Investigation of the aqueous adsorption capacity of a 6-connected Zr-MOF for anionic and cationic dyes in comparison with other traditional porous materials

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Abstract. A 6-connected Zr-MOF (MOF-808) was successfully synthesized via the solvothermal method with the assistance of formic acid (HCOOH). The resulting MOF showed high crystallinity and thermal stability, which was verified by powder X-ray diffraction (PXRD) measurement, scanning electron microscopy (SEM) and thermogravimetric analysis (TGA). As can be expected, the obtained material possessed high porosity with an extremely high specific BET surface area (SA_BET) of 2372 m²/g. The adsorption capacity of MOF-808 for anionic dyes (i.e. sunset yellow, quinoline yellow, and methyl orange) and cationic ones (i.e. methylene blue and malachite green) in aqueous solutions was respectively investigated. For comparison purpose, the adsorption experiments were also carried out using other traditional porous materials, including commercial microporous activated carbon and synthesized mesoporous SBA-15 with BET surface areas of 1030 m²/g and approximately 800 m²/g, respectively. It was demonstrated that the efficiency of MOF-808 in trapping anionic dyes in water was significantly higher while carbon and silica materials exhibited better performances for the case of cationic dyes.

1. Introduction

Water is the root of every creature on Earth and among indispensable elements of life. However, during the industrial revolution, the water sources, including both surface water and groundwater, were fouled by toxic effluents [1]. The explosive growth in manufacturing textile, painting, leather, pharmaceuticals and personal care products has led to the heavy discharge of organic toxins such as dyes, solvents, surfactants, and organometallic compounds into the water environment [2–6]. These compounds may be not only potentially carcinogenic, but also mutagenic to humans [7]. To address this concern, numerous researches employing different techniques, e.g. aqueous adsorption, catalytic decomposition, biodegradation and membrane separation, have been conducted [8–11]. Among them, adsorption is considered as a competitive strategy due to its undeniable advantages, including mild operation conditions, low investment and nonexistence of hazardous byproducts [12]. Studies regarding adsorption processes usually utilize porous materials such as activated carbon, zeolites and polymers since this removal process relies mainly on the porosity of used adsorbents [13–15]. However, the lack of controllable pores in these traditional trapping agents greatly hindered them from expanding their
capture scope. As an emerging class of porous materials, metal-organic frameworks (MOFs) have recently gained the attention in adsorptive removal of toxins with outstanding efficiency [16].

Metal–organic frameworks (MOFs) are geometrically well-defined materials, constructed by linking the metal–containing clusters and organic linkers to form open frameworks with colossal porosity [17]. Due to the diversity in combining of structural building blocks, more than 80 000 structures of MOFs have been reported with various pore sizes, leading to the wide range of MOFs applications [18]. Since the potential of this new platform in adsorption field was perceived, several MOFs have been employed in the aqueous removal of organic dyes [19-21]. Nonetheless, due to the water instability of the majority of MOFs, their use in wastewater treatment is still limited. In recent years, Zr-MOFs with remarkably improved water and chemical stability have been studied as promising adsorbents for aqueous adsorption [22-24]. By simply adding acetic acid or HCl into the precursor solution, the acid-promoted UiO-66 (12-connected Zr-MOF) was successfully synthesized and exhibited the selectivity towards anionic dyes (84.4 and 13.2 mg/g for methyl orange and methylene blue, respectively). The selectivity was not observed with the materials prepared without the assistance of acid [25]. In other circumstances, DUT-67 (8-connected Zr-MOF) was employed in removing mercury and methylmercury ions in aqueous solution with high efficiency (90 % and 55 %, respectively) [26]. However, studies about the aqueous trapping behavior of 6-connected Zr-MOFs have rarely been reported in the literature.

Herein, we report the adsorptive removal of organic dyes from aqueous solution utilizing MOF-808, which is a representative 6-connected Zr-MOFs, constructed from zirconium clusters and 1,3,5-benzenetricarboxylate linkers. Both anionic dyes (i.e. sunset yellow, quinoline yellow and methyl orange) and cationic dyes (i.e. methylene blue and malachite green) were employed in the study to clarify the capture tendency of MOF-808 towards different charged compounds. The comparison with other popular trapping agents such as activated carbon and SBA-15 (porous silica) was also executed.

2. Materials and Methods

All reagents and starting materials were purchased from commercial suppliers and were used as received without any further purification.

2.1. Synthesis of MOF-808

MOF-808 was prepared according to a modified synthetic method described in the previous study [27]. A mixture of ZrOCl₂·8H₂O (0.81 g, 2.5 mmol) and 1,3,5-benzenetricarboxylic acid (BTC) (0.18 g, 1.60 mmol) was dissolved in a mixture of DMF (37 mL) and HCOOH (38 mL) [27]. The obtained solution was mixed, capped tightly and then placed in an oven at 80 °C for 48 hours. The solid product was collected by decanting the mother liquor and washed in sequence with DMF (3×100 mL) and acetone (5×100 mL) at room temperature. The resulting product was eventually activated at 120 °C for 5 hours under a vacuum, giving 0.40 g of solid white powders of MOF-808 (Zr₆O₂(OH)₆(BTC)₉(HCOO)₃(H₂O)₂, M = 1354 g/mol, yield = 64 % based on ZrOCl₂·8H₂O).

2.2. Adsorption Study

Investigation of the adsorption capacity of MOF-808 was conducted for anionic dyes (i.e. sunset yellow, quinoline yellow, and methyl orange) and cationic ones (i.e. methylene blue and malachite green) (Figure 1) in water, respectively. A typical adsorption experiment was carried out in 15 ml of a solution containing 500 mg of dye/L with 10 mg of the fresh MOF-808 for 120 min at room temperature under vigorous stirring. After the adsorption, MOF-808 was separated from the solution by simple centrifugation. The concentration of sunset yellow, quinoline yellow, methyl orange, methylene blue and malachite green was determined by UV-Vis spectroscopy Thermo Scientific G10S UV-Vis device (Thermo Scientific, Waltham, Massachusetts, USA) at 481, 442, 464, 664 and 618 nm respectively.
2.3. Material Characterization

Nitrogen physisorption measurements were conducted using a Micromeritics 2020 volumetric adsorption analyzer system (Micromeritics, USA). Samples were pre-treated under a vacuum at 150 °C for 5 h. The specific surface area was calculated using the Brunauer-Emmett-Teller (BET) model in a relative pressure range of 0.05–0.3 p/p₀ while the pore size distribution was assessed using the density functional theory (DFT) method based on slip modern.

X-ray powder diffraction (XRD) patterns were recorded using a Cu Kα radiation source on a D8 Advance Bruker powder diffractometer (Bruker AXS, Germany) with a Ni filter. The measurements were performed in a 2θ range of 2–30 ° with an angular step size of 0.01 ° and scanning rate of 0.6 ° per min.

Morphology of the synthesized MOF-808 was confirmed by scanning electron microscopy (SEM) using a Hitachi SU 8010 FESEM microscope (Hitachi, Japan).

Thermal gravimetric measurements (TGA) were investigated using a TA Instruments SDT Q600 Thermal Gravimetric Analyzer (TA instruments, USA). In detail, the sample was placed in an alumina pan and heated from 40 to 900 °C with a ramping rate of 10 °C min⁻¹ under 60 mL min⁻¹ of airflow.

Fourier transform infrared spectroscopy (FT-IR) measurements were recorded using a Bruker Alpha instrument (Bruker AXS GmbH, Germany) equipped with a diamond crystal. Each spectrum was accumulated from 32 scans at a resolution of 4 cm⁻¹ recorded in the 4000–550 cm⁻¹ range.

3. Results and Discussion

3.1. Characterization of MOF-808

MOF-808 is a Zr-based MOF, consisting of Zr₆ node and 1,3,5-benzenetricarboxylate (BTC) linkers with 3D open channels as can be seen in Figure 2. In earlier synthesis procedures, the modulated synthesis strategy was employed to achieve large phase-pure Zr-MOF crystals [28]. The presence of modulator molecules (i.e. monocarboxylic acid and inorganic acid) is of paramount importance in tuning crystal features such as size, morphology, and crystallinity [27, 29]. In this study, MOF-808 was obtained via the solvothermal method with the assistance of formic acid (HCOOH). The powder X-ray diffraction (PXRD) pattern of the resulting material (Figure 3) showed reflections at 2θ of 4.2, 8.2, 8.6, 10.0, and 10.9° which were related to planes (111), (311), (222), (400), and (333), respectively. These signature peaks were sharp and narrow, indicating the high crystallinity of the synthesized MOF-808.
material. Moreover, the obtained XRD result was well-matched with the simulated patterns and previously reported ones [27, 30].

Figure 2. Schematic representation of MOF-808.

Figure 3. PXRD patterns of MOF-808.

Morphology of synthesized MOF-808 was then analyzed by scanning electron microscopy (SEM). It was observed that MOF-808 included large and regular octahedral crystals in the size of approximately 800 nm (Figure 4). The support of formic acid accelerated the formation of large and high-quality crystals, which was in good agreement with literature [29]. Because of the high valent state of Zr$^{4+}$ ions, their affinities towards carboxylate compounds were strong, leading to fast precipitation and possible structural disorder. In addition, the Zr centers in ZrOCl$_2$.8H$_2$O molecules are connected by bridging hydroxyl groups, which can reinforce the tendency to form amorphous zirconium hydroxides. Therefore, formic acid was employed in this synthesis procedure to compete with the organic linkers in binding into the metal nodes and reduce the growing speed of the crystals. Interestingly, in the case of MOFs with unsaturated metal centers less than 12-connected such as MOF-808, modulators also functioned as a structure directing agent by occupying the remaining coordination sites along with solvent molecules and stabilizing MOF structure [31].
Figure 4. SEM images of MOF-808

Besides, the porosity of MOF-808 was discovered upon nitrogen physisorption measurements. The physisorption behaviour of MOF-808 performed a typical type I isotherm (Figure 5a), which is represented for a typical microporous system with no further adsorbed nitrogen observed at relative pressure above 0.3 and no hysteresis between the adsorption and desorption isotherms. According to these adsorption data, BET surface area of the material was determined to be extremely high, 2372 m²/g, and the average pore size was in the range of 1.4–1.5 nm.

Figure 5. N₂ physisorption isotherm (left) and pore size distribution (right) of MOF-808.

Figure 6. FT-IR spectra of BTC linker and MOF-808 sample.
FT-IR spectroscopy was employed to detect chemical functional groups and structural properties (Figure 6). In the spectrum of the activated MOF-808 sample, the band of 1670–1550 cm$^{-1}$ belonged to the stretching vibration of the carboxylate group (-COO$^-$). The bands in the 1445 cm$^{-1}$ and 1380 cm$^{-1}$ regions are related to the C=C bond and the stretching mode of O–H in the aromatic ring of the organic linker. The band in the area of 760–655 cm$^{-1}$ was assigned to the vibrations of Zr-O in the MOF framework. In conclusion, the resulting spectrum exhibited a general shape similar to previous reports [32].

3.2. Adsorption Studies

According to outstanding chemical and thermal stability, zirconium-based MOFs were a new group of promising adsorbents for diverse organic pollutants in water [28, 33]. MOF-808 with the 6-connected structure contained a large number of uncoordinated Zr-nodes and hydroxyl groups. These groups played the roles as Lewis and Brønsted acid sites, thereby generating strong affinities to negatively charged ions, such as oxometallates and dye anions [29, 34]. In this study, the anionic dye trapping capacity of synthesized MOF-808 was investigated and compared with traditional porous materials including activated carbon and SBA-15 silica. Furthermore, the study was expanded with methylene blue (MB) and malachite green (MG) to investigate the efficiency of MOF-808 in the removal of cationic dyes.

As observed from Figure 7, MOF-808 displayed the excellent efficiency in capturing anionic dyes, with the capacity of 513, 672, and 666 mg/g for sunset yellow (SY), quinoline yellow (QQ), and methyl orange (MO), respectively. Activated carbon showed much lower uptakes for anionic dyes (i.e. 80-100 mg/g) while no efficiency in adsorption was observed for the case of SBA-15. This phenomenon could be explained by the lack of positively charged sites on the surface of SBA-15, leading to no affinity of silica towards anions [35]. In previous studies, to improve the adsorption capacity of activated carbon and silica for anions, these materials were modified with acid to increase the number of surface acidic sites [36].

![Figure 7](image_url)

**Figure 7.** Adsorption capacity for anionic dyes of MOF-808 ($S_A^BET = 2372 m^2/g$), commercial activated carbon ($S_A^BET = 1030 m^2/g$) and SBA-15 ($S_A^BET = 800 m^2/g$). * Data was collected from ref. [29].

When compared with other MOF-based materials, MOF-808 was among the top list of efficient adsorbents for anionic dyes (Table 1). The nature and functionalized UiO-66 (12-connected Zr-MOFs) showed modest performances in capturing MO anions (Entry 3–6_Table 1) [37-39]. Meanwhile, the structure of MOF-808 with reduced cluster connectivity facilitated the formation of additional acid sites, consequently enhancing the removal of anionic dyes. In addition, the trapping capacity of MOF-808
could be improved by controlling the amount of modulator in synthesis procedure [29]. Notably, Zr-porphyrinic MOFs including PCN-222 and PCN-224 exhibited excellent removals of MO with the capacity of 592 and 1001.4 mg/g, respectively (Entry 7–8, Table 1) [34, 40]. The best performance so far of PCN-224 for trapping MO was assigned to the presence of pores suitable for trapping MO molecules as well as multiple adsorption sites for MO anions [34]. However, it should be noted that the linkers used for the synthesis of such Zr-MOFs were indeed costly, thereby limiting their application scope. Less attention has been paid to Sunset Yellow and Quinoline Yellow with only a few reports found in these dyes, in which MIL-101 and MOF-199-mediated adsorption performance was poor (60–80 mg/g) as compared to MOF-808 (Entry 1–2, Table 1) [41,42]. It is obvious that the anion adsorption capacity of MOF-808 is impressive and promising for further investigation.

Table 1. Comparison of the anionic trapping capacity of MOF-808 with other MOF-based materials.

| No. | Sample                        | Adsorption Capacity (mg/g) | Ref.  |
|-----|-------------------------------|-----------------------------|-------|
|     |                               | Sunset Yellow | Quinoline Yellow | Methyl Orange |
| 1   | MOF-199                       | –              | 65.4            | –             | [41] |
| 2   | MIL-101@graphene oxide        | 81.3           | –               | –             | [42] |
| 3   | UiO-66                        | –              | –               | 83.7          | [37] |
| 4   | UiO-66 (nanoparticle)         | –              | –               | 172.5         | [38] |
| 5   | Fe_{3}O_{4}@SiO_{2}@UiO-66   | –              | –               | 219           | [39] |
| 6   | Fe_{3}O_{4}@SiO_{2}@UiO-66-NH_{2} | –     | –               | 130           | [39] |
| 7   | PCN-222                      | –              | –               | 592           | [40] |
| 8   | PCN-224                      | –              | –               | 1001.4        | [34] |
| 9   | MOF-808                       | 513            | 672             | 666           | This work |

Figure 8. Adsorption capacity of cationic dyes onto MOF-808 ($SA_{BET} = 2372$ m$^2$/g), commercial activated carbon ($SA_{BET} = 1030$ m$^2$/g) and SBA-15 ($SA_{BET} = 800$ m$^2$/g).

The removal of cationic dyes was also investigated to extend the application scope of MOF-808 (Figure 8). The adsorption capacities of MOF-808 were 249 and 237 mg/g with respect to MG and MB. Meanwhile, SBA-15 showed higher uptakes of 324 and 271 mg/g for MG and MB, respectively. Interestingly, the cation trapping capacities of activated carbon were recorded at 736 and 528 mg/g, respectively, which were 3-fold and 2-fold higher than the values obtained with MOF-808. It could be
concluded that these carbon and silica materials have the strong affinity to cations, which is attributed to the large number of oxygen atoms in surficial Si=O and C=O groups [43, 44]. However, it cannot be denied that MOF-808 also exhibited comparable activity in trapping cationic dyes due to strongly polarized functional group and high porosity.

Similar to MOF-808, UiO-66 was built up based on the Zr core, but its original generation showed a poor performance in the removal of MB (Entry 3, Table 2) [37]. Notably, Song and co-workers have recently synthesized another UiO-66 form with pores enlarged to the meso range (~ 3.4 nm) [45]. As expected, this pore expansion resulted in a dramatic capacity improvement with the MB uptake of 543.5 mg/g [45]. PCN-222, a Zr-MOF with the mesoporous structure (~ 3.2 nm), also showed an excellent MB adsorption capacity of 906 mg/g (Entry 5, Table 2) [40]. Therefore, increasing the pore size of Zr-MOFs should be intensively investigated as an efficient approach to enhance adsorption activity. Besides, functionalizing acidic Al-based MOFs with electron-rich amino groups was applied to obtain NH$_2$-MIL-100(Al) as an ideal adsorbent for cationic dyes (Entry 2, Table 2) [46]. Only a composite of Cu-MOF and Fe$_3$O$_4$ was previously used to remove MG from water with a significantly lower capacity of 114 mg/g [47]. Obviously, the cation trapping activity of MOF-808 was considerable without any further modification or special linker.

The removal of dyes from aqueous solution using MOFs was suggested to be conducted via both chemical and physical routes [34]. Weak interactions such as electrostatic and π−π interactions and relatively strong interactions such as hydrogen bonding and coordination bonding can be generated between dye molecules and the MOF surface [48-50]. It was indicated that adsorption behavior was closely related to material properties and dye structure. In the case of MOF-808, the structure containing a low number of cluster connectivity (6-connected structure) possessed uncoordinated Zr sites which readily formed acidic hydroxyl groups (Zr-OH) in an aqueous phase. The −SO$_4$ group of the anionic dyes was therefore attracted by such Brønsted sites [34, 51, 52]. In addition, similar to other Zr-MOFs such as UiO-66, the adsorption of MOF-808 could be obtained by π−π interactions between the aromatic rings in the dye molecule and in the BTC linker [37]. Notably, the acid-base interaction between the Zr cluster and the organic dyes seemed to be dominant in the removal of dyes using MOF-808 since the trapping capacity for anionic dyes was superior to cationic ones.

**Table 2.** Comparison of the cationic trapping capacity of MOF-808 with other MOF materials.

| No. | Sample | Adsorption Capacity (mg/g) | Ref. |
|-----|--------|---------------------------|------|
| 1   | Cu-MOF$_x$/Fe$_3$O$_4$ | 113.7 | Malachite Green | [47] |
| 2   | NH$_2$-MIL-100(Al) | – | Methyl Blue | [46] |
| 3   | UiO-66 | 69.8 | | [37] |
| 4   | UiO-66 with expanded pore diameter* | – | 543.5 | [45] |
| 5   | PCN-222 | – | 906 | [40] |
| 6   | MOF-808 | 249 | 237 | This work |

As mentioned above, Zr-MOFs are well-known with high hydrolytic stability, therefore after aqueous adsorption under ambient conditions, their crystalline structure could be preserved. Molavi and co-workers synthesized UiO-66 for the adsorptive removal of methyl orange and methyl blue [37]. In this study, using PXRD measurements, the authors proved that the UiO-66 crystallinity remained almost unchanged over four adsorption cycles. A similar result was observed in the work of Zhang et. al. on using a functionalized UiO-66 to remove organic dyes from water [51]. However, it was noticed that pore walls in the MOF-808 framework could be broken up during adsorption and regeneration, leading to a significant drop in porosity, e.g. from 3291 to 1481 m$^2$/g after the fourth use as reported in literature [29]. Therefore, intensive investigation of structural characteristics of MOF-808 after the adsorption experiment should be investigated to gain more insight into the stability and potential of MOF-808 for aqueous applications.
4. Conclusion
A 6-connected zirconium-based MOF, MOF-808, was simply synthesized upon the solvothermal treatment and applied as a highly efficient absorbent for the aqueous removal of organic dyes. Due to possessing the prominent features, i.e. abundant acidic sites, high surface area and high water stability, MOF-808 showed outstanding trapping capacities at ambient conditions for anionic dyes in comparison with activated carbon, mesoporous silica and many previously reported MOFs while no further surface modification was needed. On the other hand, the adsorption efficiency of MOF-808 for cationic dyes was comparable to that of SBA-15 and other MOFs. Our obtained results demonstrated the great potential of this Zr-MOF in the adsorption of polluting anions in water. More intensive studies on MOF-808 should be conducted with a focus on trapping highly toxic anions at extremely low concentration and improving its adsorption capacity via different methods.

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