Research Article

An Effective Microwave-Assisted Synthesis of MOF235 with Excellent Adsorption of Acid Chrome Blue K

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An efficient, rapid, and fast kinetics of the metal-organic framework MOF235 was successfully prepared by microwave-assisted thermolysis strategy. Fourier transform infrared spectra (FTIR), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), N₂ adsorption-desorption isotherm, and scanning electron microscopy (SEM) were used to characterize the samples. Experimental results revealed the suitability of MOF235 for use as an adsorbent for acid chrome blue K. The maximum adsorption capacity of acid chrome blue K onto MOF235 can reach to 591.79 mg g⁻¹ at 293 K. The sorption behavior fitted to the pseudo-second-order kinetic model and the Langmuir isotherm. Adsorption of acid chrome blue K is a spontaneous and endothermic process. Therefore, the as-prepared MOF235 displays a great potential for environmental purification.

1. Introduction

Organic dyes are typical pollutants found in many important industries and receive major worldwide concern. Dyes are main organic pollutants because they are toxic or carcinogenic to mammals and other living organisms even at low concentrations [1]. Efficient removal of organic dyes from wastewater has become a severe problem due to its ecological, biological, and environmental importance [2]. Acid chrome blue K is an azo dye and has been used as a spectrophotometric and electrochemical reagent for protein assay [3].

Dye effluent is usually treated by conventional methods such as adsorption, coagulation, electrochemical degradation, and photocatalysis. Adsorption is considered as one of the simplest, highly effective, and economically beneficial methods to remove the dye [4]. Effective removal of these dyes from aqueous solution is gaining more and more interests [5].

Traditional adsorbent materials, such as clay minerals, activated carbon (AC), carbonaceous materials, ordered mesoporous carbon, carbon nanotubes, and graphene oxides (GO), have been widely applied in the adsorption of organic dyes. However, these adsorbent materials generally have several disadvantages in dye adsorption including small pore

sizes or pore volumes and low adsorption capacity [6]. Therefore, the development of advanced porous materials for more effective and inexpensive adsorption is highly valuable [7].

In parallel to the conventional porous materials, much research effort has been devoted to find new adsorbent. Metal-organic frameworks (MOFs) show many interesting characteristic features such as a large specific surface area, low framework density, versatile functionality, controlled porosity, and tailorable structure [8]. These excellent properties have attracted increasing attention for adsorption [9], catalysis, storage, and electrochemistry in recent years [10].

The MOF235 is orange hexagonal single crystals. The crystal structure of the MOF235 is built up from corner sharing octahedral iron trimers. Linear terephthalic acid is connected, and each iron atom is trivalent. MOF235 synthesized by a solvothermal method has been investigated, and it performs high capacity to adsorb CH₄ due to the high pore volume and large number of open metal sites [11].

Microwave-assisted synthesis has been widely revealed distinct advantages as an alternative method for the chemical synthesis of organic and inorganic materials [12]. Microwave irradiation has attracted much attention. It has the

advantages of short reaction time, high reaction rate, and conservation energy.

It has been demonstrated that the microwave-assisted synthesis was one of the most efficient routes to carry out many reactions [13]. Microwave irradiation can accelerate the crystallization of porous materials that require several days or several hours compared with the conventional solvothermal synthesis [14]. A great diversity of MOFs has been prepared quickly and in high yield by a microwave-assisted routes, such as MOF-5, MIL-88B, MIL-53, and UiO-66. The microwave irradiation power, irradiation time, concentration of the reagents, and solvent system were deeply investigated [15]. Microwave heating have a beneficial effect on the properties and performance of materials [16].

In this work, orange octahedral crystals of MOF235 were successfully prepared by a facile and green synthetic route under microwave heating. Moreover, the adsorption capacity for acid chrome blue K with MOF-235 is about 591.9 mg g^{-1} . The textural and chemical properties of the MOF235 were reported. The adsorption kinetics and mechanism were investigated using the adsorption isotherm technique. The effectiveness of the MOF235 in the adsorption was evaluated. This work provided a fast preparation method for MOF235 and development of a highly successful and efficient adsorbent in acid chrome blue K wastewater treatment.

2. Materials and Methods

2.1. Materials. Ferric chloride hexahydrate (FeCl_3), terephthalic acid (Sigma-Aldrich, >99%), *N,N*-dimethylformamide (DMF), and absolute ethanol were purchased from Shanghai Chemical Reagent Inc. of the Chinese Medicine Group. All reagents were used as received.

2.2. Synthesis of MOF235. In a typical synthesis, a mixture of 0.3015 g terephthalic acid was dissolved in 30 mL DMF solvent and the mixture was stirred for 10 min. Then, 0.3000 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was added into the solution and stirred for 10 min; the reactant mixture of 30 mL and 30 mL of ethanol was dispersed into the round Pyrex flask [11]. The round flask was put into ultrasonic and microwave-assisted extraction (Shanghai CW-2000) and irradiated simultaneously by microwaves under the protection of nitrogen for a specified period. After cooling down to room temperature, the orange resultant solid was collected by centrifugation, washed with DMF-ethanol (1:1, *v/v*) mixture and finally dried in a vacuum at 423 K for 12 h and the orange octahedral crystals of MOF235 were obtained.

2.3. Adsorption Test. In a typical preparation process, 10 mg of MOF235 was added to a solution with 50 mL of acid chrome blue K at initial concentration from 40 ppm to 140 ppm. The solution was shaken under mechanical conditions for different times. The residual concentration of the dyes was determined at the maximum wavelength using UV-vis spectroscopy. The adsorption capacities were calculated using the calibration curve.

2.4. Characterizations. The synthesized MOF235 were thoroughly characterized by various sophisticated analytical tech-

FIGURE 1: XRD patterns of the as-synthesized MOF235 with different microwave times.

niques. X-ray diffraction (XRD) experiments were conducted using an X-ray diffractometer with the Cu target (SmartLab) operating at 36 kV and 25 mA from 5 to 70°. Fourier transform infrared (FTIR) spectra of powder samples were obtained in the $400\text{--}4000 \text{ cm}^{-1}$ range with a resolution of 4 cm^{-1} using a Nicolet Nexus 870 FTIR spectrometer. X-ray photoelectron spectroscopy (XPS) analysis was carried out in the Thermo Scientific K-Alpha spectrometer using monochromated Al K α X-rays. Ultraviolet absorbance spectra were collected on a Shimadzu UV 3600 plus spectrophotometer over a range of 200–800 nm. Nitrogen adsorption-desorption isotherms were measured at 77 K by multipoint BET and a Barrett-Joyner-Halenda (BJH) method using a Micromeritics ASAP 3020 analyzer. The samples were heated at 150°C for 12 h prior to each test. The morphology and microstructure were observed by a field emission scanning electron microscope (SEM: Hitachi S-4800). Thermogravimetric analysis was determined from room temperature to 800°C with a heating rate of 10°C/min using a Pyris1 TGA-1.

3. Results and Discussion

3.1. Characterization of MOF235. The crystalline nanostructures and phase purities of the MOF235 samples were recorded with X-ray diffraction (XRD) in Figure 1. Strong major peaks of the samples were observed at $2\theta = 9.7^\circ$, 10.8° , 12.6° , 19.0° , 22.0° , and 26° , which are in good consistent with the previously reported literature [17]. In the XRD patterns, the minimum microwave time to obtain complete crystallinity MOF235 was required for 15 min. The sharp crystalline of the reflections derived from the MOF235 increases with the prolonged reaction time under microwave. This indicated that the well crystallinity of MOF235 in the samples is proportional to the reaction time about 20 min [18, 19]. The results described the advantages of the effective and convenient microwave-assisted method. The intensities of all crystallinity peaks which increased gradually in short periods could be attributed to the fast heating precursor and avoid thermal gradients [20, 21]. It also demonstrated that the increased irradiation time leads to high yields and

FIGURE 2: FTIR of the as-synthesized MOF235 with different microwave irradiation times.

FIGURE 3: Nitrogen adsorption-desorption isotherms of the MOF235.

larger crystals [22]. It indicated that microwave heating was more effective than the convenient heating.

Fourier transform infrared (FTIR) spectra of the MOF235 were shown in Figure 2. All of the samples display similar spectra in general. The main spectra peaks of MOF235 were at 3440 cm^{-1} , 2931 cm^{-1} , 1597 cm^{-1} , 1398 cm^{-1} , 1016 cm^{-1} , 810 cm^{-1} , 748 cm^{-1} , respectively. The peaks observed at 1597 cm^{-1} were attributed to the asymmetric C–O bonds. The peaks at 1398 cm^{-1} were attributed to symmetric stretch vibrations in the carboxyl groups. According to the lower frequencies, the bands around 810 cm^{-1} , 748 cm^{-1} , and 710 cm^{-1} were attributed to the mixture with the C–H vibration, C=C stretch, OH bend, and O–C–O bend in H_2BDC [23].

Nitrogen adsorption isotherms of the samples prepared under different preparation conditions are shown in Figure 3. All samples confirmed a type I sorption isotherm, indicating a typical microporous structure. The results

FIGURE 4: TG of the as-synthesized MOF235.

revealed that the BET surface area was significantly increased from 444.25 , 583.20 , 602.72 , 620.53 , and $729.21\text{ m}^2\text{ g}^{-1}$ of the samples by microwave heating time. It proves that the microwave-assisted preparation of MOF235 is feasible and efficient. The pore size distribution is an important influence factor of an adsorbent. It will have a great impact on the size and shape of a given dye molecules. The average pore size of MOF235 is 4.13 nm . When the adsorbent pore diameter is as large as 1.7 times that of the adsorbate, adsorption can reach to the most effective results [24]. However, the amount of micropores decreased significantly after the incorporation. The increase in the BET surface area and micropore volume of the MOF235 could be attributed to the loss of the well-ordered structure; the pore can be an efficient space for large substrates which allows the dye to go inside the pore and react with MOF235.

The thermal stability of MOF235 was carried out by TGA to investigate the material stability and the correlation between the reaction time and the structural integrity. As shown in Figure 4(a), the TGA curves of all the samples exhibit a degradation at the temperature of 400°C . The weight loss of the samples MOF235 showed similar thermal curves. The maximum structural integrity and robustness of the MOF235 were achieved with a microwave irradiation time of 25 min. Moreover, the thermal stabilities of these MOF235 were consistent with those obtained using the previously reported solvothermal approach [25]. With the extended response time from 5 to 10 min, the sample crystals were still underdeveloped and with some unreacted precursors attached in the pore. The full growth of crystals was completed after 15 min. The TGA curve results showed that the MOF235 have high thermal stability in the adsorption dye reaction [26].

X-ray photoelectron spectral (XPS) analyses were applied to analyze the elemental distribution, surface composition, and bond of the samples and their valence states [27]. It shows that the sample surface consists of carbon, nitrogen,

FIGURE 5: XPS spectra of MOF235: (a) survey, (b) C1s, (c) Fe2p, and (d) N1s.

oxygen, and iron, with binding energies of C1s, N1s, O1s, and Fe2p, respectively (Figure 5(a)). As shown in Figure 5(b), the fitting result of the C1s spectrum of MOF235 suggests the existence of two peak components of C=C and C-C (284.65 eV) and O-C=O (288.67 eV), indicated that there were two types of chemical environments for carbon in the samples [28]. The two characteristic peaks at 711.90 and 725.80 eV (Fe2p region) indicated the presence of Fe^{3+} species (Figure 5(c)). The peak centered at 400.7 eV can be assigned to graphitic N, in agreement with the N states in N/C (Figure 5(d)), indicating the change of chemical environment of nitrogen in MOF235.

The MOF235 morphology of the microwave heating method under different times was studied by SEM in Figure 6. The well-crystallized octahedral morphological materials are built up from corner-sharing octahedral iron trimers that are connected through terephthalic acid linear links. The Fe_3O plane of each trimer has $\text{Fe}-(\mu_3\text{-O})\text{-Fe}$ angles (120°) and the two adjacent Fe ion separation of $3.33 \text{ \AA}$ [17]. The octahedral crystallites have the particle size around

450 nm and a uniform size distribution. The SEM images show that the materials have an obvious advantage of a higher degree of crystallinity with the extension of microwave irradiation time.

3.2. Adsorption Properties of Acid Chrome Blue K on MOF235. Adsorption equilibrium of acid chrome blue K onto MOF235 was studied at six initial concentrations of 40, 60, 80, 100, 120, and 140 ppm. As shown in Figure 7, it was clearly shown that the adsorption quantity of acid chrome blue K increased sharply within 50 min and no marked changes are observed with further increases in contact time. It is suggested that the samples were allowed reacting for 200 min to ensure adsorption equilibrium. During the desorption process, the equilibrium adsorption amounts of acid chrome blue K were 94.81, 189.60, 259.84, 366.05, 464.84, and 591.79 mg g^{-1} , respectively. The results indicated that the adsorption amounts were improved with the increase of the initial concentrations and it could reach the

FIGURE 6: SEM micrographs of MOF235: (a) 10 min, (b) 15 min, and (c) 15 min.

FIGURE 7: Adsorption amounts of acid chrome blue K with different initial concentrations.

maximums of 591.79 mg g^{-1} from the 140 ppm initial concentration aqueous solution [29].

Adsorption kinetics were investigated to study the mechanism of adsorption which is important for adsorption efficiency of the acid chrome blue K in aqueous solutions [30]. The pseudo-first-order and pseudo-second-order models

were both usually applied to the experimental data [31]. The models are used to explore the potential rate controlling the phenomenon in the adsorption of acid chrome blue K onto MOF235 [32]. The adsorption rate is proportional to the square of the number of unoccupied adsorption sites, and the rate equation is

$$\frac{dq}{dt} = k_2(q_e - q)^2, \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{q_e^2 k_2} + \frac{t}{q_e},$$

where q_e and q_t (mg/g) refer to the amounts of acid chrome blue K adsorbed at equilibrium and at time t , respectively. k_2 ($\text{g mg}^{-1} \text{ min}^{-1}$) represents the rate constants [33]. It is obvious that the experimental kinetic data fitted better with the pseudo-second-order rate model. The higher coefficients of R^2 and lower normalized standard deviation are better than those in the pseudo-first-order kinetic model [34]. As shown in Figure 8, all the coefficients of the pseudo-second-order model were higher than 0.999 for all the initial acid chrome blue K concentrations at 293 K. It implied that the

FIGURE 8: Langmuir plots of the isotherms.

adsorption process was a combination of physical and chemical adsorption [35].

The pseudo-first-order kinetics model is a widely used monolayer adsorption model. The equilibrium adsorption data of acid chrome blue K on MOF235 was further evaluated through Langmuir and Freundlich isotherm equations [36, 37]. The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical adsorption sites [29]. The Freundlich isotherm is applied for multilayer adsorption of adsorbates with a heterogeneous surface and a nonuniform distribution of adsorption heat [38]. Their linear forms are as follows:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_0} + \frac{1}{Q_0 b}, \quad (2)$$

where q_e is the amount of adsorbate adsorbed per unit mass of adsorbent (mg g^{-1}), C_e is the retained adsorbate concentration at equilibrium (mg L^{-1}) [39], Q_0 is the maximum adsorption capacity, and b is the adsorption energy, obtained from the slope and intercept of the plot of C_e/q_e against C_e , respectively [40], and found to be 131.8 mg g^{-1} and 0.0485 L mg^{-1} with the correlation of 0.995.

4. Conclusions

In summary, the microwave-assisted thermolysis strategy is simple, rapid, and robust, thereby providing a promising route for the synthesis of MOF235. Acid chrome blue K can be efficiently removed with MOF235. The adsorption kinetics and adsorption isotherms were investigated in detail. All of the MOF235 exhibited high adsorption capacities and fast adsorption kinetics. It is indicated that MOF235 under microwave can be applied in the highly attractive adsorbent for the removal of acid chrome blue K.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

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