Applications of Metal–Organic Frameworks

Subjects: Chemistry, Applied

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Metal–organic frameworks (MOFs) have been broadly applied to numerous domains with a substantial surface area, tunable pore size, and multiple unsaturated metal sites. Hollow MOFs and their composites as well as their applications can be used in organic catalysis, electrochemical sensing, and adsorption separation.

hollow metal–organic framework catalysis sensing adsorption

1. Electrochemical Sensing

Owing to the diversity in structure and composition of MOFs, their highly regulable chemical properties, and their facile interactions with guest molecules, the enrichment and removal of some target substances in MOFs can be accomplished simply and can lead to prominent electronic changes and a corresponding electrical response. Thus, MOFs have been a burgeoning platform for sensors that are able to detect and interact with many analytes, including heavy metal ions, biomolecules, toxic substances, and factory waste gases [1][2][3]. Nevertheless, the inherent shortcomings of MOF materials, including their poor electrical conductivity and tardy mass-transfer rate, limit their application in the field of electrochemical detection. To a large extent, pristine MOFs hinder the mass transfer of analytes on the material and affect the output of detection signals, which leads to great obstacles in their application in the field of electrochemical sensing. Meanwhile, the poor electrical conductivity of MOFs themselves further hinders the output of electrical signals, resulting in the inability to obtain high–sensitivity electrochemical sensors.

Hollow MOFs greatly enhance their mass-transfer capacity due to their unique hollow cavity and thin shell. Yao's group fabricated MIL-101 hierarchical hollow cages and constructed an electrochemical sensor using hollow MIL-101 to measure nitrofuranone (NFZ) [4]. The sensor's linear range was wide (0.030–55 M), while its detection limit was low (10 nM). After six parallel measurements, the recoveries remained in the range of 93.2–103%. The good detection performance of the sensor was ascribed to the huge specific surface field and quick electron-transfer rate. The internal cavity of the hollow MIL-101 was conducive to the aggregation of NFZ and provided more catalytically active sites. Similarly, Du et al. reported an unlabeled electrochemical adaptive sensor using a bimetallic, hollow MOF for the efficient detection of adenosine [5]. Adenosine is a potential tumor marker and has key signal transmission functions in the nervous system. It has many important physiological and pharmacological functions, including the dilation of coronary vessels, the regulation of the immune response, and the inhibition of the inflammatory response when the myocardial oxygen supply is reduced or its load is increased. Therefore, the detection of AD is very important for clinical diagnosis. By altering the Zn^{2+}/Ni^{2+} ratio, they prepared three kinds of hollow Zn/Ni–MOFs with different morphologies. When the Zn^{2+}/Ni^{2+} ratio was 1:2, a Zn/Ni–MOF with a layered,

hollow, microsphere structure was obtained. In this morphology, the immobilized number of aptamer chains was greatly increased, thereby facilitating AD binding. Bimetals can not only tune the morphology of MOFs but can also enhance their electrochemical properties and catalytic activity. The content of Ni^{2+} has an important effect on the anchoring of the aptamer chain because of the sturdy adsorption between the nickel center and $–NH_2$. Thus, the layered structure and synergistic effect of nickel and zinc will help to improve the sensor property for AD.

In addition, the combination of hollow MOFs with metals, metal oxides, and conductive polymers, etc. can solve the problem of poor conductivity. Liu et al. immobilized single platinum atoms on a hollow Zr–MOF (HPCN–222) for the electro-catalytic sensing of levodopa [6]. The linear range of the PtHPCN–222/GCE sensor was between 0.1–1 and 1–130 μM and had excellent recovery properties. The usage of Pt atoms was substantially enhanced by using the hollow PCN–222, with its high specific surface area, as the carrier of Pt atoms. In addition to increasing the substrate's diffusion rate, PtHPCN–222's hollow structure and wide surface area offered many redox-active sites that could interact with levodopa. Moreover, the load of Pt atoms provided more catalytic active centers and enhanced the response signal to the target.

2. Catalysis

As burgeoning porous materials, hollow MOFs have obtained extensive attention in catalysis. As a result of inheriting the merits of pristine MOFs with large surface areas and plenty of catalytic active centers, the intermediate cavities peculiar to hollow MOFs greatly shorten the diffusion path length of reactants and improve the catalytic performance. Therefore, hollow MOFs have great potential for organic catalysis with high activity, excellent selectivity, and fast kinetics. Liu et al. achieved the preparation of a hollow, mesoporous, hierarchical structure MIL–101 (HM–MIL) [7]. They carefully designed a bimetallic MOF with different metal–ligand bond stabilities and spatial distribution. By the enhanced nucleation process and selective etching treatment, hollow and mesoporous shells of MOFs were realized and used for catalytic reactions with 1–Chloro–4–ethenylbenzene as a probe. The void diameter and shell thickness of MIL–101 can be well adjusted by adjusting the response parameters. Benefiting from a higher porosity and wider channels, the mass-transfer resistance of guest molecules in HM–MIL was greatly reduced, and thus the transport rate was increased. Meanwhile, the rapid diffusion of 1–Chloro–4–ethenylbenzene in MOFs greatly improved their selectivity, as the rapid diffusion of 4-chlorostyrene in MOFs reduced their further oxidation. After five cycles, HM-MIL still exhibited excellent stability. Li et al. fabricated a hollow ZIF-8 as an excellent catalyst for cycloaddition reactions [8]. This kind of nano–catalytic carrier had a flexible shell and mesopore, which can effectively promote the regulation and diffusion of a matrix of various sizes. With 1, 3–cyclohexanedione and enal as the target compounds, the catalytic conversion and selectivity of the material to the target compounds reached 89% and 99.9%, respectively. Beyond that, the material could be utilized roughly ten times without losing any of its functionality. Zhang et al. constructed a Co–MOF–74 hollow structure for the first time with a thin shell measuring approximately 50 nm [9]. It also performed well for the thermocatalytic cyanosilication of aldehydes and the photocatalytic oxidation of thioanisole. This hollow Co–MOF–74 was composed of 8−18 nm nanoparticles and was different from its counterpart of an ordinary, three-dimensional structure. Such a structure had a greater availability of the active sites and an improved ability to capture light, and

therefore exhibited excellent photocatalytic activity for thioanisole. When the reactants were catalyzed by the hollow CO–MOF–74, the benzene ring connected aldehydes with electron–drawing groups, which were more reactive than those with electron-donating groups. The aldehydes were activated by unsaturated Co, whispering the reactants. A similarly effective heterogeneous catalyst has been employed with a double-shell hollow MOF. For example, an Au/MOF possessing a double shell was successfully synthesized and used for the tandem catalytic synthesis of imines from benzyl alcohol and aniline under air atmosphere and solvent-free conditions [10]. The highest turnover frequency recorded was 170 h^{-1}, and the conversion rate and selectivity were above 99%. It is well–known that hollow structures have been shown to enhance catalytic activity because of their characteristic properties in promoting mass transfer. The synergistic effect of hollow structures with Au is also crucial for the cascade catalytic reaction.

In order to achieve more catalytic effects, it has also become a trend to combine hollow MOFs with other active materials. For instance, a Zr/Ce, bimetallic, hollow-MOF nanoreactor was successfully synthesized by using a non-toxic and colloidal carbon template [11]. By adjusting the Ce/Zr ratio, the structure of nanospheres changed to some extent. When the Ce/Zr ratio was 1:4, the most ideal hollow structure was created, which was applied to accelerate the transformation of 2–furanaldehyde into 2–furylmethanol. The Lewis acid site of Zr(IV) in UIO–66 can activate furfural and transfer the dissociated hydrogen from the alkol to furfural to form furfural alcohol. However, with the Ce content increase, the activity of the composite decreased. This is because the Lewis acidity of Zr is higher than that of Ce. Therefore, excessive Ce doping will reduce the Lewis acidity of MOF and reduce its catalytic capacity. After testing the recovery performance of the catalyst, it was found that the conversion rate of furfural was maintained at 86% after three cycles, which proved that the material was relatively stable. Additionally, a new type of hollow core–shell nanoreactor also was proposed, which had Au and Zn/Ni–MOF–2 as the core and shell [12]. A hollow, bimetallic, MOF–embedded gold structure was fabricated using a simple, one–step strategy without employing a template for selective oxidizing alcohol. With methylbenzene and air as the solvent and environmental oxidant, respectively, the conversion rate of benzyl alcohol could be as high as 98%. Moreover, the selectivity of aldehyde and the conversion of benzyl alcohol did not change significantly after the reactor was reused five times, which proved that hollow Au@Zn/Ni–MOF–2 has good stability. Among them, small and well-distributed Au nanoparticles are the key to improve catalytic properties. The hollow Zn/Ni–MOF shell and Au nanoparticle core promoted the enhanced activity through a synergistic effect. In addition, bimetals were also used to be compounded with hollow MOFs for efficient catalysis. Oh et al. encapsulated highly active, bimetallic PdCo nanoparticles in a hollow ZIF–67 for the reduction of 4–nitrophenol. [13] Through the thermal decomposition of the PS@ZIF–67/Pd^{2+} under diverse temperatures, a hollow MOF and its derived carbon materials can be obtained. Pd^{2+} and Co^{2+} were successfully reduced to metal Pd and Co during pyrolysis. The hollow ZIF of the PdCo–loaded alloy revealed efficient catalytic activity for reducing 4–nitrophenol. The conversion from 4–nitrophenol to 4–aminophenol was completed within two minutes, and the TOF value was 57 min^{-1}, which was more than that of the reported catalysts. This was not only due to the unique, hollow MOF skeleton, but also the excellent catalytic activity of the bimetal and the electronic effects between Pd and Co, which caused the bimetal to have better catalytic activity than a single metal.

In summary, the unsaturated metal sites, porous structures, and internal cavities of hollow MOFs can be applied in catalysis. In addition, within the loading or decoration of different external active components, hollow MOFs can show excellent performance in different catalytic systems.

3. Adsorption and Removal

In recent decades, the pollution of water and the discharge of toxic, polluting chemicals are becoming more and more serious, leading to a battery of ecological problems such as global climate change, harm to humans, and botany destruction. Therefore, the exploration of high-efficiency environmental treatment materials is imminent and has received extensive attention [14]. N, O, and other elements rich in the ligands of MOFs can effectively capture radionuclides and heavy metal ions through the coordination or ion–exchange reactions, and their empty cavities can reduce the mass–transfer resistance and improve the flux. For example, Zhang et al. prepared a neoteric uranium adsorbent, a diaminomaleonitrile (DAMN) –modified chromium terephthalate (III) double-shell hollow (DSHM) metal–organic skeleton, by using the post-synthesis method of coordinating an unsaturated site by grafting amino groups [15]. This was a new idea of using a hollow–MOF adsorbent to remove radioactive particles from seawater. The hollow MOFs have abundant exposed and accessible unsaturated metal sites, providing more chances for the coordination of $–NH_2$ and –C=N with uranium ions. Adsorption experiments under simulated seawater conditions showed that DSHM–DAMN demonstrated a high selectivity for uranium, and the removal efficiency of uranium could still reach 85% in the presence of other particles. This is attributed to the use of a functionalized amino group as a hard base, which binds preferentially to uranium as a hard acid. Thus, DSHM–DAMN has a high affinity for uranium. Similarly, Cho et al. successfully used a ferrocyanide–functionalized ZIF–8 for efficiently and selectively removing radioactive Cs^+ from wastewater [16]. After modification with FC, a ZIF–8 with a hollow structure was obtained. Due to a higher affinity between FC and Cs^+, the composites had a high efficiency and selectivity for Cs^+ removal, with a maximum adsorption capacity of 422.42 $mg \cdot g^{-1}$. By adjusting the FC/ZIF–8 ratio between 0.4 and 0.8, the maximum adsorption capacity also changed from 257.41 to 422 $mg \cdot g^{-1}$. At the same time, in a wide range of pH, the composite materials had a relatively stable adsorption of Cs+, and adsorption capacity was more than 400 $mg \cdot g^{-1}$. Therefore, ZIF–8–FC can adsorb radioactive Cs^+ in various environments. In addition, Wang et al. synthesized a Fe_3O_4@ZIF–8 magnetic material and utilized it to adsorb heavy metal ions Cu^{2+} and Pb^{2+} [17]. The adsorption of Cu^{2+} and Pb^{2+} on Fe_3O_4@ZIF–8 can reach equilibrium in 20 and 60 min, and the values of Q_{max} are 724.4 and 301 $mg \cdot g^{-1}$, according to the Langmuir model. Moreover, the composite material can be reused at least four times. With the exception of inorganic ions, organic molecules such as drugs can also be quickly and efficiently removed by adsorption. For instance, Zhang et al. synthesized a tannic–acid–etched ZIF–8/GO–hollow–composite membrane for adsorbing organics and salts for the first time [18]. The hollow ZIF–8 enlarged the interlamellar spacing of GO sheets, promoted the formation of water channels, and notably reduced the transfer resistance of water. The prepared material showed good performance in the disposition of wastewater, with a more than 99% repulsive rate of the composite film to Congo red dye. Furthermore, Li et al. prepared MOF–808 for the adsorption of the anti-inflammatory drug diclofenac [19] and used yttrium-stabilized zirconia hollow fiber as a scaffold, which is expected to be used commercially. In this study, the Q_{max} of diclofenac reached 833 $mg \cdot g^{-1}$; meanwhile, the removal rate was close to 95% within one hour. In practice,

this structure solved the problem that the adsorbent was difficult to separate after the adsorption process finished. Additionally, the hollow fiber structure greatly improved the superficial area of adsorbents and solved the problem of uneven flow distribution, which was inevitable in the traditional, packed–bed adsorption tower. Similarly, Wang et al. combined a hollow Co–MOF–74 with electrospinning fiber membranes for removing polycyclic aromatic hydrocarbons by [20]. The composite material had strong hydrophobicity, outstanding mechanical properties, and, after adsorption, the filter and membrane were easy to recover when compared with powder MOFs. The synthesized, hollow nanofiber membranes maintained a high recovery rate after repeated reuse, and the adsorption capacity of seven PAHs reached 161–214 $mg \cdot g^{-1}$. All these results proved that H–NFMs had great application prospects in the field of water treatment. Surprisingly, not only single pollutants but also mixed, inorganic–organic liquid pollutants also can be effectively removed using hollow MOFs. Ma et al. prepared a multi-layer, coaxial MOF hollow tube through freeze-drying for removing inorganic–organic mixed liquid pollutants with a removal efficiency of 94% [21].

4. Other Applications

In addition to the above centralized applications, hollow MOFs also have a very wide range of other applications, such as for drug transport, electrochemical energy storage, electro-catalysis, and so on.

In recent years, the application of hollow MOFs in drug transportation and disease treatment has been a growing trend. Since the pore size, pore volume, and pore structure of hollow MOFs can be easily regulated by changing reaction parameters or adding active substances, they can be used as a potential platform for material embedding. Liu et al. synthesized a hollow Fe–MOF–5–NH_2 and grafted it with biomolecules [22]. The hollow Fe–MOF–5–NH_2 was able to hold more drugs, with a drug load of up to 35%, benefiting from its hollow structure. Drug release can be regulated by pH changes for more precise cancer treatment. Additionally, Jiang et al. prepared TCBPE–based nanotubes for the first time with a hollow structure that were capable of emitting intense fluorescence [23]. The embedded adriamycin anticancer medication was supplied in a self-directed manner, and this hollow MOF structure was very biocompatible and photostable. Drug loading is substantially higher than with pristine MOFs, reaching up to 36%, thanks to their hollow structure and strong π–π stacking. Similarly, the release of adriamycin is pH dependent. Hollow MOFs have been used as electrode materials due to their inorganic–organic hybrid properties and adjustable structure. However, the inherent poor conductivity of MOFs prevents their application in batteries to some extent. In general, this problem can be effectively solved by adding conductive materials. For example, Hao et al. reported a hollow, Co–MOF–74, porous structure composed of super-fine nanomaterials that was used as an anode for lithium-ion batteries [24]. By adjusting the reaction parameters to optimize the material, the optimized hollow Co–MOF–74 had a large specific surface area, coordination defects, and a hierarchical porous structure, which was conducive to lithium storage. It had a high reversible capacity (820 mAh g^{-1} at 1000 ma g^{-1}) and circulatory stability (996 mAh g^{-1} at 1000 mA g^{-1} after 470 cycles). Supercapacitors also attract great attention in the field of energy storage because of their advantages, such as fast charging and high energy density. Research on simple and high–performance supercapacitors has been in progress. Among many candidate materials, hollow MOFs have great application potential. Han et al. combined transition metal oxides with hollow

MOFs as electrode materials for self-sustaining batteries, and the synthesized Co–MOF@$CoCr_2O_4$ had a high specific capacity and excellent cyclic stability [25]. As for electro-catalysis, hollow–structure MOFs can effectively increase the reaction of active sites and improve the mass-transfer efficiency due to their abundant exposed active sites and large internal cavities, thus benefiting the electro-catalytic performance. Gu et al. reported a novel, in situ, simultaneous empty–doping method for the construction of bimetallic, doped, CoM–MOF hollow nanospheres for electrochemical hydrogen production [26]. Due to its rigid structure and the synergistic effect between the cavity and the bimetal, the charge–transfer resistance of the composite is only 42 Ω, and the phase-education of the solid Co–MOF has better electro-catalytic performance. In addition, hollow–MOF–derived materials have also been used in the field of electrochemical conversion [27][28][29][30]. By adjusting the morphologies of MOFs, the resultant materials can produce superior electro-catalytic properties in terms of energy conversion and storage applications.

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