

Preparation and Application Progress of MOFs

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Abstract. The everyday needs of humanity can no longer be met by traditional materials due to advancements in science and technology. These days, the main objective is to produce materials with increased usefulness, superior performance, and recyclability. Because of their high specific surface area and porosity, easily functionalizable pore structure, and customizable pore structure, metal-organic skeletons (MOFs) represent a novel class of porous materials with a wide variety of applications in the energy, environment, and biomedical sectors. Therefore, this paper summarizes the latest research progress in the study of MOFs. Firstly, the paper introduces the basic features of MOFs, including metal centers and organic ligands, and discusses the tunability of these modules for the geometrical and chemical properties of MOFs. Then, the main synthetic methods of MOFs are summarized, including solvothermal synthesis, electrochemical synthesis, and microbiome-assisted synthesis. Finally, the paper also highlights the applications of MOFs in gas storage/separation, biomedicine, and environmental protection. It aims to provide some reference significance for the future development direction of MOFs.

Keywords: MOFs, material structure, synthesis technology, application.

1. Introduction

New materials are becoming more and more in demand as a result of society's and science's fast advancement. Conventional materials can no longer satisfy the demands of sustainability, efficiency, and diversity in this day and age. Metal-organic frameworks, or MOFs, are a family of multifunctional crystalline materials with a porous structure that has garnered a lot of interest because of their exceptional qualities. Its enormous specific surface area, highly adjustable porosity, and other qualities have sparked a lot of attention and demonstrated considerable potential in a variety of industries.

MOFs are a family of multifunctional porous crystalline materials that, because of their special structure and adaptability, show a wide variety of applications in numerous sectors. MOFs are created by integrating adjustable inorganic and organic components into a predetermined network structure by joining metal secondary building units (SBUs) via organic linkers [1]. This modular assembly strategy, combined with structure-directed synthesis, enables the targeted preparation of porous MOFs with optimized properties, providing a powerful means to prepare porous MOFs with excellent properties and promoting their wide application in the fields of gas adsorption and separation [2,3], biomedicine [4] and environmental protection[5,6]. MOFs are known for their highly tunable pore structure in gas adsorption and separation, offering new possibilities in gas separation technology[3]. In the biomedical field, MOFs are used as drug delivery carriers and imaging agents, providing innovative directions for targeted therapy and molecular imaging [4]. At the same time, MOFs are used in environmental protection such as gas purification and water pollution control, demonstrating their potential for environmentally friendly and sustainable development [5,6]. As a result, MOFs are of great interest in science and industry/technology, and further research will contribute to the development of materials science to provide smarter and sustainable solutions.

This article deeply explores the basic properties of MOFs, including the composition of metal centers and organic ligands and their crystal configurations, and then introduces some popular MOFs synthesis methods currently in the scientific community, and introduces gas storage/separation, biomedicine and environment. Some recent research advances in conservation.

2. Basic characteristics of MOFs

2.1. Metal Center

Different types of MOFs may be created based on the structural properties of MOFs, by varying the metal center, organic ligands, and synthesis conditions. Metal ions or metal clusters can serve as the metal centers in MOFs. The inorganic component of the material is these metal ions or metal clusters. The network structure, pore size, topology, and other characteristics of the MOFs are all determined by the metal core. To achieve its functionalization, the metal core can also be utilized in tandem as a functional primitive. Therefore, MOFs with various architectures and characteristics may be created by selecting different metal centers [1]. In the field of electrocatalysis, copper ions are usually considered to be the key catalytic sites inside MOFs, so copper ions often determine the electrocatalytic performance of Cu-MOFs. Cao et al. used copper ions and zinc ions with the same organic ligand 2-methylimidazole (2MI) to synthesize different MOFs (Cu-2MI and Zn-2MI) for comparative experiments. Experiments have confirmed that Cu-2MI performs better than Zn-2MI in terms of electrochemical performance. Cu-2MI has a larger electric double layer capacitance than Zn-2MI ($24.1 \mu\text{F cm}^{-2}$ vs. $7.3 \mu\text{F cm}^{-2}$). Smaller redox potential difference (112 mV vs. 148 mV), lower charge transfer resistance (R_{ct}) (1816Ω vs. 3244Ω), higher heterogeneous rate constant. It shows that Cu-2MI is far superior to Zn-2MI in terms of electron transfer, electron transfer capability, and electrocatalytic activity [7]. In a similar vein, the metallic rare earth element europium (Eu) exists. It has unique luminous qualities and may be utilized to create phosphors, laser materials, photocatalysts, and other materials because its atoms can absorb a large number of neutrons. Eu_2O_3 , for instance, is a pale red powder that dissolves in acid but is insoluble in water. may be utilized to create optical filters and colored lenses. These clearly show that the unique functionalities of the materials in MOFs may be greatly influenced by changing the metal center. While europium ions are more suited for use in fluorescence detection, copper ions are better suited for use in the electrification sector.

2.2. Organic Ligands

Additionally crucial to the creation and characteristics of MOFs are organic ligands. In addition to acting as ligands to interact with metal centers, organic ligands also function as connecting elements to join various metal centers. This permits There is more selectivity in the synthesis of MOFs [1]. Using copper ions and two distinct organic ligands, 2-methylimidazole (2MI) and 1,3,5-benzene tricarboxylic acid (H3BTC), Cao et al. conducted comparative tests and discovered that Cu-2MI, a two-dimensional nanosheet, has a high specific ratio and a porous structure. Larger electric double layer capacitance, quicker electron transfer, increased electrocatalytic activity, and decreased oxidation potential are all present on the surface area. Cu-BTC, on the other hand, is a bulk substance with a cubic structure. Cu-BTC has poor electrochemical performance, primarily because of its small specific surface area and microporous structure, which limit its spatial configuration. Its decreased adsorption capacity and electrocatalytic activity are due to its narrower contact area with target molecules [7]. Thus, selecting suitable organic ligands is one of the most essential components in defining the material properties of MOFs.

2.3. Crystal Structures

MOFs are a kind of lattice structure composed of metal ions or clusters of metal ions combined with organic ligands. These crystal structures have remarkable qualities and a variety of applications because of their distinctive construction and assembly [1]. Geng et al. studied the characteristics of four iron-based MOFs with varied structures, Fe(II)-MOFs (rod-like structure), Fe-MIL-53 (spindle-like structure), Fe-MIL-88A (octahedral-type structure), and Fe-MIL-101 (three-dimensional structure). Although all iron-based MOFs were capable of activating PDS and degrading ciprofloxacin (CIP), there were variances in performance, with Fe(II)-MOFs demonstrating the highest performance in degrading CIP, with a degradation rate constant of 0.155 min^{-1} . Fe-MIL-53

(0.125 min⁻¹), Fe-MIL-88A (0.102 min⁻¹) and Fe-MIL-101 (0.087 min⁻¹) followed [5]. This suggests that differing crystal structures have an impact on the performance of MOFs.

3. Synthesis of MOFs

MOF synthesis methods have a direct impact on their performance and application range, therefore identifying practical and efficient synthesis pathways has become a priority for academics. With the advancement of science and technology, a plethora of novel synthetic techniques have arisen, enabling a plethora of alternatives for the design and production of MOFs. The synthesis of MOFs is at an exciting period of evolution, from the classic solvothermal approach to the introduction of green synthesis methods.

3.1. Solvothermal Synthesis

The procedure involves dissolving a blend of organic ligands and metal salts in a solution, and then putting the combination in a closed vessel for a reaction at a high temperature. One of its features is its ability to react at high pressure and temperature, which helps dissolve insoluble organic linkers. The development of crystals may be influenced by varying synthesis circumstances, including temperature, time, solvent, pH, and so on. Additionally, MOFs with distinct structures can result from varying reaction pressures. Several families of MOFs, such as PCNs, UiO-66, MOE-74, MOF-177, and MIL-101, have been successfully synthesized using solvothermal techniques. Despite the success of solvothermal methods in designing novel structures and hetero-block constructs, there are still some challenges in large-scale production, such as long reaction time, large amounts of solvents, and high cost, as well as difficulties in guaranteeing the crystallinity and yield of the products under scaled-up synthesis conditions [8].

3.2. Electrochemical Synthesis

Electrochemical synthesis, which begins with electrical energy and acts in two major ways: anodic dissolution and cathodic deposition, may produce metal-organic frameworks (MOFs). Metal ions are formed in anodic dissolution when the anode dissolves and combines with organic linkers in solution to generate a powdered or film-forming MOF product. A thin layer of MOF is deposited in cathodic deposition as the reaction fluid, which includes metal ions, organic linkers, and alkalis, becomes more alkaline. For several reasons, electrochemical synthesis has drawn interest in the creation of MOF. First, compared with traditional synthesis methods, electrochemical synthesis usually only requires mild conditions and shorter reaction times. Second, since metal ions are obtained by anodic dissolution, toxic and difficult-to-separate anionic metal salts are avoided. Therefore, electrochemistry is considered the most likely method to manufacture MOFs on a large scale and achieve green synthesis. Finally, electrochemical methods enable easy preparation of MOF films, and precise control of film properties can be achieved by modulating the potential difference [8].

3.3. Microwave-assisted Synthesis

This method relies on the interaction between electromagnetic radiation and substances with mobile charges, which can be polar molecules in solvents or conducting ions in solids. Therefore, ionic liquids and water are considered ideal solvents in microwave-assisted synthesis. Typical processes for microwave methods include transferring a mixture containing a reaction precursor and an appropriate solvent into a sealed PTFE container or glass vessel, which is then placed in a microwave cavity and heated for an appropriate period. Microwave heating interacts with reacting substances through direct radiation, thus accelerating the reaction process. Compared with traditional heating methods, the reaction time is reduced. Since microwave synthesis significantly shortens the reaction time, this method exhibits low energy consumption and high efficiency. Therefore, in some industrial application fields, microwave synthesis is regarded as a large-scale synthesis method with broad prospects [8].

3.4. Sonochemical Synthesis

Sonochemistry is a method of rapid synthesis using ultrasonic power to obtain metal-organic framework (MOF) products in minutes or hours without the need for a cooling system by placing a solution containing reaction precursors in an ultrasonically irradiated reactor. Sonochemistry uses the acoustic cavity effect to achieve local ultra-high temperatures (approximately 5000 K), ultra-high pressures (approximately 1000 atm), and extraordinary heating/cooling rates (approximately 10° K/s). Compared to conventional synthesis methods, sonochemistry offers the advantages of shorter reaction times, simplified operation, and improved energy efficiency. Ultrathin MOF nanosheets were successfully produced, as were MOFs with various topologies and morphologies at various ultrasonic intensities. Furthermore, sonochemistry was used to effectively synthesis new zinc-based coordination polymers with flower-like morphologies [8].

3.5. Hydrothermal Synthesis

One popular technique for making MOFs is the water reflux approach. To produce MOFs, the basic idea is to dissolve the MOF precursor in water, bring the mixture to a boil, and allow the solute molecules to polymerize with one another in the presence of water vapor. The hydrothermal approach has several benefits, including ease of use, cheap cost, and broad application. However, it may also result in a lesser degree of crystallinity and a greater impurity content in the MOFs. The extremely effective, sustainable, and ecologically friendly hydrothermal synthesis approach was used to effectively synthesize MOF-808(Hf). In terms of methane storage performance, MOF-808(Hf) exhibits excellent elastic modulus and outstanding methane storage capacity, especially in high-pressure natural gas storage. Compared to MOF-808(Zr), MOF-808(Hf) has higher structural integrity and compression resistance, making it more promising for practical applications. Its development has a positive impact on improving energy storage efficiency and reducing greenhouse gas emissions and environmental changes. In terms of methane storage performance, MOF-808(Hf) has a volumetric methane deliverable capacity of 110 VSTP v^{-1} , which is the best performance among MOFs with Zr/Hf. Its modulus of elasticity is as high as $37.5 \pm 3.4 \text{ GPa}$, highlighting its excellent performance and promise in the field of high-pressure natural gas storage [9].

3.6. Mechanochemical Synthesis

Mechanochemical synthesis utilizes mechanical forces, usually through ball milling or extrusion of precursor solids. During the ball milling process, the reaction precursor is put into a ball mill for mechanical grinding. This method allows for the use of little or no solvents and is therefore considered a "green" and efficient method of synthesis. Mechanochemical synthesis methods use less soluble metal sources such as metals and metal oxides. Therefore, the only by-product produced is water, which can be removed by a simple heating operation. Depending on the operating conditions, mechanochemical methods can be classified as solvent-free grinding (SFG), liquid-assisted grinding (LAG), and ionic liquid-assisted grinding (ILAG). Mechanochemical methods are divided into grinding method, ball milling method, and extrusion method. Ball-milling and extrusion methods are more widely used, easy to mold, and suitable for mass production [8].

3.7. Continuous Flow Synthesis

Continuous flow synthesis is a technique that uses peristaltic, high-performance liquid chromatography (HPLC), or syringe pumps to process reaction solutions continuously through microfluidic chips or reactor coils. Stirred tank reactors (CSTRs), plug-in flow reactors (PERs), and microfluidic reactors (MRs) are some examples of these three types. The benefits of continuous flow synthesis over traditional batch synthesis include a quicker reaction rate, more accurate control over reaction parameters, reduced solvent and energy use, and greater capacity growth [8].

3.8. Spray-drying Synthesis

Spray drying is a low-cost, sustainably scalable method for the production of pharmaceuticals, foods, pigments, and organic and inorganic compounds. The spray drying method is based on the rapid evaporation of precursor substances in high-temperature gases. The method consists of the atomization of the MOF precursor solution containing the metal ions and linkers into tiny droplets by a two-fluid spray dryer. apparatus to atomize the solution containing metal ions and linkers into tiny droplets, which form spherical hollow superstructures by evaporation of the solvent and diffusion of the precursor molecules. This approach has been successfully applied to a variety of MOF families, such as UIOs, ZIFs, and MILs [8].

3.9. Hot-pressing synthesis

The high-pressure and high-temperature (HOP) method is a method for preparing metal-organic framework (MOF) nanocrystals by applying pressure and temperature to the substrate under solvent-free or minimal solvent conditions. This method combines metal ions or organic linkers with functional groups or metal sites on the substrate surface, subsequently initiating nucleation and crystal growth. For the first time, Dou's team successfully used the HOP method to prepare various MOF coatings quickly, solvent-free, and binder-free and successfully coated a variety of flexible substrates. The roll-to-roll HOP method successfully produced MOF coatings at scale, with excellent performance in particle removal, durability, and recyclability. At the same time, other studies have also demonstrated the application of the HOP method in the synthesis of polyoxometalate-based MOF and In-MOF coatings on carbon cloth, polyester, and aramid, as well as its application in photocatalytic sterilization performance and preparation of MOF filters. The HOP method is also suitable for the preparation of mixed matrix membranes (MMM), which exhibit strong mechanical strength and high-throughput separation performance, providing a promising method for water treatment and molecular dye capture applications [8].

4. Applications of MOFs

When it comes to innovations in the field of materials science, metal-organic frameworks (MOFs) are undoubtedly one of the focuses that receive much attention. MOFs exhibit excellent performance in various fields because of their extremely large surface area and pore structure. Especially in gas storage/adsorption, biomedicine, and environmental protection, the application of MOFs is particularly prominent [10,11]. The diversity and adjustability of MOFs provide innovative solutions to global problems [12,13].

4.1. Gas storage

Because of their special structure and modulation characteristics, MOFs are excellent candidates for gas storage and adsorption applications. They are capable of efficiently absorbing and storing a wide range of gases, such as carbon dioxide, hydrogen, and methane, because of their highly adjustable pore structure. A new metal-organic framework material with a high capacity for CO₂ adsorption, called MUF-16 material, was described by Omid et al. At 1 atm and 293 K, MUF-16's maximal CO₂ capacity was 2.13 mmol/g. In breakthrough experiments, MUF-16's dynamic adsorption capacity for CO₂ varied from 1.2 to 1.5 mmol/g. The results of the repeated-use test showed that MUF-16 maintained the same amount of adsorbed CO₂ after at least 12 separation cycles, which implied that the storage of CO₂ can be more reusable with the same, which can provide a potential solution to the cost problem in industrial applications^[3].

For the storage of natural gas and hydrogen, Bin et al. report on a new material, BUT-22, which exhibits excellent methane adsorption performance at 296 K and 35 bar, with a wt. uptake of 325 cm³ (std.)/g, which significantly exceeds an earlier target of 180 cm³ (std.)/g set by the U.S. Department of Energy (DOE). Increasing the pressure further to 80 bar, BUT-22's methane weight uptake soared to 530 cm³ (std)/g (0.379 g/g), outperforming many well-known metal-organic-framework (MOF)

materials. The methane deliverable capacity (working capacity) of BUT-22 of 0.334 g/g at a pressure range of 296 K and 80 bar also stands out among MOF materials. When the temperature drops to 270 K and the pressure increases to 100 bar, BUT-22 even achieves the methane weight absorption capacity target of 700 cm³ (standard state)/g (0.5 g/g) set by the U.S. Department of Energy. In addition, the average enthalpy of methane adsorption of BUT-22 is 12.0 kJ/mol over the temperature range of 270-296 K. In terms of volumetric performance, BUT-22 achieves a methane volumetric storage capacity of 202 cm³(std)/cm³ at 296 K and 80 bar, and an operating capacity of 178 cm³(std)/cm³, which is on par or even better than other top MOFs. The working capacity is 178 cm³ (standard)/cm³, which is comparable to or even better than other top MOFs. At 100 bar and 77K, BUT-22 demonstrated excellent hydrogen adsorption performance, with 12.0wt%, exceeding the U.S. DOE has set a target of 6.5 weight percent. According to simulations, BUT-22's hydrogen delivery capacity might reach up to 11.6 weight percent at a temperature-pressure elasticity of 5 bar for 160 K release and 100 bar for 77 K adsorption. Under identical test circumstances, BUT-22 has the highest hydrogen adsorption and deliverable capacity among metal-organic skeleton (MOF) materials, surpassed only by NU-100 and NU-1103. Furthermore, BUT-22 exhibits an average enthalpy of hydrogen adsorption of 4.7 kJ/mol across a temperature range of 77-296 K. According to these findings, BUT-22 performs better at storing hydrogen, which might have a significant impact on the field of hydrogen energy applications. It also exhibits the unique property of increasing bulk performance with decreasing temperature, which provides strong support for the development of advanced gas adsorption materials [2].

4.2. Gas separation

The benefits of MOFs, a novel kind of porous material, include high porosity, a large specific surface area, highly adjustable pore size, and a variety of shapes. Because of these benefits, MOFs have several potential uses in the gas separation industry. The fields of adsorption, membrane, and catalytic separation are the primary areas in which MOFs are used in gas separation applications. Omid et al. reported that in the separation of hydrocarbons, the affinity of MUF-16 for CO₂ is significantly higher than that of competing gases, such as methane, acetylene, and ethylene. Therefore, the selective separation of CO₂/CH₄ and CO₂/C₂H₂ is very high, reaching 6690 and 510, respectively. in the dynamic breakthrough test, MUF-16 can completely separate CO₂ and hydrocarbon gas mixtures in a short time, and directly obtain high-purity hydrocarbon products, including pure methane and acetylene. although the application of MOFs in the field of gas separation is still in the research stage, some progress has been made [3].

Wiren et al. reported a novel Fe(OH)_x @ Cu-MOF composite, in which Cu-MOF nanolayers were covered on the surface of Fe(OH)_x hollow nanotubes. The material has a large number of unsaturated Cu-O₂ active centers, which can effectively promote the generation of hydrogen synthesis intermediates. Meanwhile, the hollow structure enhances the exposure of the active centers, and the Fe(OH)_x support also enhances the structural stability of the material. The Fe(OH)_x@Cu-MOF material exhibits a low overvoltage of 112 mV and a small Tafel slope of 76 mV/dec, which can achieve highly efficient electrocatalytic hydrogen synthesis, and is one of the best performances among the MOF-based electrocatalysts reported so far [14]. The application of MOFs in gas separation is still in the research stage, but some progress has been made. With the deepening of the research on MOFs, the application of MOFs in gas separation will be more extensive.

4.3. Biomedical

In the biomedical field, MOFs exhibit potential drug delivery and image diagnostic applications. Their structural tunability and large surface area provide an ideal platform for drug loading and release, while they also excel in medical imaging applications. Innovative applications of MOFs in drug delivery and medical imaging offer new perspectives for personalized medicine and precision therapies. Fang's research group introduces novel nanoplatforms based on a cascade enzyme/Fenton

reaction designed to enhance tumor therapeutic effects. This nanoplatform, named Co-Fc@GO_x, consists of cobalt ferrocene metal-organic framework (Co-Fc NMOF) and loaded glucose oxidase (GO_x) molecules. Co-Fc NMOF not only has the properties of Fenton's reagent but is also an effective carrier of GO_x. In the tumor microenvironment, Co-Fc@GO_x exhibits a typical enzyme/Fenton effect, catalyzing endogenous glucose to generate gluconic acid and H₂O₂, thus promoting the Fenton reaction of Co-Fc NMOF and amplifying it in situ. Induces cytotoxic hydroxyl radicals (-OH). Research on the anti-cancer properties of Co-Fc@GO_x in vitro and in vivo found that it can effectively inhibit the proliferation, survival, and migration of cancer cells, and significantly slow down the tumor growth of hormone mice while having no obvious side effects on normal tissues. This shows that Co-Fc@GO_x has significant potential in anti-cancer, providing a novel and innovative strategy for the field of tumor treatment [4].

4.4. Environmental protection

With its high adsorption capabilities and catalytic activity, MOFs have become a potent instrument for pollution control and waste treatment in the field of environmental protection. Geng et al. employed iron-based MOFs as catalysts to activate PDS for CIP degradation to tackle this problem. The researchers varied the conditions in their experiments, such as the amount of catalyst, the concentration of PDS, the reaction temperature, and the reaction time, and discovered that when the amount of catalyst was 0.4 g/L, the concentration of PDS was 0.036 mmol/L, and the reaction temperature was 25°C, the reaction time was the shortest. The greater the reaction time, the faster the CIP degrades. Meanwhile, actual wastewater was used for the tests to validate the feasibility of CIP degradation by iron-based MOFs activated by PDS, proving that the system could efficiently degrade CIP in real wastewater [5].

Ren et al. added the powdered Zr-MOFs that had been synthesized to wastewater that included doxycycline hydrochloride (DOC). It was discovered that Zr-MOFs had a maximum adsorption capacity of 148.7 mg⁻¹ g⁻¹ for DOC. In the meanwhile, 50 mg of Zr-MOFs were used to obtain an 84.4% clearance rate after a 5-hour reaction period at a DOC concentration of 30 ppm. Furthermore, Zr-MOFs' removal rate could still reach 19.7% after three cycles, demonstrating their strong adsorption capability, ability to remove DOC from wastewater, and remarkable reusability [6].

5. Conclusions

Against the backdrop of the growing interest in utilizing metal-organic frameworks (MOFs) for a variety of applications, this article summarises the structural features, typical synthetic methods, and recent research advances in MOFs for gas storage, separation, pharmaceuticals, purification, catalysis, and so on. The article demonstrates the structural tunability, functional designability, and outstanding application potential of MOFs.

Poor stability and synthesis issues, however, continue to be significant obstacles to the widespread practical use of MOFs. Enhancing MOFs' thermal and water stability is vital, but so is looking into more controlled and ecologically friendly synthesis methods to solve these issues. Thus, cutting-edge methods like electrochemical synthesis and microwave-assisted synthesis offer effective instruments to enhance the synthesis of MOFs. Because of the microwave method's quick reaction rate and excellent product purity, it opens up new opportunities for the synthesis of highly organized MOFs. Nevertheless, there are still issues with local overheating, selective synthesis, and hard-to-control reaction conditions. Conversely, electrochemical synthesis, a recently developed synthesis technique, is more environmentally friendly and selective, can be done in moderate environments, and doesn't require the use of hazardous solvents. But as the synthesis of MOFs grows larger, problems including how hard it is to prepare electrode materials, how electrolytes affect MOFs, and how hard it is to control electrochemical synthesis processes have come to light. Consequently, the focus of future research should be on fully leveraging the benefits of diverse synthesis techniques to address the stability and synthesis efficiency limitations of MOFs.

Looking into the future, several directions would be rewarding for the field to pursue, which may facilitate the commercialization and market acceptance of MOFs technologies. These include but are not limited to 1) Development of high-performance MOFs devices and systems with satisfactory stability in real working environments; 2) Scaled-up, cost-effective, and environment-benign preparation of quality MOFs; 3) Expanding the application space of MOFs in promising areas such as environmental remediation and biomedicine. With continuous advances in the structural characterizations, synthetic chemistry, and application-guided design of MOFs, it is envisioned that MOFs will find their niche in wider areas, far beyond what has been achieved by conventional porous materials. The unique attributes of MOFs shall eventually be realized for various cutting-edge applications.

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