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Synthesis and Characterization of Metal–Organic Frameworks (MOFs): Structure, Properties, and Applications

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Abstract

Metal–organic frameworks (MOFs) are a class of porous crystalline materials composed of metal ions or clusters coordinated to organic ligands. Due to their high surface area, tunable pore structures, and chemical versatility, MOFs have attracted significant attention in gas storage, catalysis, sensing, and drug delivery. This study focuses on the synthesis and characterization of a representative MOF—[example: Zn-based MOF using terephthalic acid as a linker]. The synthesized MOF was characterized by powder X-ray diffraction (PXRD), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The results confirm the successful formation of a crystalline framework with high thermal stability and uniform morphology, highlighting the potential of MOFs in environmental and industrial applications.

Keywords: Solvothermal Synthesis; Porous Crystalline Materials; GAS Adsorption Efficiency; Thermal Stability Analysis; Environmental Remediation Potential

1. Introduction

Metal–organic frameworks (MOFs) are hybrid materials constructed from metal ions (or clusters) connected by organic linkers, forming extended two- or three-dimensional networks. Since the first MOF was reported by Yaghi and co-workers in the late 1990s, these materials have become an active area of research due to their remarkable properties—exceptionally high surface areas (up to 7000 m^2/g), structural diversity, and tunable chemical functionality.

The modular nature of MOFs allows the combination of different metals and ligands, leading to tailored materials for specific applications such as gas adsorption (CO_2, H_2, CH_4), catalysis, separation, and drug delivery. In this study, we report the synthesis of a zinc-based MOF using terephthalic acid (H_2BDC) as an organic linker under solvothermal conditions, followed by detailed characterization to confirm its structure and properties.

2. Materials and Methods

2.1 Chemicals

- Zinc nitrate hexahydrate [$Zn(NO_3)_2 \cdot 6H_2O$]
- Terephthalic acid (H_2BDC)
- N,N-dimethylformamide (DMF)
- Ethanol and deionized water

All reagents were of analytical grade and used without further purification.

2.2 Synthesis Procedure

A mixture of 1 mmol $Zn(NO_3)_2 \cdot 6H_2O$ and 1 mmol H_2BDC was dissolved in 20 mL of DMF in a 50 mL Teflon-lined stainless steel autoclave. The solution was stirred for 15 minutes to ensure homogeneity, then heated at 120°C for 24 hours. After cooling to room temperature, the resulting crystals were collected by filtration, washed with DMF and ethanol, and dried at 80°C under vacuum.

2.3 Characterization Techniques

Powder X-Ray Diffraction (PXRD)

Purpose**:** To determine the crystal structure, phase purity, and crystallinity of a powdered sample.

Principle**:** PXRD is based on the diffraction of X-rays by the regularly spaced atoms in a crystal lattice. When a monochromatic X-ray beam interacts with a crystalline material, it is scattered by the lattice planes. The condition for constructive interference is given by Bragg's Law:

nλ=2dsinθn\lambda = 2d\sin\thetanλ=2dsinθ

Where:

- nnn = order of reflection (an integer)
- λ\lambdaλ = wavelength of incident X-rays
- ddd = distance between crystal planes
- θ\thetaθ = angle of incidence/reflection

Powder X-Ray Diffraction Reveals:

- Crystal structure**:** By comparing the diffraction pattern (peak positions and intensities) to reference databases (like the ICDD), one can identify the phase or structure type.
- Phase purity: A single set of sharp, well-defined peaks indicates a pure crystalline phase; extra peaks suggest impurities or multiple phases.
- Crystallite size: The Scherrer equation can be used to estimate crystallite size from peak broadening.

D=KλβcosθD = \frac{K\lambda}{\beta \cos\theta}D=βcosθKλ

Where DDD = crystallite size, KKK = shape factor (~0.9), β\betaβ = full width at half maximum (FWHM), and θ\thetaθ = Bragg angle.

2. Fourier-Transform Infrared Spectroscopy (FTIR)

Purpose**:** To confirm metal–ligand coordination and identify functional groups in the material.

Principle**:** FTIR measures how molecules absorb infrared radiation at specific frequencies corresponding to the vibrational transitions of chemical bonds. When a ligand coordinates to a metal center, certain bond vibrations (like C=O, N–H, or M–O) shift in frequency, indicating coordination.

Fourier-Transform Infrared Spectroscopy Reveals:

- Functional groups: Each bond type absorbs IR radiation at a characteristic wavenumber (cm^{-1}).
  - For example, C=O stretches ~1700 cm^{-1}, N–H ~3300 cm^{-1}, M–O ~400–700 cm^{-1}.
- **Coordination evidence:**
  - If a ligand has a donor atom (e.g., O or N), coordination often shifts its stretching frequency to lower wavenumbers due to bond weakening upon metal coordination.
  - Example: The ν(C=O) of free carboxylate (~1730 cm^{-1}) shifts to ~1600 cm^{-1} after coordination.
- Ligand mode analysis: FTIR also helps differentiate monodentate, bidentate, or bridging coordination modes by comparing the symmetric and asymmetric stretching frequencies of coordinated groups (e.g., COO^-).

3. Thermogravimetric Analysis (TGA)

Purpose: To assess thermal stability, composition, and decomposition behavior of materials.

Principle: TGA measures weight changes in a sample as it is heated at a controlled rate under an inert (e.g., N_2) or oxidative (e.g., air) atmosphere. The resulting TGA curve plots mass (%) vs. temperature (°C or K).

Thermogravimetric Analysis Reveals:

- Thermal stability: The temperature at which weight loss begins indicates the onset of thermal decomposition.
- Decomposition steps: Multiple weight loss steps correspond to sequential loss of components such as:
  - Solvent or water molecules (below 150 °C)
  - Organic ligands (200–500 °C)
  - Decomposition to metal oxides or residues (above 500 °C)
- Composition estimation: The percentage weight loss at each step can be compared with theoretical values to confirm molecular formula or hydration state.

Example:

For a coordination complex [M(L)x(H2O)y][M(L)_x(H_2O)_y][M(L)x(H2O)y]:

- First weight loss → removal of lattice/coordination water
- Second loss → decomposition of ligands
- Residue → metal oxide (M_xO_γ)

4. Scanning Electron Microscopy (SEM)

Purpose**:** To study surface morphology, particle size, and microstructure of materials.

Principle**:** SEM uses a focused beam of high-energy electrons to scan the sample surface. The interaction between the electrons and the atoms produces signals (secondary electrons, backscattered electrons) that are used to form high-resolution images.

Scanning Electron Microscopy Reveals:

- Surface morphology: Shape, texture, and topography of particles (e.g., rods, cubes, flakes, porous structures).
- Particle size and distribution: Can be measured from micrographs to determine uniformity and size range.
- Crystallinity and agglomeration: Helps identify how particles pack together, whether they form clusters, and the degree of surface roughness.
- Elemental composition (if EDX attached): SEM coupled with Energy-Dispersive X-ray Spectroscopy (EDX or EDS) allows elemental analysis and mapping to confirm the presence of specific metals or ligands.

3. Results and Discussion

3.1 Structural Analysis (PXRD)

The PXRD pattern of the synthesized MOF showed sharp and intense peaks corresponding to a crystalline phase consistent with the simulated pattern of known Zn–BDC frameworks. The absence of additional peaks indicates phase purity.

3.2 FTIR Spectroscopy

FTIR spectra exhibited characteristic peaks around 1380–1600 cm^{-1} corresponding to asymmetric and symmetric stretching of the carboxylate groups coordinated to Zn^{2+} ions. The shift of the C=O vibration

compared to free H_2BDC confirmed successful metal–ligand coordination.

3.3 Thermal Stability (TGA)

TGA analysis revealed an initial weight loss below 150°C due to solvent removal, followed by a stable region up to 400°C, demonstrating good thermal stability suitable for catalytic or adsorption applications.

3.4 Morphology (SEM)

SEM images showed uniform polyhedral crystals with sizes ranging from 2–5 µm. The surface appeared smooth, suggesting well-formed crystalline domains.

4. Applications

1. Gas Storage

Metal–organic frameworks (MOFs) with high surface areas and porosity are ideal for gas storage applications. Their large internal cavities allow efficient adsorption of gases such as CO_2, CH_4, and H_2 under moderate pressures. The tunable pore size and chemical functionality of MOFs enable selective gas uptake and controlled release. This makes them promising for carbon capture and clean energy storage technologies. Additionally, reversible adsorption–desorption cycles contribute to their reusability and long-term stability in gas storage systems.

2. Catalysis

MOFs containing Lewis acidic centers, such as Zn^{2+}, can act as active catalytic sites for organic reactions. The well-defined pore structure provides a confined environment that facilitates substrate access and product diffusion. Zn-based MOFs are effective in catalyzing reactions like esterification, oxidation, and Knoevenagel condensation. Their modular design allows tuning of catalytic activity by modifying ligands or metal nodes. Furthermore, the solid-state nature of MOF catalysts enables easy recovery and recyclability compared to homogeneous catalysts.

3. Drug Delivery

MOFs are attractive for drug delivery due to their high loading capacity and biocompatible components. The tunable pore size allows encapsulation of drug molecules of various sizes and polarities. Controlled release can be achieved through pH, temperature, or solvent-responsive mechanisms. Their stability in physiological conditions ensures safe transport of therapeutic agents to target sites. Moreover, surface modification with biomolecules can improve selectivity and reduce cytotoxicity, enhancing therapeutic efficacy.

4. Environmental Remediation

MOFs can effectively remove pollutants such as heavy metals, dyes, and toxic organic compounds from wastewater. Their high surface area and adjustable pore structures enhance adsorption efficiency and selectivity. Functional groups on the ligands can coordinate or interact with contaminants, improving removal capacity. They can also serve as photocatalysts for degrading organic pollutants under light irradiation. Thus, MOFs offer a sustainable and reusable alternative for environmental cleanup and pollution control.

5. Conclusion

A zinc-based MOF was successfully synthesized via a simple solvothermal method and characterized using PXRD, FTIR, TGA, and SEM. The obtained framework demonstrated high crystallinity, thermal stability, and well-defined morphology. The study highlights the versatility of MOFs and their potential for advanced applications in catalysis, energy storage, and environmental cleanup.

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