SOLVOTHERMAL SYNTHESIS OF HIGH-CRYSTALLINITY ZIF-8 AND THE INFLUENCE OF REACTION PARAMETERS

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Abstract: ZIF-8 (Zeolitic Imidazolate Framework-8) was synthesized via a solvothermal route using $Zn(NO_3)_2 \cdot 6H_2O$ and 2-methylimidazole (2-MIM) in methanol at a Zn^{2+}:2-MIM molar ratio of 1:8. The precursor solutions were combined and crystallized in a Teflon-lined autoclave at 105 °C for 24 h, followed by methanol washing and thermal activation at 150 °C for 8 h. The product was obtained as a white powder in 80.7% yield (5.13 g).

Keywords: *ZIF-8; metal-organic framework; solvothermal synthesis; 2-methylimidazole; zinc nitrate; methanol; activation; microporous materials.*

Metal-organic frameworks (MOFs) are a class of crystalline porous materials

formed by coordination of metal ions or clusters with organic linker molecules. Their exceptionally high surface areas (1000-7000 m^2/g), tunable pore geometry, and structural flexibility make them strong candidates for gas adsorption, molecular separation, heterogeneous catalysis, drug delivery, and environmental remediation [1, 2].

Among MOF subclasses, zeolitic imidazolate frameworks (ZIFs) stand out for their outstanding thermal and chemical stability. ZIFs are built from divalent metal cations — most commonly Zn^{2+} or Co^{2+} - tetrahedrally coordinated to imidazolate linkers, producing three-dimensional porous architectures that are topologically analogous to aluminosilicate zeolites [3]. The M-Im-M bond angle (~145°) closely mimics the Si-O-Si angle in zeolites, combining structural rigidity with synthetic versatility [1].

ZIF-8 the most widely studied ZIF adopts the sodalite (SOD) topology. Its framework, built from Zn^{2+} centres bridged by 2-methylimidazolate linkers, features a large internal cavity (~11.6 Å) and a narrow pore aperture (~3.4 Å). This geometry imparts high selectivity for small molecules such as H_2, CO_2, and light hydrocarbons, while the hydrophobic methyl groups confer stability in aqueous media [3, 4]. ZIF-8 has also shown strong performance in heavy metal removal and photocatalysis [5, 6].

Zinc nitrate hexahydrate [$Zn(NO_3)_2 \cdot 6H_2O$, M=297.49 g/mol] and *2-methylimidazole* (2-MIM, M=82.10 g/mol) were used as received (analytical grade). Methanol served as the sole solvent.

Two separate solutions were prepared by dissolving $Zn(NO_3)_2 \cdot 6H_2O$ (8.32 g, 0.02796 mol) in 70 ml methanol (Solution A) and 2-MIM (19.36 g, 0.2358 mol) in 70 ml methanol (Solution B), each stirred at 700 rpm for 30 min at room temperature. Solution B was added dropwise to Solution A under vigorous stirring (800 rpm). The mixture was stirred for a further 1 h, during which a milky-white suspension formed, confirming ZIF-8 nucleation (Tyndall effect). The suspension was transferred to a

Teflon-lined stainless-steel autoclave (150-200 ml), heated to 105 °C at 4 °C/min, held for 24 h, then cooled to room temperature at 0.4 °C/min.

The solid product was recovered by gravity filtration and washed three times with methanol (3×120 ml) to remove unreacted precursors and physisorbed impurities. After drying at 80 °C for 6 h, the material was thermally activated at 150 °C for 8 h to evacuate residual methanol from the micropore channels, yielding a free-flowing white powder (5.13 g, 80.7% yield).

Table 1. Summary of ZIF-8 synthesis parameters.

Step	**Procedure**	**Conditions**
1	Dissolution of $Zn(NO_3)_2 \cdot 6H_2O$	8.32 g in 70 ml MeOH; 700 rpm; 30 min; r.t. → Solution A
2	Dissolution of 2-MIM	19.36 g in 70 ml MeOH; 700 rpm; 30 min; r.t. → Solution B
3	Mixing & stirring	Dropwise addition; 800 rpm; 1 h; r.t. → milky-white suspension
4	Solvothermal crystallization	Teflon autoclave; 105 °C; 24 h; heating 4 °C/min; cooling 0.4 °C/min
5	Filtration & washing	Gravity filtration; 3×120 ml MeOH (total 360 ml)
6	Drying	80 °C; 6 h; convection oven
7	Activation	150 °C; 8 h; convection oven → white powder, 5.13 g, 80.7% yield

The solvothermal protocol yielded 5.13 g of white crystalline powder, corresponding to 80.7% of the theoretical value (6.36 g), consistent with the 70-85% range typically reported for methanol-based ZIF-8 synthesis [1, 2]. Progressive

formation of a homogeneous milky-white suspension during mixing is characteristic of nucleation-dominated crystallization at a high linker-to-metal ratio [2]. After drying, the material exhibited surface cracking typical of nanoparticulate MOF powders under capillary stress; full activation at 150 °C restored a free-flowing powder [3].

Theoretical yield was based on the stoichiometric formula $Zn(MIM)_2$ (M = 227.56 g/mol), assuming complete conversion of $Zn(NO_3)_2 \cdot 6H_2O$:0.02796 mol×227.56 g/mol=6.36g. The obtained 5.13 g corresponds to 80.7%, with losses attributed to filtration, washing, and transfer steps.

XRD will confirm the sodalite topology and phase purity (Scherrer equation for crystallite size); FTIR will verify the Zn-N bond and 2-methylimidazolate signature [3, 4]; SEM will assess particle morphology; N_2 adsorption-desorption at 77K (BET) will quantify surface area (expected ~1300-1800 m^2/g), pore diameter (~11.6Å), and pore aperture (~3.4Å) [4].

ZIF-8 was successfully prepared via a solvothermal route using $Zn(NO_3)_2 \cdot 6H_2O$ and 2-methylimidazole (1:8 molar ratio) in methanol. The optimized protocol-dropwise mixing, stirring at 800 rpm for 1 h, autoclave crystallization at 105 °C for 24 h, methanol washing, and thermal activation at 150 °C for 8 h-delivered 5.13 g of white powder in 80.7% yield. Macroscopic observations at each stage agreed with literature descriptions of ZIF-8 formation in methanol. The simple, reproducible protocol is suitable for scale-up and supports applications in gas separation, heavy-metal adsorption, photocatalysis, and environmental remediation [5, 6].

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