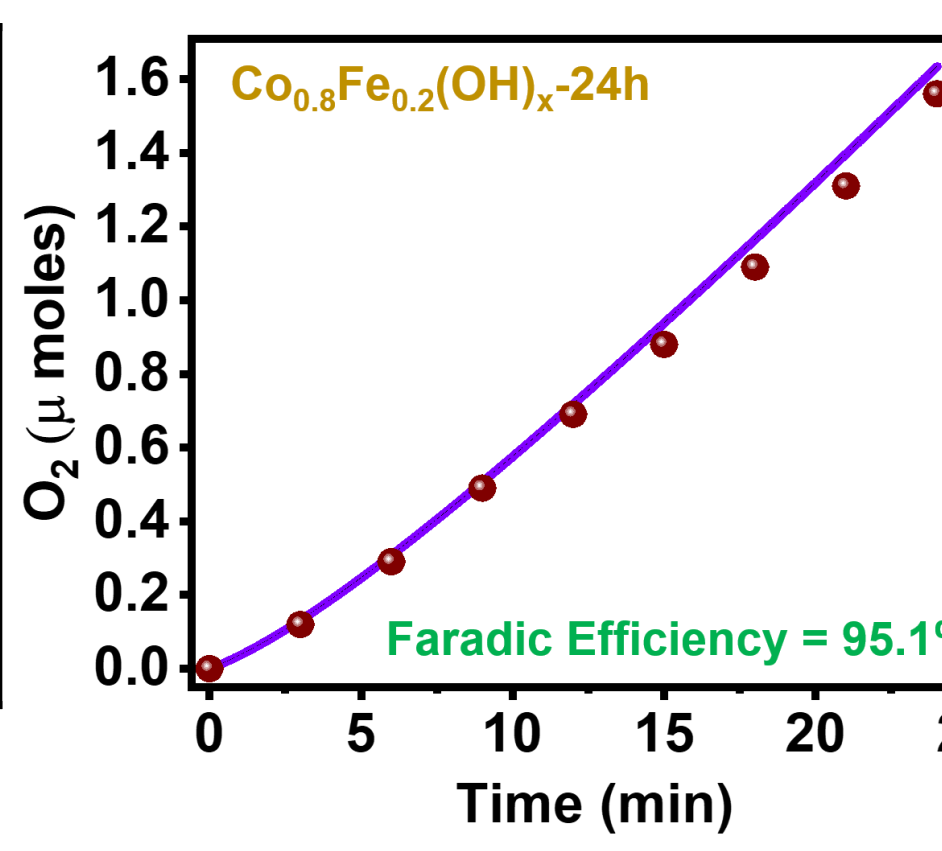


Sample	Overpotential (mV)	Tafel Slope (mV/dec)	Mass activity (A/g) @ $\eta=350$ mV	TOF (s ⁻¹)	R_{ct} (Ω)	R_p (Ω)
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -15 min	406	81	22.9	0.17	22.2	1438.0
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -1 h	395	75	27.8	0.21	19.2	729.8
$\text{Co}_{0.8}\text{Fe}_{0.2}(\text{OH})_x$ -4 h	370	64	74.8	0.56	17.7	271.0
CoFe20-LDH 12 h	346	56	191.8	1.41	7.4	45.9
CoFe20-LDH 24 h	330	50	309.1	2.28	5.9	35.3
CoFe20-LDH 36 h	348	62	169.5	1.26	7.6	66.8

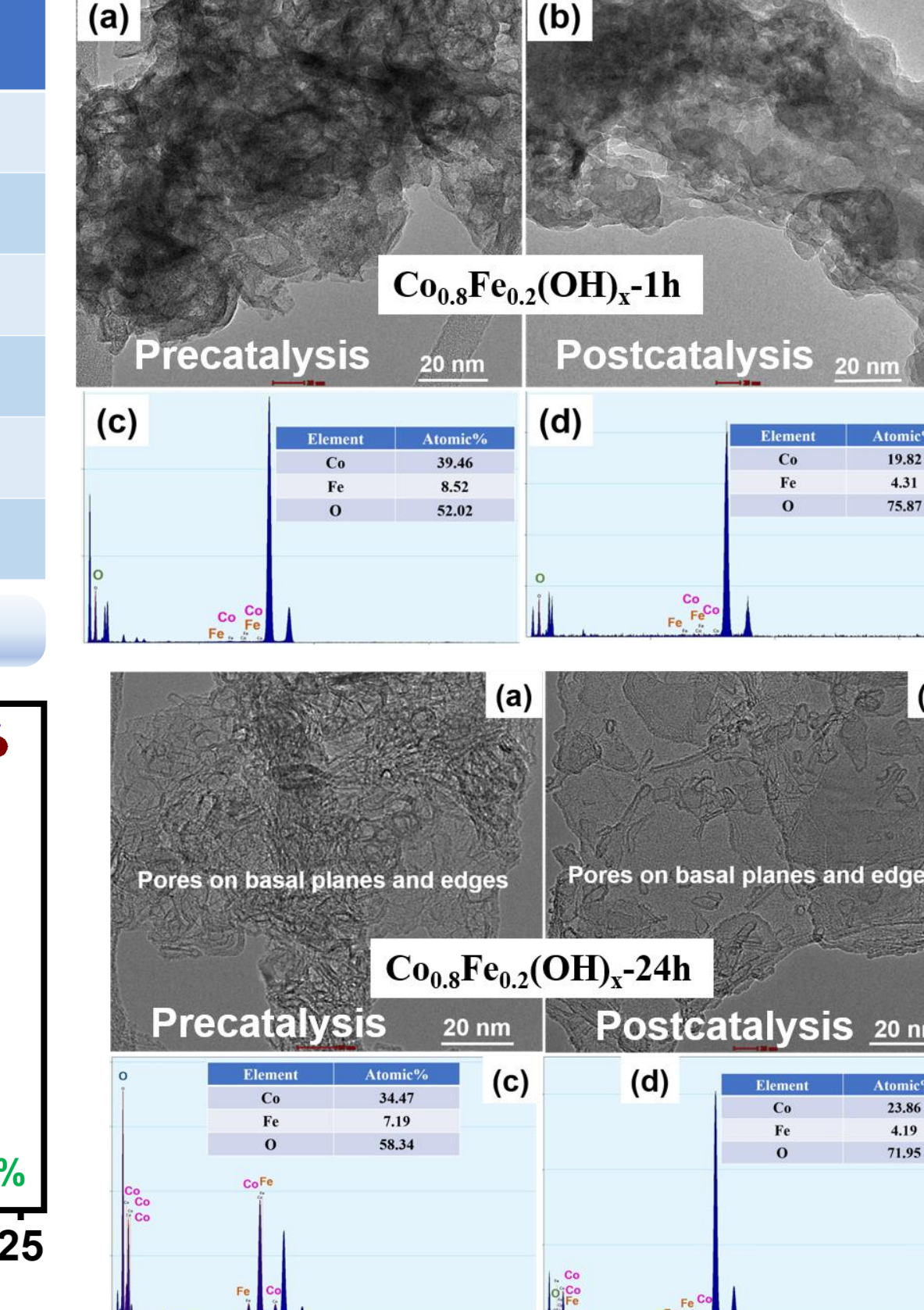
Stability Test



Product Confirmation



TEM Results



Conclusions

- ❖ Ultrathin CoFe-nanomesh for OER highlights the significant role of pore morphology in enhancing catalytic performance and stability
- ❖ It emphasizes how manipulating pore shapes at the nanoscale can lead to better catalysts for energy applications

Acknowledgments

- ❖ Financial support from SERB and CSIR, New Delhi
- ❖ Commonwealth Chemistry Committee

References

1. S. Sharma, A. Paul, ACS Appl. Nano Mater. 2024, 7, 8567–8579
2. S. Sharma, A. Paul, Chem Asian J. 2024, e202401156