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Microwave-Assisted Synthesis of ZIF-8 and the Influence of the Reactant-to-Solvent Ratio on CO_2 Adsorption

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Introduction

Metal-Organic Frameworks (MOFs) such as ZIF-8 stand out due to their high thermal/chemical stability and microporosity, making them benchmark materials for CO_2 capture [1]. Microwave-assisted synthesis offers a rapid and efficient fabrication route; however, crystal growth and defect density depend critically on precursor concentration. This work evaluates the impact of solvent (DMF) reduction on ZIF-8 crystallization kinetics, correlating structural, morphological, and surface chemical properties.

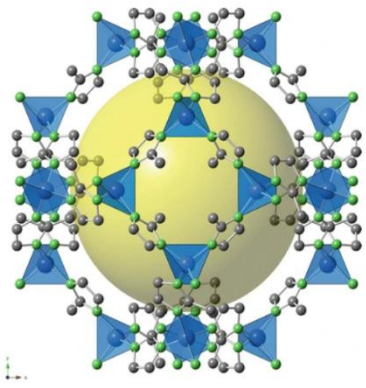


Figure 1. ZIF-8 framework: Zn (blue), C (black), N (green). H omitted. Yellow sphere: central pore. [2]

Methodology

ZIF-8 was synthesized based on Butova et al. [3].

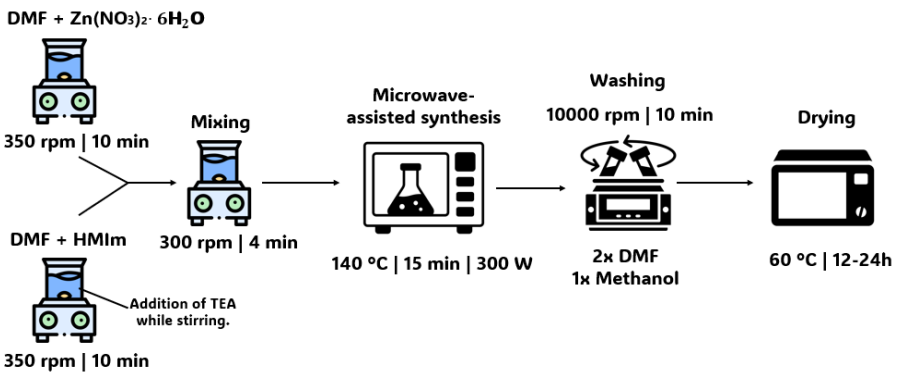


Figure 2. Schematic of the microwave-assisted synthesis method.

Sample	Zn^{2+}	HMIm	TEA	DMF	CI (%)
ZIF-8 (578 DMF)	1	2	3	578	84.6
ZIF-8 (288 DMF)	1	2	3	288	90.1
ZIF-8 (155 DMF)	1	2	3	155	84.9
ZIF-8 (78 DMF)	1	2	3	78	78.2

Table 1. ZIF-8 synthesis molar ratios.

Results and Discussion

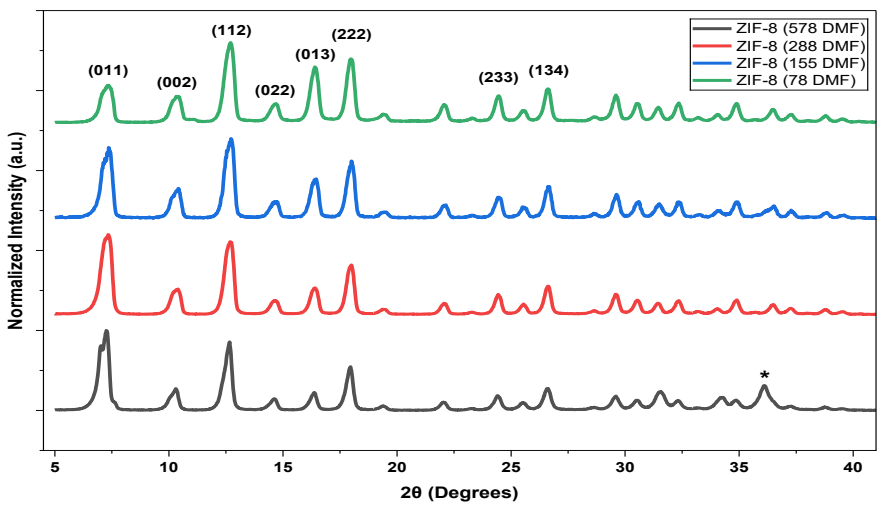


Figure 3. Normalized XRD patterns confirming the sodalite ZIF-8 phase in 15 min. Crystallinity Index (CI) peaks at 288 DMF (90.1%), followed by 155 DMF (84.9%), 578 DMF (84.6%, with trace ZnO*), and 78 DMF (78.2%, guest occlusion).

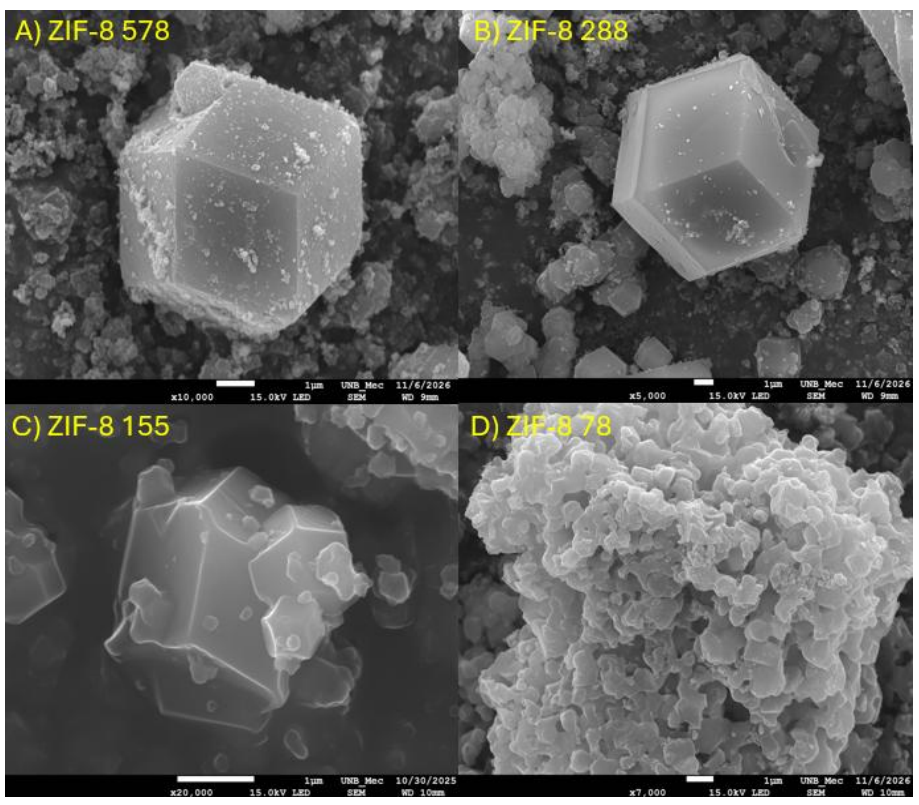


Figure 4. SEM morphological evolution: A) 578 DMF: rhombic dodecahedron with surface debris/ZnO. B) 288 DMF: clean-faceted dodecahedron. C) 155 DMF: intergrown polyhedra. D) 78 DMF: coalesced sub-micron aggregates.

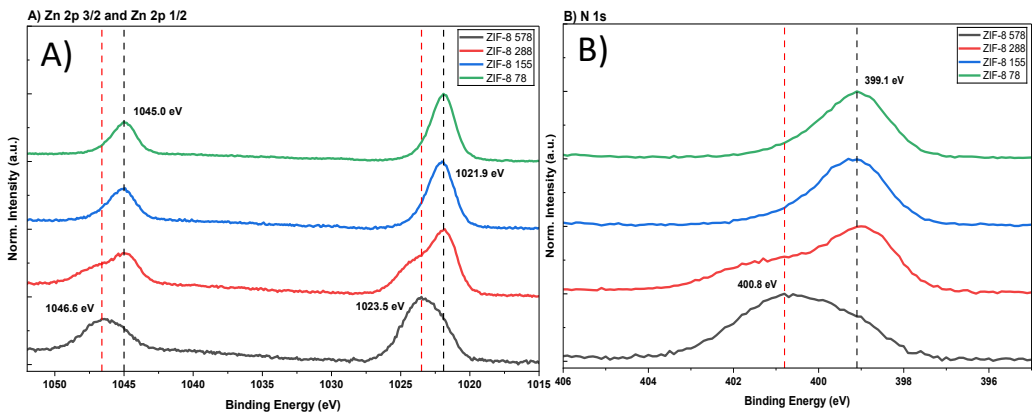


Figure 5. High-resolution XPS spectra of A) Zn 2p and B) N 1s. High dilution (578 DMF) yields broader, shifted peaks due to differential charging across debris-covered microcrystals and incomplete linker deprotonation (Zn-OH/ZnO and protonated -NH- shoulders). Increasing reactant/TEA concentration (155 and 78 DMF) narrows the peaks and triples N 1s intensity, implying Zn-N coordination.

Conclusions

Microwave synthesis rapidly produced crystalline ZIF-8 within 15 min. The DMF ratio regulates supersaturation and TEA deprotonation kinetics: high dilution (578 DMF) leaves uncoordinated –NH- and trace ZnO, whereas extreme supersaturation (78 DMF) triggers particle coalescence and pore occlusion (CI = 78.2%). Notably, 288 DMF maximizes crystallinity (90.1%), while 155 DMF cuts solvent use by 46% with preserved polyhedral morphology (84.9%).

References and Acknowledgements

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[3] BUTOVA, V. V. *et al*. **Mendeleev Commun.**, v. 26, n. 1, p. 43-44, 2016.

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