

Rational Design and Development of Metal-Organic Frameworks

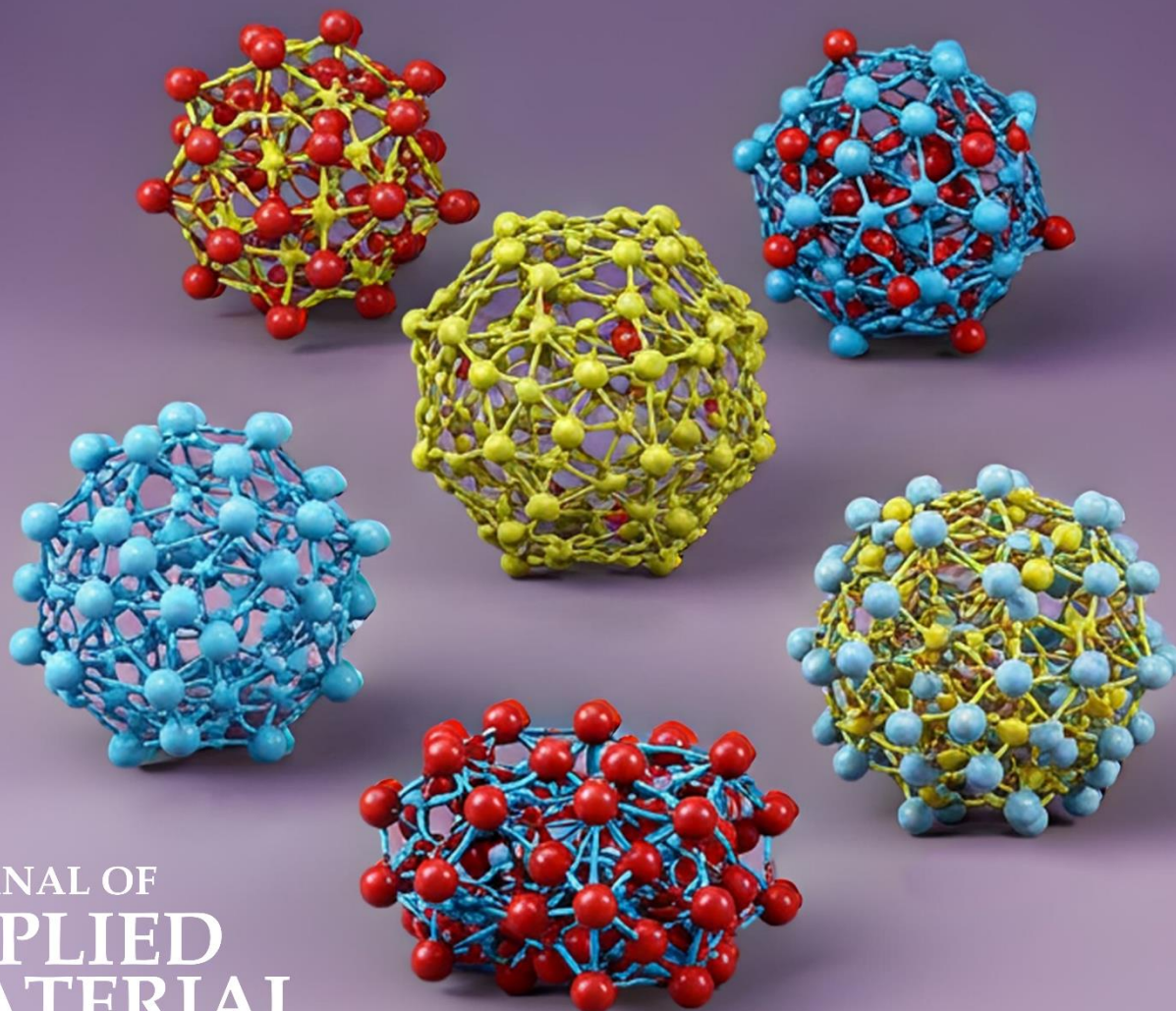
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Editor's note: Metal-organic frameworks (MOFs) are an innovative class of hybrid materials that hold great promise for tackling energy and environmental challenges. This invited mini-review provides an overview of recent developments by Ghasempour regarding the rational design of MOF topology. These advancements have significantly improved the catalytic, sensing, and sorption capabilities of these advanced porous materials.

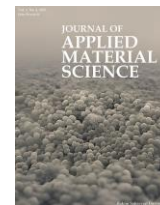
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Invited Review

Rational Design and Development of Metal-Organic Frameworks

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Abstract

Metal-organic frameworks (MOFs) represent a revolutionary class of hybrid materials with immense potential to address energy and environmental challenges, such as gas sorption and separation, as well as the detection and degradation of toxic chemicals. Recently, several commercial technologies utilizing MOFs have been developed for applications in the food safety industry, as well as for the storage and transportation of toxic gases like Arsine (AsH_3) and Boron trifluoride (BF_3). Despite these significant advancements, one of the most important and unresolved challenges in the synthesis of MOFs is controlling their network topology, which refers to the geometric arrangement of building blocks within the 3D architecture of the framework. Effective topology control is crucial for predicting the final structure and its properties, which depend on that structure. By carefully selecting the size, geometry, and connectivity of the building blocks, we can create a variety of network topologies that contribute to the design of MOFs with both high stability and permanent porosity. In this minireview, our recent studies on how controlling the topology affects the structural variations and enhances the catalytic, sensing, and sorption capabilities of MOFs are discussed.

Keywords: Metal-organic frameworks; Topology; Secondary building units.

1. Introduction

Metal-Organic Frameworks (MOFs) are an emerging class of crystalline porous materials, formed through coordination bonds between metal ions or clusters and multi-donating organic linkers, which are often referred to as molecular building blocks [1-3]. These structures can be developed in two or three dimensions, creating a periodically ordered porous network. One of the most notable features of MOFs is their exceptionally high porosity, with reported surface areas reaching up to $7,800 \text{ m}^2/\text{g}$. This makes them the leaders in surface area

among all traditional porous materials, such as zeolites and activated carbons [4, 5].

The structural diversity of MOFs is remarkable [6], as these highly porous materials consist of a wide variety of organic (linkers) and inorganic (metal nodes) secondary building units (SBUs) [7]. Metal nodes can take the form of single metal ions or clusters consisting of two, three, four, six, eight, or even twelve nuclei, or even infinite chains [8]. Organic ligands can also connect to two, three, four, six, eight, or twelve metal nodes, resulting in three-dimensional frameworks of MOFs with diverse underlying network topologies [9].

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For over two decades, MOFs have been at the forefront of scientific research, especially in applications that address various energy and environmental challenges [10-13]. The performance of any MOF in a particular application is largely determined by its building units, namely the metal clusters and organic ligands [14, 15]. As such, porosity and stability are considered the most critical properties of MOFs [16].

Despite these impressive characteristics, controlling the geometric arrangement of SBUs within the overall MOF network, referred to as the underlying network topology, presents one of the most significant challenges in the design and development of these materials [17]. Consequently, understanding, predicting, and classifying network topologies are essential for designing novel MOFs with specific properties. This focus on topological exploration has become increasingly important for chemists working with MOFs in recent years [18, 19].

Designing the network topology in MOFs can be approached through two main methods: modifying the connectivity of metal clusters [20] or altering the topological geometry of linkers [21]. Metal clusters

typically adopt established geometries that are difficult to modify for changing connectivity. Therefore, logically designing linker geometry is crucial for discovering specific topologies that serve as ideal platforms for MOF design [9]. For instance, the first MOF was reported in 1995, featuring a framework composed of zinc acetate ($Zn_4O(COO)_6$) clusters and a well-known ditopic benzene dicarboxylate linker [22]. This design has been expanded upon to create a series of isorecticular MOFs (IRMOFs) by substituting the original linker with various ditopic linkers in a process known as reticular synthesis [23].

Despite their potential, the susceptibility of zinc acetate clusters to hydrolysis limits their applicability in gas adsorption and separation, as these processes often occur in humid environments [24]. This limitation prompted the design of MOFs that incorporate binuclear M_2 -paddlewheel clusters (where $M = Zn$ or Cu), which exhibit improved water stability. However, combining these clusters with ditopic linkers typically results in a 2D layered coordination polymer instead of a 3D framework. In contrast, the combination of a tricarboxylate linker, benzene tricarboxylate, and a

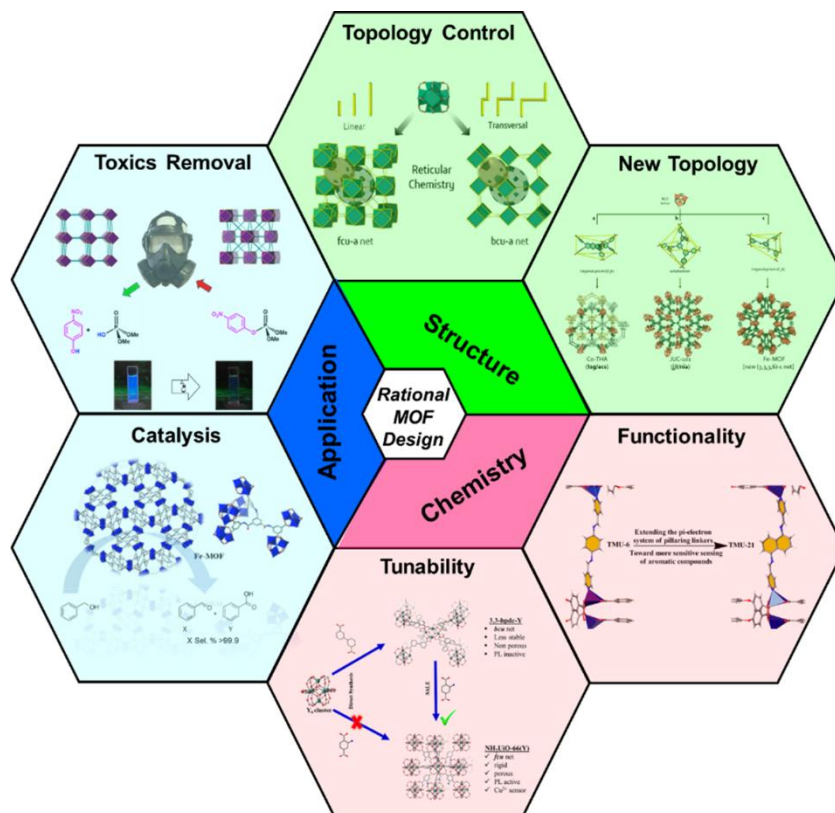


Figure 1. Rational design and intelligent development of MOFs.

copper paddlewheel cluster has led to the creation of the highly porous and stable 3D framework HKUST-1 ($[\text{Cu}_3(\text{BTC})_2 \cdot 3\text{H}_2\text{O}]_n$) [25], which has served as a benchmark in adsorption studies for many years [23]. Choosing linkers with flexible branches, desired dihedral angles, and specific topological shapes offers several advantages, particularly when using multitopic linkers [26].

Today, the application of topology, a branch of mathematics, to molecular building blocks or secondary building units (SBUs) has enabled the synthesis of a myriad of MOFs based on predicted topologies [27-29]. In conjunction with these methods, researchers have recently developed new design strategies to further enhance the rational design of MOFs. These include supermolecular building blocks (SBBs) [30], supermolecular building layers (SBLs) [31], and the merged-net approach, which combines two highly ordered topologies into one net. This strategy is particularly useful for rational MOF design [32].

This minireview focuses on the role of rational design in MOFs, considering their structure, chemistry, and applications (Figure 1). It emphasizes how intelligent control over MOF growth can lead to various outcomes, including: 1. Predictability of final MOF topology, 2.

Enhanced detection and degradation of nerve agents, 3. Discovery of new topologies, 4. Conversion of undesired structures into desired structures, 5. Selective oxidation of alcohols, 6. Adsorptive removal of organic dyes.

3. Topology control

Knowledge, prediction, and classification of network topology are important for designing novel MOFs with specific characteristics, and, as such, topological exploration in MOFs has become an important focus of MOF chemists in recent years [33]. The geometric arrangement of the secondary building units (SBUs), which include linkers and metal ions, in MOFs is very similar to children's toy building blocks. The logical selection of the type, size, shape, and arrangement of these SBUs is crucial in determining the final geometry of the structure, as well as its physical and chemical properties geometry misgeometry ge. Consequently, discovering novel methods to control and even predict the network topology has become one of the most exciting research areas in MOF chemistry. One such strategy introduced is called "net-clipping". According to the proposed hypothesis, changing the geometry of

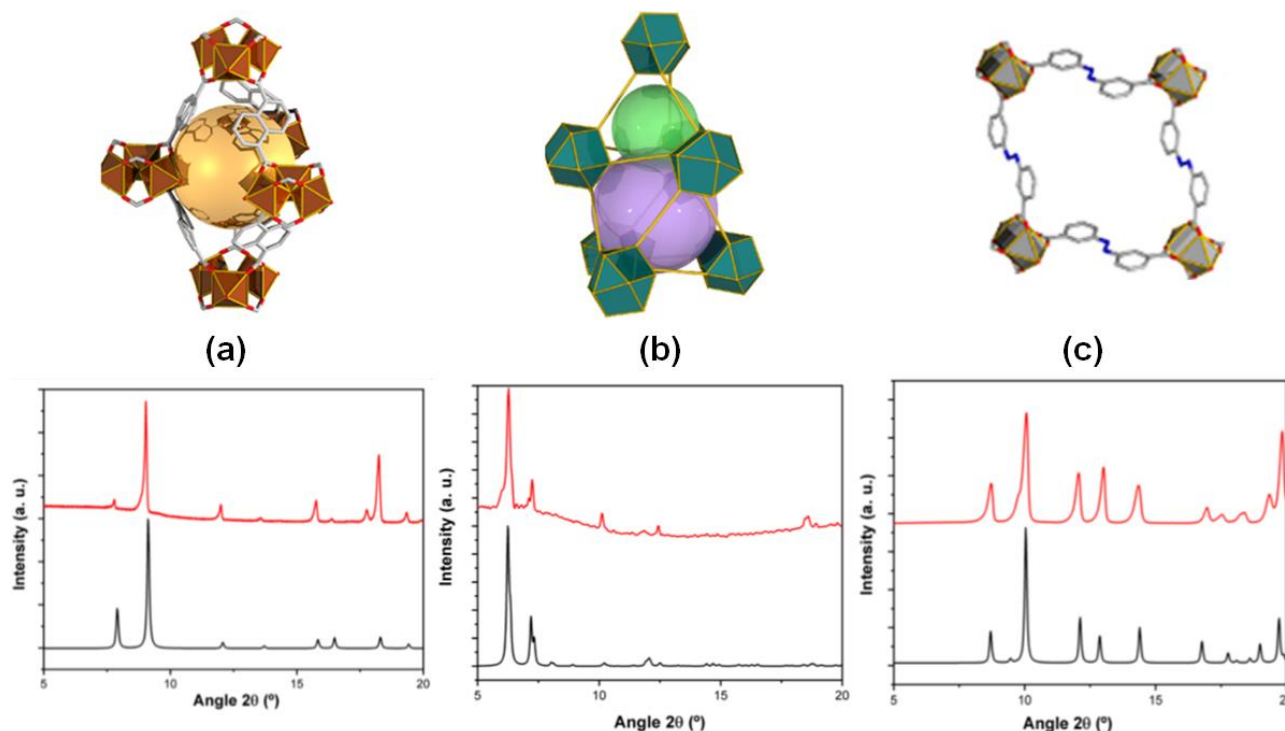


Figure 2. Crystallographic structure and XRD of Cu-3,3'-ABDC (a), Tb-3,3'-BPDC (b), and Fe-1,5-NDC (c). The black and red lines are related to simulated and experimental patterns, respectively. Reprinted with permission from [34]. Copyright 2020 American Chemical Society.



Figure 3. Function-topology relationship in the catalytic hydrolysis of DMNP in two Zr-MOFs.

the ligands from linear to zigzag allows for the prediction of the final MOF topology [34].

Computational data indicate that applying this method to 17 reported MOFs results in 10 structures exhibiting unique underlying topologies. Additionally, investigations of some existing structures in the literature have experimentally validated the results derived from this method. To further demonstrate this approach as a valuable strategy for topology control, three new MOFs, Cu-sql-3,3'-ABDC, Tb-fcu-3,3'-BPDC, and Fe-acs-1,5-NDC, were synthesized as single crystals and identified using X-ray diffraction. Interestingly, even though these three synthesized MOFs consist of different building blocks in terms of type, number, and geometry of the metal clusters, all three frameworks exhibit the same topology predicted by the net-clipping method. We reasoned that MOF structures made of zigzag ligands could be anticipated via rational clipping of the connecting groups of more symmetric SBUs in nets. Figure 2 represents the determined structure through single X-ray crystallography analysis and the compared XRD pattern of prepared MOFs by net-clipping.

3. Topology-function relationship

Among different types of MOFs synthesized to date, highly chemically, mechanically, and thermally stable

zirconium (VI)-based MOFs (Zr-MOFs, containing Zr_6 cluster, $[Zr_6O_4(OH)_4]^{12+}$) show amazing hydrolysis rates toward organophosphate nerve agents (OPNs) [35]. In addition to various parameters such as morphology [36], defect [37], and functionality [38], network topology can positively affect the hydrolysis rate of OPNs. The importance of controlling network topology in enhancing the catalytic rate of MOFs for the removal of organophosphates, particularly as nerve agent simulants, has been emphasized [39]. A comparative study was conducted on the catalytic capabilities of two Zr-MOFs that were constructed from identical SBUs but exhibited different topologies: Zr-bcu-tmuc and Zr-fcu-tmuc [40]. This study focused on the hydrolytic degradation of DMNP, which serves as a nerve agent simulant [40].

Despite being made up of the same molecular building blocks, the distinct differences in catalytic ability between these two Zr-MOFs can be attributed solely to their differing network topologies. The findings indicate that the 8-connected Zr-bcu-tmuc network offers greater accessibility to the active Zr(VI) sites compared to the 12-connected Zr-fcu-tmuc network, resulting in a several-fold increase in the hydrolysis rate. Furthermore, fluorescence sensing experiments revealed that Zr-bcu-tmuc exhibits significantly higher sensitivity to DMNP than Zr-fcu-tmuc, corroborating the catalytic results (Figure 3). This study paves the way for scientists

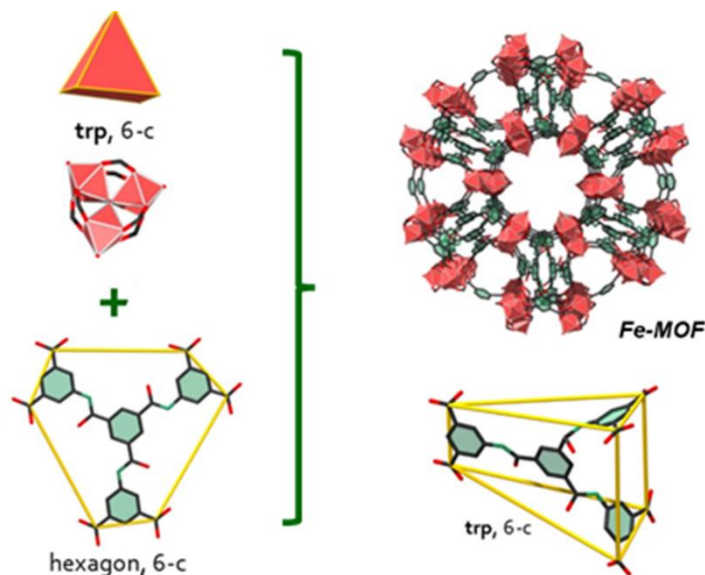


Figure 4. Schematic representation of flexible linker-based Fe-MOF structure.

to design more effective MOFs for countering chemical threats by manipulating topology.

4. Topology discoveries

Among the various rational design strategies that have emerged over time, selecting suitable SBUs with the appropriate connectivity and directionality can facilitate the assembly of MOFs with highly regular, predictable topologies [20]. The majority of studies in the field of MOFs focus on combinations of octahedral Zn_4O clusters [41], square-like paddlewheel Zn_2/Cu_2 clusters [25], and Zr_6 clusters [42] with various carboxylate ligands. One exciting study was the discovery of the gea net as an ideal blueprint net by Guillerm et al. Their geometrical analysis of the gea-a net revealed that transposition of pre-known copper paddlewheels to MOP into the anticipated gea-MOF platform required the employment of a multicarboxylate ligand with two branches that contain 120° angle dicarboxylic acid extremities and a third branch with a 90° angle dicarboxylic acid extremity [43].

Despite the numerous reported structures, MOFs composed of MIII triangular prismatic metal clusters, such as iron, chromium, and aluminum, are less studied. This limited focus is likely due to a relative lack of understanding regarding their formation conditions [44]. In this work, we present a geometry mismatch

strategy for assembling a non-interpenetrated, porous iron-based MOF, identified as Fe-MOF. By combining trigonal prismatic (trp, 6-c) metal clusters with a 6-connected hexagonal linker, components that initially appear incompatible due to the absence of a net for their assembly, we successfully discovered a new topology (Figure 4). The Fe-MOF structure is formed through the pillaring of a new type of supramolecular building layer, characterized by a rare 4-nodal, 3,3,3,6-connected topology. This structure demonstrates high water stability and significant porosity, featuring functionalized one-dimensional hexagonal channels. The findings highlight the potential for discovering new topologies, even when utilizing a common building block such as a basic Fe acetate cluster [45].

5. Converting the undesired structure to the favorite network

The main method for synthesizing metal-organic frameworks (MOFs) is through a self-assembly process that occurs under solvothermal conditions. However, this method can be difficult to control, resulting in the formation of undesirable networks [46]. To address this issue, post-synthesis techniques can effectively modify these undesirable networks into more desirable structures. Consequently, research has concentrated on controlling the topology of MOFs.

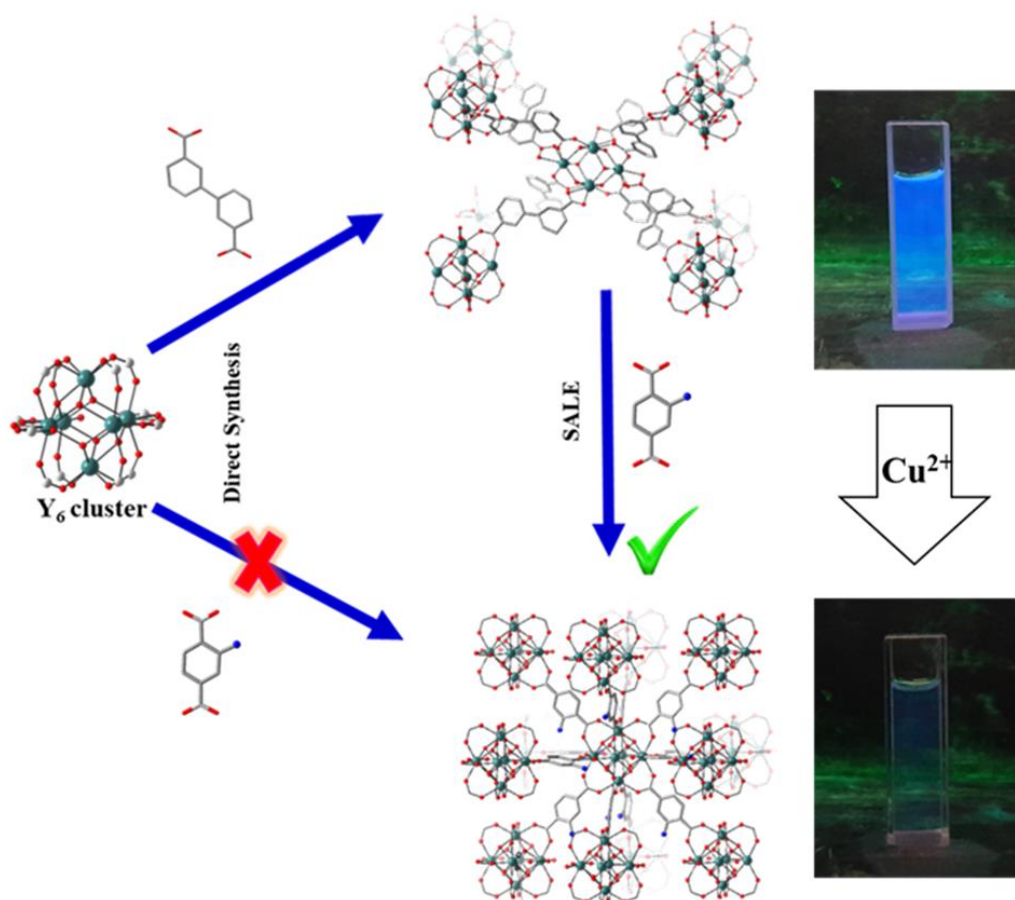


Figure 5. Representation of solvent-assisted linker exchange synthesis of $\text{NH}_2\text{UiO-66(Y)}$. Reprinted with permission from [47]. Copyright 2022 American Chemical Society.

One such method employed was solvent-assisted linker exchange (SALE), which was utilized to prepare an yttrium-based aminated UiO-66 framework—this framework cannot be created through direct synthesis. Notably, SALE successfully transformed a completely non-porous initial structure, 3,3-bpdc-Y, into a porous framework. Additionally, it altered the topology from an unstable 8-connected bcu network to a much more stable 12-connected fcu topology (Figure 5). Importantly, SALE not only resulted in the creation of the relatively highly porous $\text{NH}_2\text{UiO-66(Y)}$ from the completely non-porous 3,3-bpdc-Y but also changed its network topology from an 8-connected bcu structure to a 12-connected fcu framework. To the best of our knowledge, this is one of the rare instances where SALE has successfully modified the entire network topology of the final MOF. Furthermore, $\text{NH}_2\text{UiO-66(Y)}$ demonstrated a promising ability to selectively detect Cu^{2+} ions at low concentrations [47].

6. Catalytic oxidation of alcohols

Iron-based metal-organic frameworks have garnered significant attention as heterogeneous catalysts for oxidation reactions [48]. Despite several reports on MOF-based catalysts for the selective oxidation of benzyl alcohol, preventing overoxidation to benzoic acid remains a challenge [49]. Therefore, the catalytic activity of Iron-based MOF was evaluated in the aerobic oxidation of benzyl alcohol in air. Due to its iron-containing clusters, high porosity, large channel dimensions, functionality, and especially high stability, Fe-MOF is an excellent candidate for the selective oxidation of benzyl alcohols. Interestingly, Fe-based MOF exhibited enhanced activity compared to the neat Fe_3O_4 cluster, achieving a yield of benzyl alcohol oxidation that increased from 31.7% to 91.9% toward benzaldehyde.

This improvement can be attributed to the high surface area and the suitable aperture size of its 1D channels, which provide an optimal environment for the oxidation process. Additionally, host-guest interactions, such as hydrogen bonding between benzyl alcohol and the amide functions in the Fe-MOF channels, can enhance the accessibility of reagents to active sites, resulting in excellent catalytic activity. In comparison to previously reported MOF-based catalysts, Fe-MOF demonstrates remarkable selectivity and activity for the catalytic oxidation of benzyl alcohol within a relatively short reaction time, achieving a yield of 91.9% in just 3 hours. This work illustrates that it is still possible to identify promising MOF-based heterogeneous catalysts, even when utilizing one of the most common building blocks, the basic Fe acetate type Fe_3O cluster [45].

7. Adsorptive removal of dyes

The structural properties of Fe-MOF, including its water-resistant nature, the presence of two types of large channels measuring 1.2 and 0.6 nanometers, a high BET surface area of 1740 m^2/g , and acyl amide functional groups, were assessed for their effectiveness in adsorbing organic dyes from aqueous environments. A range of anionic and cationic dyes was tested, including methyl orange (MO), methyl red (MR), congo red (CR), rose-bengal B (Ros B), rhodamine B (Rhod B), and methylene blue (MB), to evaluate the efficiency of Fe-MOF. The presence of acyl amide groups enhances the interaction between the framework and anionic dyes, particularly with MO, which exhibited an adsorption capacity of 232.5 mg/g .

Interestingly, Fe-MOF demonstrated one of the fastest adsorption rates for MO, with a kinetic constant of 0.013 $\text{g}/\text{mg}\cdot\text{min}$. This rate surpasses that of several well-known metal-organic frameworks (MOFs) such as UiO-66, MIL-53, UiO-66-NO₂, and ZIF-67. The study also explored the adsorption mechanism, uptake kinetics, and selectivity of the framework regarding different dyes. Overall, the water-stable Fe-MOF, characterized by significant permanent porosity and functionalized pores, enhanced the size and charge-selective removal of dyes through preferred hydrogen bonding and π - π interactions. The experimental results indicated that Fe-MOF has a greater affinity for anionic dyes over cationic dyes, achieving substantial adsorption capacities of 232 mg/g for the toxic MO dye and 120 mg/g for the MB dye, indicating its high efficiency in this application [50].

8. Conclusions

In summary, this study highlighted how the rational design and intelligent control of MOFs network topology affects their structure-dependent properties and consequently their capability toward diverse application areas including; (1) detection and catalytic destruction of the organophosphorus nerve agents, (2) precise identification of copper ions in aquatic environments, (3) investigation of selective catalytic properties, and (4) correlation between structure and the adsorption of organic dyes. An innovative, creative, and efficient technique for deducing the network topology of MOFs built up from zigzag linkers, called net-clipping, was introduced. Designing MOFs with unique topology results in structures with excellent applicability in detecting and hydrolysis of organophosphorus nerve agents, adsorptive removal of dyes, and copper (II) ion sensing, which are significant environmental hazards in today's industries. Additionally, the study shows that it is possible to discover a novel porous iron-based MOF named Fe-MOF even using incompatible SBUs, which acts as a heterogeneous catalyst for the selective oxidation of benzyl alcohol to benzaldehyde under mild conditions. The compilation of these exceptional applications highlights the effectiveness of this design approach and the extraordinary capabilities of the porous MOF materials across a wide range of chemical industry sectors.

Conflict of Interest


The authors declare no conflict of interest.

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