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Research Progress on Synthesis and Applications of Metal-Organic Frameworks

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Abstract. Metal-organic frameworks (MOFs) are a family of crystalline porous materials featuring large specific surface areas, tunable pore geometries, and structurally designable architectures, which endow them with tremendous application potential in adsorption, separation, and catalysis. This article systematically reviews the structural properties, state-of-the-art synthetic strategies, and pore activation techniques of MOFs. Representative synthetic routes, including hydrothermal/solvothermal, electrochemical, external-field-assisted, and mechanical synthesis methods, are comprehensively compared in terms of their technical characteristics. The underlying mechanisms by which diverse activation procedures regulate pore purification, framework structural stability, and functional performance improvement of MOFs are clearly elucidated. In addition, this work summarizes the research advances of MOFs in diverse application scenarios, covering gas separation, water treatment, organic catalysis, and photocatalysis, with a particular focus on establishing their structure–performance relationships. Finally, the industrial application bottlenecks and future green development directions are discussed, aiming to guide the design and optimization of high-performance MOF materials.

Keywords: metal-organic frameworks, synthesis, adsorption and separation, catalysis

1. Introduction

Metal-organic frameworks (MOFs) represent crystalline porous solids constructed when metal ions or metal clusters coordinate with organic linkers to form self-assembled networks [1]. Progress in MOF research has greatly advanced the innovation of modern porous materials. Relying on the "molecular building-block" strategy, MOFs overcome long-standing limitations that restricted simultaneous structural and functional tuning in traditional porous substances. This feature makes MOFs a powerful experimental platform to tailor functional materials at molecular level and decode how material structures govern their practical behaviors. Four distinctive structural traits account for MOFs' dominant position within porous-material research. To begin with, their extraordinarily large surface area and pore volume create ample space for molecular adsorption and mass transport. Second, pore dimensions (ranging from 0.5-10 nm) and topological configurations can be precisely adjusted to achieve selective identification and separation of target molecules. Third, plentiful

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reactive centers originating from metal nodes, ligand skeletons and surface functional groups supply multiple reaction sites for catalytic transformations. Finally, flexible structural tailor ability enables researchers to modify material physicochemical properties by rationally pairing metal secondary building units with organic linkers [1, 2]. Even with these appealing merits, practical deployment of MOFs still faces multiple challenges. For instance, many high-performance MOF frameworks suffer poor water-resistant stability, which severely hinders their large-scale real-world implementation.

Synthetic routes and post-synthetic activation approaches follow varied reaction pathways and kinetic behaviours, and these factors collectively shape MOF crystals in terms of morphology, crystallinity, pore architecture and structural robustness [1, 3]. This review covers five mainstream fabrication approaches: hydrothermal, solvothermal, electrochemical, ultrasonic-assisted and mechanochemical synthesis. We analyse how these fabrication conditions alter MOF structures and performances, with the goal of supporting the targeted development of MOFs for designated functional scenarios. Most laboratory-level fabrication protocols are difficult to scale-up. Therefore, developing cost-effective, green synthesis-activation workflows remains a core challenge for MOF researchers. Beyond synthesis, post-synthetic activation is indispensable to realise MOFs' full application capacity. Techniques including solvent exchange, thermal treatment and supercritical CO_2 processing exert major influences over adsorption-separation efficiency and catalytic activity [4]. We further elaborate on working mechanisms and feasible optimisation routes for these three activation protocols, hoping to offer practical references for boosting MOFs' real-world performance. Adsorption-separation and catalysis stand out as the two most promising application domains for MOFs, where their modifiable porous architectures deliver remarkable functional outputs [5-7].

Taken together, MOFs deliver high research significance for fundamental studies whilst showing great promise toward real-world industrial deployment. This paper comprehensively discusses MOFs' structural features, fabrication protocols, activation strategies as well as their performance in adsorption and catalysis. Special emphasis is placed on unravelling the inherent correlations between synthesis-activation workflows, material structures, functional properties and end-use behaviours. This work expects to lay a holistic theoretical foundation for MOF structural optimisation, performance promotion and industrial translation, and meanwhile inspire further exploration of innovative high-performance porous functional materials.

2. Preparation and activation methods for MOF materials

2.1. Synthesis methods for MOF materials

2.1.1. Hydrothermal/solvothermal methods

The hydrothermal/solvothermal method was the first to be employed and remains the most widely used method for the preparation of MOFs [2]. The 'hydrothermal method' refers to any homogeneous or heterogeneous chemical reaction occurring in the presence of water at temperatures above the boiling point and pressures greater than 1 atmosphere; whereas the 'solvothermal method' refers to chemical reactions carried out in the presence of organic solvents (protonic or non-protonic solvents) [8, 9]. This technique features high yield, low operating temperature and extremely low reaction pressure [10, 11]. . The basic principle involves dissolving metal salts and organic ligands in water or an organic solvent, placing the mixture in a sealed reaction vessel, and conducting a thermal reaction under autogenous pressure to promote the formation of crystalline MOF products [1, 4] (Figure 1).The main advantages of this method lie in the fact that high-temperature and high-

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pressure conditions significantly increase the solubility of the reactants and promote crystal nucleation and growth, thereby yielding MOF materials with high crystallinity and good phase purity.

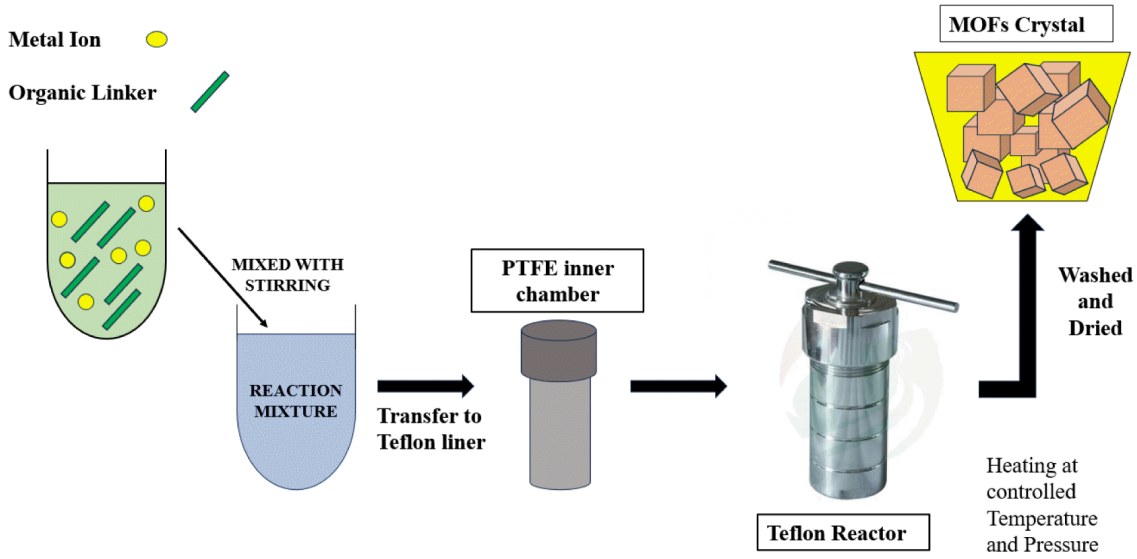


Figure 1. Schematic diagram of the general process flow for the preparation of MOFs via the hydrothermal/solvothermal method [12]

The solvothermal method is particularly effective in controlling the morphology and size of MOF crystals. By adjusting parameters such as reaction temperature, reaction time, solvent polarity and pH, the rates of crystal nucleation and growth can be effectively controlled [13]. Furthermore, the choice of solvent has a decisive influence on the stability of the MOF framework—the use of high-boiling-point polar solvents such as DMF and DEF favours the formation of MOF structures with high connectivity and high stability [14].

2.1.2. Electrochemical synthesis

Electrochemical synthesis is a novel synthetic technique in the field of MOF preparation. This technique replaces traditional metal salts with an anodic metal plate. Upon applying an electric current, metal ions are released and self-assemble with organic ligands to form MOF crystals [1, 3, 4]. The morphology and purity of the product are regulated by various parameters, including voltage, current, solvent, ligands and electrolyte concentration [15]. Compared with the solvothermal method, electrochemical synthesis offers distinct advantages: the reaction is completed swiftly at ambient temperature and pressure, significantly shortening the synthesis cycle; no salt by-products are generated throughout the process, simplifying the product purification procedure. Moreover, the grain size, microstructure and defect density of MOFs can be precisely tuned by modifying electrical parameters and electrolyte composition. Nevertheless, this method exhibits obvious application constraints. Synthetic process strongly relies on the machinability and electrochemical activity of metal electrodes. So far, only a handful of MOFs (e.g., Cu- and Zn-based MOFs) can be stably obtained, while this technique is hardly applicable to high-melting-point metal systems including zirconium and chromium [16, 17].

DOI: 10.54254/2755-2721/2026.CH37774

2.1.3. Ultrasonic and microwave-assisted methods

Both ultrasonic- and microwave-assisted methods leverage external-field enhancement for rapid MOF synthesis (Figure 2), with core advantages of shortened reaction time and improved synthetic efficiency [18]. The ultrasonic method utilizes the liquid-phase cavitation effect to enhance mass transfer and mixing of reactants and reduce the crystal nucleation barrier; it also enables the preparation of MOF materials with special morphologies that are challenging to obtain via conventional processes.

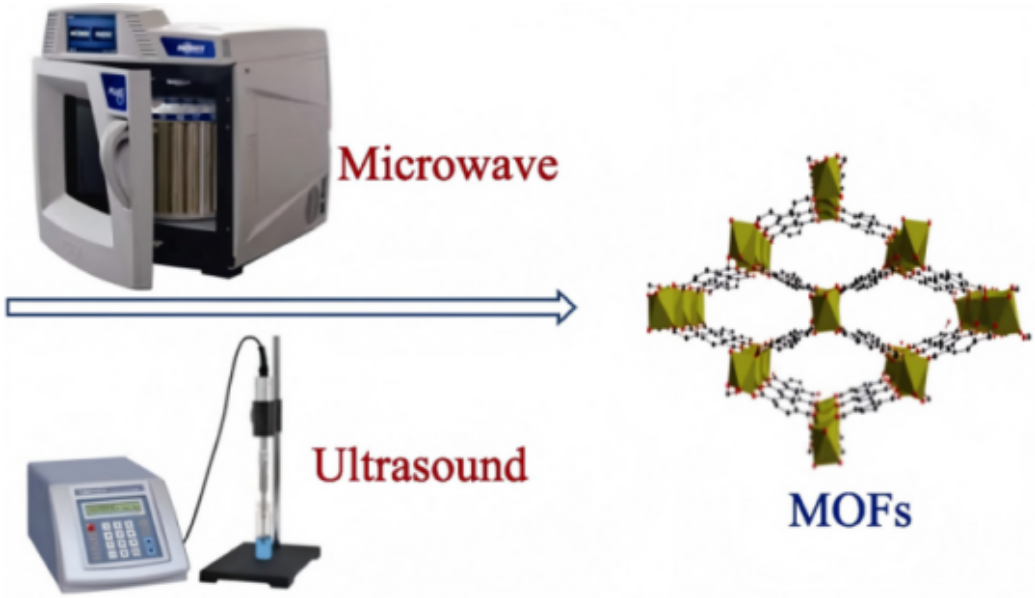


Figure 2. Schematic illustration of MOF synthesis via ultrasonic and microwave methods [18]

The microwave-assisted method utilizes the selective heating of polar molecules by microwaves to achieve uniform heating of the system, thereby avoiding the temperature gradients associated with traditional heat conduction. Featuring fast reaction rates, uniform product particle sizes and high energy utilization, it enables the rapid preparation of photocatalytic MOFs such as MIL-53 (Fe), and is currently also used for the large-scale production of MOF derivatives. Although external-field synthesis processes offer excellent synthesis efficiency, there are significant limitations to their large-scale application: the frequency and intensity of ultrasonication directly affect product performance. Meanwhile, differences in microwave equipment and radiation energy can also significantly alter the reaction outcomes [3].

2.1.4. Mechanical synthesis

The mechanical synthesis method is a green and efficient process for MOF preparation and has attracted considerable research interest in recent years [19]. This method relies on the mechanical force generated by ball milling and grinding to drive the coordination reaction between solid raw materials. This reaction does not require a solvent or only requires a small amount of solvent, and has the advantages of simple operation, fast reaction rate and low pollution. The synthesis process is as follows: mix the metal salt and organic ligand in the appropriate proportion and put them into the ball mill. The impact and friction force of the grinding balls provide the energy required for the reaction, thereby generating MOFs through a solid-phase reaction (Figure3). This solid-phase

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synthesis method enables the mechanical synthesis method to possess prominent advantages of environmental friendliness and low cost [3, 4].

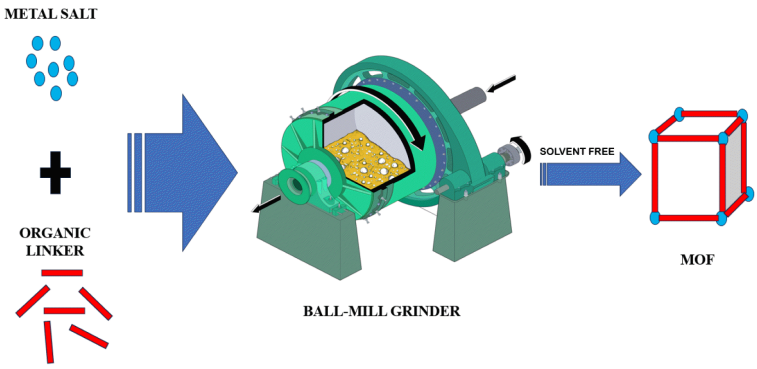


Figure 3. Schematic diagram of the mechanical synthesis of MOFs [12]

2.2. Activation methods for MOF materials

2.2.1. Solvent exchange method

Solvent-exchange coupled with vacuum-assisted heating represents a commonly adopted activation strategy for MOFs. Acetone and dichloromethane are widely selected for this procedure. These low-boiling-point and low-surface-tension solvents displace high-boiling residues within MOF cavities and partly strip coordinated solvent molecules [20]. After displacement, mild heating or vacuum conditions are applied to fully evaporate the exchanged solvent species, opening MOF channels and purifying internal pore environments [21]. Low-surface-tension solvents optimize gas-liquid interfaces inside pores and mitigate capillary stress during desolvation. Such an effect protects flexible MOF lattices against irreversible structural failure (Figure 4) [4]. This protocol is well-suited for high-porosity flexible MOFs. Nevertheless, large solvent consumption, time-intensive procedures and high costs limit its laboratory application and bring environmental concerns. Optimizing solvent-recycling processes can mitigate such environmental drawbacks.

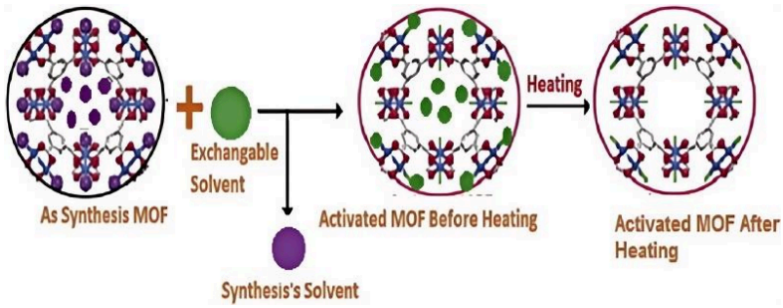


Figure 4. Solvent exchange activation method [4]

2.2.2. Thermal activation method

Thermal activation is a simple, low-cost method using atmospheric (inert) or vacuum heating to remove free and coordinated solvents, thereby opening MOF pores (Figure 5) [22]. Activation conditions depend on the material's thermal stability: thermally robust UiO-66 and MIL-101(Cr) tolerate high temperatures, whereas heat-sensitive MOF-5 requires strict temperature control to

DOI: 10.54254/2755-2721/2026.CH37774

avoid decomposition [4]. Balancing desolvation efficiency and framework stability is critical, as excessive temperature or duration can cause ligand degradation, bond cleavage, or framework collapse. Thermogravimetric analysis is typically used to optimize heating profiles and vacuum parameters to ensure both efficient desolvation and structural integrity [4].

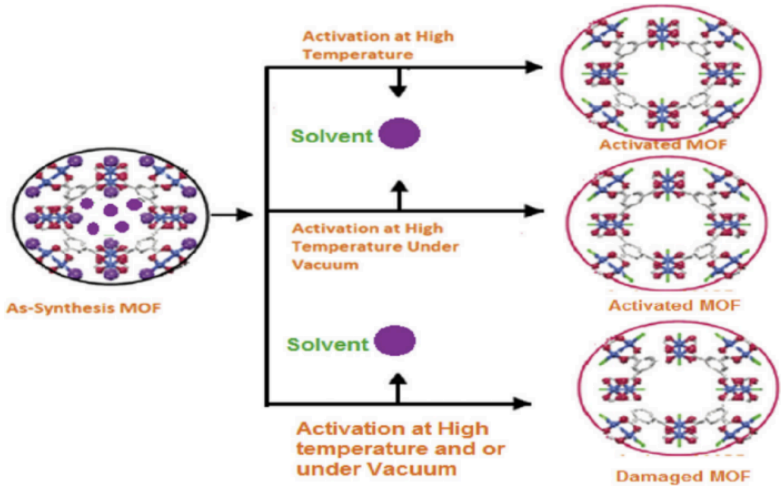


Figure 4. Solvent exchange activation method [4]

2.2.3. Supercritical CO2 activation method

Supercritical-CO_2-based activation stands out as a practical post-treatment option for MOFs, as it enables effective solvent removal under relatively gentle operating conditions [23]. Prior to supercritical treatment, ethanol is introduced within pores to displace high-boiling residual solvents trapped inside MOF cavities. Gradient decompression is implemented right after CO_2-mediated extraction, which removes capillary stress and thus protects fragile MOF frameworks against structural breakdown. This technique fits particularly well for ultra-microporous and flexible MOF structures. Materials processed in this manner can reach porosity values close to their theoretical limits, and recycled CO_2 supplies help cut down overall reagent expenditure. Even with these merits, broad adoption remains constrained for ordinary laboratory settings. The whole workflow demands specialized high-pressure sealing hardware, whose procurement and routine maintenance bring substantial cost burdens. For this reason, developing low-cost simplified supercritical-CO_2 setups would greatly promote laboratory-scale studies on fragile MOF materials.

3. Applications of MOF materials in adsorption and catalysis

Owing to the unique structural advantages of MOFs, the pore size, surface functional groups and active sites can be selectively modified by controlling the metal nodes and organic ligands, demonstrating outstanding application potential in the fields of adsorption and heterogeneous catalysis [24].

3.1. Applications of MOFs in adsorption

3.1.1. Gas adsorption and separation

Gas adsorption is the earliest and most mature research field of MOFs. Current studies focused primarily on industrial applications such as clean energy storage and the separation of low-carbon

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hydrocarbons. For clean energy storage, the hydrogen storage capacity of MOF-5 at 78 K is comparable to that of carbon nanotubes. NU-100 achieves a hydrogen adsorption capacity of 99.5 $mg \cdot g^{-1}$ at 77 K and 5.6 MPa [25]. For practical high-temperature operating conditions, a defect-modified Pt-de-MgMOF can achieve 2.55 wt% reversible hydrogen storage at 160 °C and 8 MPa, balancing both capacity and kinetic performance [26]. Due to the high compatibility between its pore size and methane molecules, IRMOF-6 exhibits excellent methane storage capacity under moderate pressure conditions (Figure 6), outperforming most crystalline porous adsorbents [25].

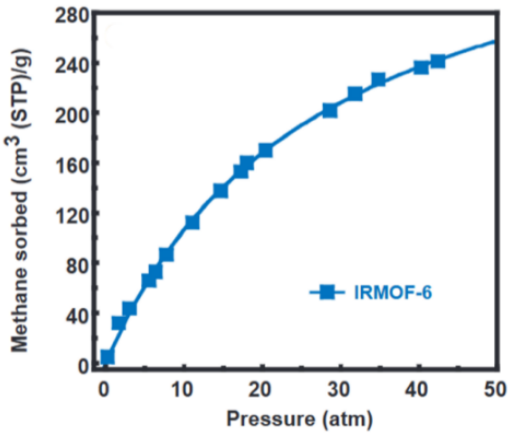


Figure 6. IRMOF-6's voluminous uptake of methane gas fitted at 298 K with the Langmuir equation [25]

Separation of light hydrocarbon mixtures represents a long-standing bottleneck within petrochemical manufacturing. UTSA-280 is notable for its scalable kilogram-level production using an aqueous route with calcium nitrate and formic acid feedstocks. Its one-dimensional pore geometry closely matches ethylene molecular dimensions and blocks ethane diffusion, yielding an IAST-predicted selectivity above 10 000. Satisfying practical-operation demands, this adsorbent can be fully regenerated simply by helium flushing at 353 K within 10 min. Such mild-cycle performance makes UTSA-280 a promising substitute for conventional cryogenic distillation, a process notorious for huge energy consumption [27]. Beyond powder-form adsorbents, composite membranes offer another feasible technical pathway. As a representative example, UiO-66-NH_2@ZIF-8 integrates macroporous transport pathways with microporous size-sieving effects, which permits removal of multiple contaminants in one-step treatment and shows encouraging prospects for real-world membrane manufacturing [28]. Even so, several practical hurdles remain: for UTSA-280, long-term cycling stability under real-world complex gas mixtures still needs further validation before industrial deployment.

3.1.2. Liquid-phase adsorption for water treatment

The pH and ionic strength of the aqueous environment pose severe challenges to the chemical stability of MOF frameworks. Currently, the well - studied stable systems are mainly focused on zirconium (Zr) - based UiO series, MOF - 808, and MIL series, which are widely used in the enrichment of organic dyes and the treatment of heavy metal pollution.In terms of organic dye adsorption, MOF -808 can be synthesized via a green aqueous route at room temperature. This material mainly relies on the synergistic effect of electrostatic attraction and π–π stacking to immobilize dye molecules. It exhibits a high saturated adsorption capacity of up to 1,500 mg/g for

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Congo Red and shows significant sieving selectivity for mixed dyes such as methyl orange and methylene blue (Figure 7) [29]. For the treatment of heavy metal ion pollution, the CF-FA-UiO-66 composite material constructed by insitu growing UiO-66-NH_2 on the surface of carboxylated cotton fabric effectively overcomes the engineering bottlenecks of traditional powder adsorbents, such as easy agglomeration and difficult recovery. Its adsorption capacity for Cr(VI) reaches 318.85 mg/g. Additionally, after covalently grafting EDTA chelating functional groups onto the MIL - 101 - NH_2 framework, its heavy metal removal efficiency is significantly improved. Notably, the spent materials after adsorption saturation can be directionally converted into iron - nickel (FeNi) - based oxygen evolution reaction (OER) electrocatalysts through pyrolysis and calcination, achieving high - value resource recycling of 'using waste to produce catalysts' [30].

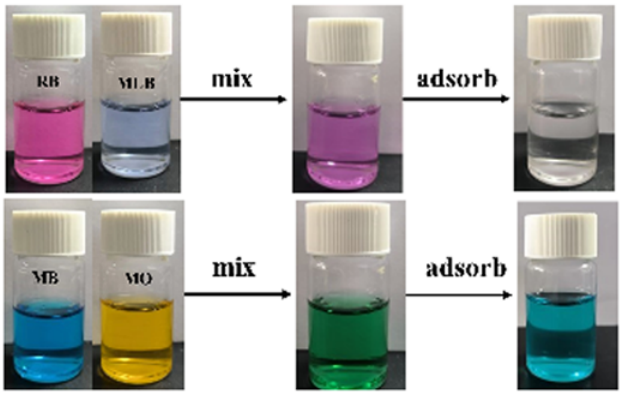


Figure 7. Digital photograph of adsorption measurements for mixed dyes: MLB (methyl blue)/RB (rose Bengal) and MO (methyl orange)/MB (methyl blue) [29]

3.2. Applications of MOFs in catalysis

3.2.1. Organic catalysis

The active sites in MOF-mediated organic catalysis include unsaturated metal sites within the framework, acidic functional groups on the ligands, and metal nanoparticles confined within the pore channels. They are suitable for reactions such as condensation, cycloaddition and asymmetric synthesis [31]. SOM-ZIF-8 possesses a three-dimensionally ordered macroporous–microporous single-crystal structure. Its multi-level pore channels significantly enhance mass transfer efficiency, and its activity and cycle stability in the Knoevenagel condensation reaction are superior to those of ordinary polycrystalline or hollow ZIF-8 [32]. CdS nanoparticles were grown in situ on a Zr-based MOF-808 support; the pore confinement effect shortened the electron transport path and suppressed the recombination of photo-generated electrons and holes, resulting in a visible-light hydrogen evolution rate 60 times higher than that of samples with simple physical loading [33].

3.2.2. Photoelectrocatalysis

Most MOFs possess a semiconductor-like electronic structure, with photo-generated electrons and holes driving reduction and oxidation reactions, respectively, enabling photocatalytic water splitting for hydrogen production, CO_2 reduction and pollutant degradation [34]. The porphyrin-based PCN series exhibits broad-range visible-light absorption capabilities, whilst Zr-Ni bimetallic PCNs can simultaneously achieve the coupling of aromatic alcohol oxidation with water splitting to produce

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hydrogen, without the need for a sacrificial agent. By encapsulating the metal nanocluster MAg_{25}within the Z-type heterojunction $AuAg_{25}$@UiO-66-$NH_{2,}$constructed from the pore channels$_{of}$UiO-66-$NH_{(2)}$, the charge separation efficiency was significantly optimized, with the hydrogen production rate reaching 5.8 times that of a single-metal system [35].

Conductive MOFs, bimetallic MOFs and heteroatom-doped MOFs are mainstream electrocatalytic materials, widely used in water electrolysis, CO_2 electro-reduction, solid-state electrolytes and energy storage electrodes. Luo et al. employed a strategy to modulate the electronic microstructure of iron-based bimetallic MOFs (MFe-BDC, where M = Mg, Zn, Cd) grown on a nickel foam (NF) substrate, utilizing them as bifunctional electrocatalysts for the oxygen evolution reaction (OER) and hydrogen evolution reaction (HER).Through rational regulation of Group IIA and IIB metal cations, an optimized bimetallic CdFe-BDC material was obtained, which exhibited outstanding OER and HER catalytic performance: an OER overpotential as low as 290 mV at a current density of 100 mA·cm^{-2} [36] (Figure 8).

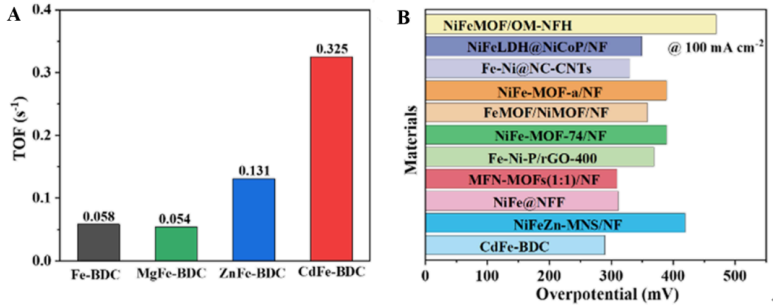


Figure 8. (A) TOF values at an overpotential of 280 mV for Fe-BDC, MgFe-BDC, ZnFe-BDC and CdFe-BDC. (B) Comparison with other recently reported advanced OER electrocatalysts [36]

4. Conclusion

In summary, metal-organic frameworks (MOFs), as a class of highly structurally tunable crystalline porous materials, require systematic coordination across the three stages of synthesis, activation and application to achieve performance-oriented, precise construction. At the synthesis stage, hydrothermal and solvothermal methods remain the mainstream approaches for obtaining highly crystalline MOFs, whilst electrochemical, ultrasound- and microwave-assisted, and mechanical synthesis methods excel in rapid synthesis, improved energy efficiency and green, large-scale production, respectively, providing diverse options to meet differentiated process requirements. At the activation stage, solvent exchange, thermal activation and supercritical CO_2 activation are suitable for framework systems with varying thermal stability, with the core objective being to maximize the exposure of pore channels and active sites. At the application stage, MOFs, owing to their pore sieving capabilities and multi-site synergistic effects, demonstrate outstanding performance in fields such as gas adsorption and separation, water treatment, organic catalysis, photocatalysis and electrocatalysis.

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