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ARTICLE TYPE

Facile synthesis of magnetic hierarchical MgO-MgFe₂O₄ composites and their adsorption performance towards Congo red

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MgO-MgFe₂O₄ composites with magnetism and adsorption capacity for Congo red were synthesized by an ion exchange route using magnesium carbonate as templates. The X-ray powder diffraction (XRD), scanning electron microscopy (SEM), and magnetic hysteresis loop were carried out to characterize the crystalline phase, microstructure, and magnetic property of as-obtained MgO-MgFe₂O₄ composites. Congo red adsorption experiments were taken to evaluate the adsorption property of the as-synthesized samples. The results showed that the as-synthesized MgO-MgFe₂O₄ composites have hierarchical structure, magnetism and adsorption performance. The optimized sample exhibited an excellent adsorption capacity (498 mg g⁻¹) for Congo red, which is much higher than many other magnetic hierarchical materials.

1. Introduction

Many textile and printing industries discharge a large amount of wastewater that contains organic dyes which is characteristic of toxicity, carcinogenicity and teratogenicity, so it will pose a threat to the living organisms if the highly coloured effluent is not treated properly.^{1,2} Up to now, a wide range of technologies have been used for the removal of toxic dyes from wastewater, including membrane filtration, chemical precipitation, biological treatment, adsorption, and photochemical degradation. Among them, adsorption is considered as the most effective and popular method because of its simplicity, high efficiency and low energy consumption.³⁻⁹

In the past few years, due to the development of nanotechnology, many efforts have been devoted to integrate magnetic components onto micro-nano sized hierarchical structure. Hierarchical structure exhibits excellent adsorption capacity due to their large specific surface area, and magnetic materials with hierarchical structures not only have potential in adsorption, but also can be easily separated by external magnetic field. For example, Yu, et al developed a hydrothermal method to prepare mesoporous TiO₂ modified with γ -Fe₂O₃, and the as-prepared γ -Fe₂O₃-TiO₂ showed good removal capacity for Congo red;¹⁰ Yin's group reported an ion exchange route of synthesis for magnetic hierarchical porous carbon sphere (MHCS) and its excellent ability of adsorbing the organic dye methyl orange;¹¹ Yao and co-workers presented a magnetic Fe₃O₄@graphene nanocomposite via a chemical deposition method, and the nanocomposite showed utilization in dye removal.¹² In spite of these successes, there are still two major challenges when using these magnetic hierarchical structures for adsorption. One comes from the comparatively low adsorption capacity and efficiency, because magnetic materials always exhibit low affinity toward

organic dyes. The other problem is the complicated synthesis methods, conventional synthesis methods of magnetic hierarchical materials need a lot of surface active agent, and the reaction should be taken under solvothermal or hydrothermal conditions, which limits the scale-up synthesis of these magnetic materials.

Recently, our group has developed a large scale synthesis approach to fabricate porous hierarchical MgO with superb adsorption property.¹³ The hierarchical MgO exhibited large specific surface area, multiple reaction channel and strong electrostatic interaction with dye molecule and thus was favourable for effective adsorption. In this present work, starting from hierarchical MgO, facile synthesis of magnetic hierarchical MgO-MgFe₂O₄ composite was created via using magnesium carbonate as template. This method is facile, low-cost, and can be easily scaled-up.^{14,15} Synthesis factors such as annealing temperature and the adding amount of FeCl₂ were investigated, in order to find the optimum condition for synthesis of the MgO-MgFe₂O₄ composite. The as-synthesized MgO-MgFe₂O₄ composites exhibited good adsorption performance for removing Congo red dye and magnetic separation performance from water. Congo red (C₃₂H₂₂N₆O₆S₂Na₂, CR), an azo anionic dye, is commonly used as a model pollutant to investigate the dye removal ability of adsorbents.^{10,12,20-22} In this paper, the optimized product showed enhanced adsorption capacity (498 mg g⁻¹) for Congo red within short time (2 min) and good magnetic separation performance (M=11.4emu/g), while most magnetic adsorbents reported in literature can only perform a adsorption capacity of lower than 200mg g⁻¹.^{10,12,20-22} Moreover, the adsorption isotherms and kinetics involved in the adsorption process were also discussed, which would provide a theoretical foundation for further applications of system design in the removal of hazardous pollutants.

2. Experimental

2.1 Chemicals and materials

All chemicals were of analytical reagent-grade. Magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), ammonium bicarbonate (NH_4HCO_3), ferrous chloride ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The Congo red stock solution (1000 mg L^{-1}) was prepared by dissolving 1000 mg Congo red powder into 1000 mL deionized water. Experimental solutions for adsorption and analysis were freshly prepared by diluting Congo red stock solution with deionized water.

2.2 Preparation of magnetic adsorbents

The hierarchical magnesium carbonate ($4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$, MCH) templates were prepared by a magnesium bicarbonate pyrolysis method as reported in our previous work¹⁴ (SI). And the $\text{MgO-MgFe}_2\text{O}_4$ composite were synthesized via a facile ion exchange route. Firstly, 0.8g template was added to 80mL of ethanol in a beaker and fully ultrasonic dispersed. Then, under continuous stirring, different amount of FeCl_2 aqueous solution (1 mol L^{-1}) were added into the suspension. After a further reaction at room temperature for 2 h, the stirring was stopped. And the as-obtained precursor was filtered off, washed several times with ethanol, and dried in blast drying oven at $60 \text{ }^\circ\text{C}$ for 4 h. Afterwards, the precursor was annealed in air at different temperatures (ranging from 400 to $700 \text{ }^\circ\text{C}$) for 4 h to obtain the hierarchical $\text{MgO-MgFe}_2\text{O}_4$ composites.

2.3 Characterization

The shapes and microstructures of as-synthesized samples were observed by scanning electron microscope (SEM, JEOL-6360LV, operated at 20kv). Powder X-ray diffraction instrument (XRD, Rigaku-DMAX 2400) was employed to investigate the crystalline phases of the samples in reflection mode (Cu-K α radiation) at scanning rate of 0.02 S^{-1} in the 2θ from 5 to 80° . The magnetization curve was measured on a Quantum Design MPMS-7 SQUID magnetometer under a varying magnetic field between -20Koe and 20Koe at room temperature, aiming to detect the magnetic properties of the samples.

2.4 Adsorption experiments

Congo red (CR) was chosen as a model molecule to determine the adsorptive property of the as-synthesized $\text{MgO-MgFe}_2\text{O}_4$ composite functioned as adsorbent. Desired concentrations (100 – 700 mg L^{-1}) of CR solution were obtained by diluting the CR stock solution with deionized water. CR removal capacity of the flowerlike $\text{MgO-MgFe}_2\text{O}_4$ composite was carried out in a batch system. 0.08 g adsorbent was added to 80 mL CR solution with different concentrations, unless otherwise stated. The mixture was firstly ultrasonic dispersed, and then the conical flask containing the mixture solution was fixed on a shaker at room temperature (Constant-temperature Shaker, ZHWY-100H, Shanghai, China). Adsorption experiments were carried out for 60 min to ensure that equilibrated reaction was obtained, except for kinetic study where the mixture solution were withdrawn from the conical flask at pre-selected time intervals. At the end of the equilibration, the hierarchical $\text{MgO-MgFe}_2\text{O}_4$ composite with adsorbed CR was separated by a magnet, and the remaining CR concentration in solution was analyzed by UV-Vis spectroscopy

(Hitachi U-4100) at 490 nm . For adsorption isotherm study, adsorption experiment was carried out by various initial CR concentrations ranging from 100 mg L^{-1} to 700 mg L^{-1} . The amount of CR adsorbed q_e (mg g^{-1}) and removal percentage R were calculated based on the formulas as given below:

$$q_e = \frac{(C_0 - C_e) \times V}{W} \quad (1)$$

$$R = \frac{(C_0 - C_e)}{C_0} \times 100\% \quad (2)$$

where C_0 (mg L^{-1}) and C_e (mg L^{-1}) are initial and equilibrium concentrations of CR solution, respectively; V (L) is the volume of CR solution, and W (g) is the dry weight of the adsorbent.

2.5 Identification of Fe content in the $\text{MgO-MgFe}_2\text{O}_4$ composite

In order to test the content of Fe in the $\text{MgO-MgFe}_2\text{O}_4$ composite, titration experiment was conducted using EDTA standard solution as complex agent and sulfosalicylic acid as indicator. The titration process need to be done under the circumstance of pH 1.5 – 2.5 and 60 – 70°C , and the $\text{MgO-MgFe}_2\text{O}_4$ composite should be fully oxidized by hydrogen peroxide to ensure that Fe^{2+} has changed to Fe^{3+} before the titration experiment was taken. Therefore, the content of Fe in the $\text{MgO-MgFe}_2\text{O}_4$ composite can be calculated as the chemical reaction formula (3). For $\text{MgO-MgFe}_2\text{O}_4$ composites synthesized by adding different amount of FeCl_2 in reaction, the Fe content of the final products is listed in Table 1.

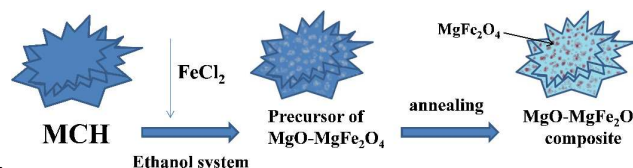
$$w(\text{Fe}\%) = \frac{\text{Fe}(\text{mol}) \times M_{\text{Fe}}}{m(\text{MgO}@/\text{MgFe}_2\text{O}_4)} \times 100\% = \frac{\text{EDTA}(\text{mol}) \times M_{\text{Fe}}}{m(\text{MgO}@/\text{MgFe}_2\text{O}_4)} \times 100\% \quad (3)$$

Table.1 Fe content in the $\text{MgO-MgFe}_2\text{O}_4$ composites with different adding amount of FeCl_2

The adding amount of FeCl_2 (mL)	1	2	3	4
Fe content (%)	6.0	11.8	17.8	23.7

3. Results and discussion

3.1 Synthesis of $\text{MgO-MgFe}_2\text{O}_4$ composites



Scheme.1 Schematic illustration of the synthesis of flower-like $\text{MgO-MgFe}_2\text{O}_4$

Scheme 1 illustrates the synthesis process of $\text{MgO-MgFe}_2\text{O}_4$ composites. In the first step, the precursor could be easily obtained by adding FeCl_2 to suspension of $4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ (MCH) and ethanol at room temperature: ($\text{MCH} + \text{FeCl}_2 \rightarrow \text{MCH-Fe}_a(\text{CO}_3)_b(\text{OH})_c + \text{MgCl}_2$). Previous study of our group has demonstrated that the hierarchical MCH particles can easily react with metal inorganic salts to form morphology preserved metal carbonates.^{14,15} In the second step, the $\text{MgO-MgFe}_2\text{O}_4$ composites were obtained by heat treating the precursor ($\text{MCH-Fe}_a(\text{CO}_3)_b(\text{OH})_c \rightarrow \text{MgO-}$

$\text{MgFe}_2\text{O}_4 + \text{CO}_2 + \text{H}_2\text{O}$).

3.2 Structure and morphology analysis

The XRD patterns of products are shown in Fig.1 and Fig.2. It can be seen that the diffraction peaks of the products in each case all correspond to the lattice of MgO with the corresponding diffraction peaks of (200), (220) and the lattice of MgFe_2O_4 with the corresponding diffraction peaks of (220), (311), (222), (422), (511), and (440), however, when the adding amount of FeCl_2 is more than 3 mL, the diffraction peak of Fe_2O_3 appears. The XRD patterns of $\text{MgO-MgFe}_2\text{O}_4$ composites obtained under different heating temperatures are presented in Fig.1, which indicates that 400°C may not be the proper annealing temperature because the diffraction peaks of MgFe_2O_4 are very weak. However, with the increase of annealing temperature, diffraction peaks of MgFe_2O_4 gradually become stronger and sharper, suggesting that a calcination temperature of 500°C or higher will be beneficial to fully form $\text{MgO-MgFe}_2\text{O}_4$ composite. The grain size of MgFe_2O_4 were determined to be 6.2nm (400°C), 9.9nm (500°C), 12.9nm (600°C) and 18.9(700°C) by Scherrer equation ($D=K\lambda/B\cos\theta$).

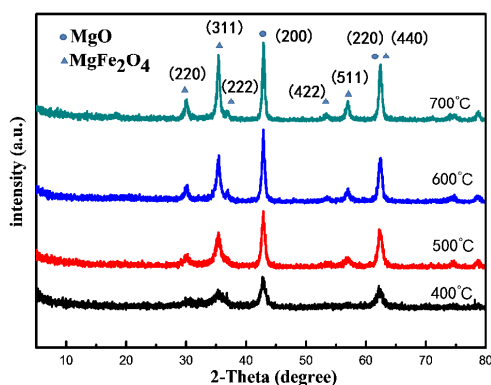


Fig.1 XRD patterns of $\text{MgO-MgFe}_2\text{O}_4$ composites with different annealing temperatures (the addition amount of FeCl_2 is certain for 2mL)

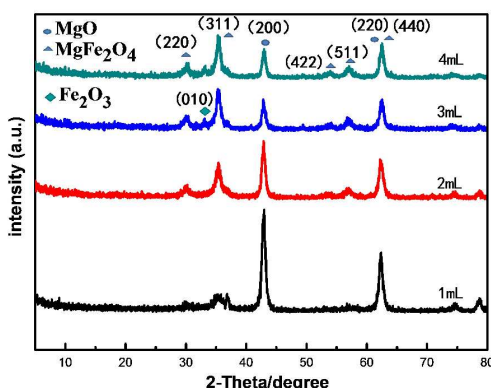


Fig.2 XRD patterns of $\text{MgO-MgFe}_2\text{O}_4$ composites with different MgFe_2O_4 content (annealing temperature is certain for 500°C)

Fig.2 shows the XRD patterns of $\text{MgO-MgFe}_2\text{O}_4$ composites with different MgFe_2O_4 content, it also clearly reveals that with the increase of the addition amount of FeCl_2 in reaction, diffraction peaks of MgFe_2O_4 gradually strengthen, and 2 mL or

more addition amount of FeCl_2 is favourable for the formation of the $\text{MgO-MgFe}_2\text{O}_4$ composite. The grain size of MgFe_2O_4 were determined to be 5.6 nm (1mL), 9.9 (2mL), 10.5 (3mL) and 12.5nm (4mL).

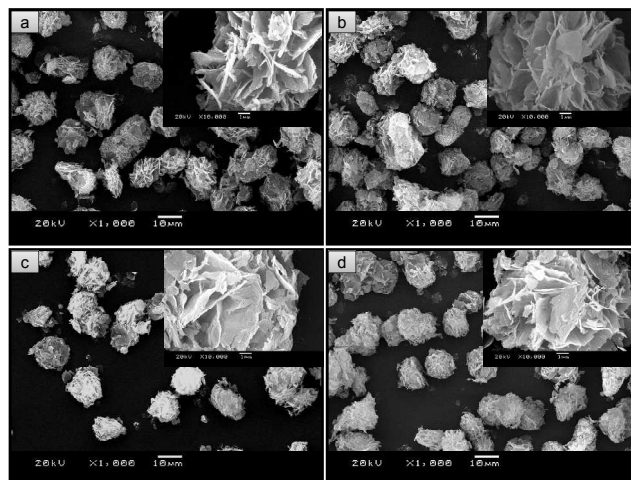


Fig.3 SEM images of $\text{MgO-MgFe}_2\text{O}_4$ composites under different annealing temperatures (the addition amount of FeCl_2 is certain for 2mL) (a) 400°C (b) 500°C (c) 600°C (d) 700°C

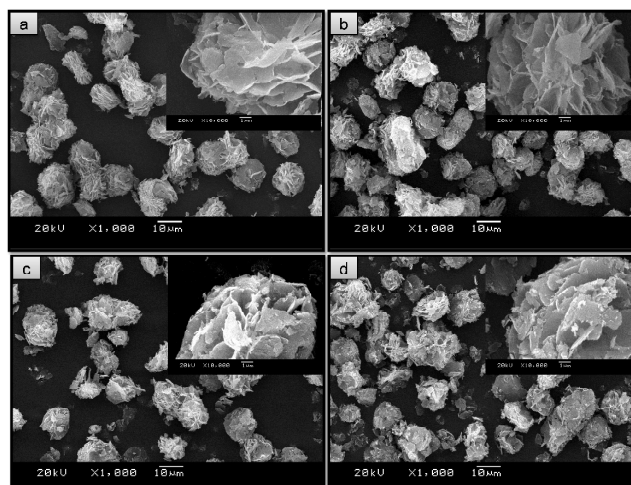


Fig.4 SEM images of $\text{MgO-MgFe}_2\text{O}_4$ composites with different MgFe_2O_4 content (the annealing temperature is certain for 500°C) (a) 1mL FeCl_2 (b) 2mL FeCl_2 (c) 3mL FeCl_2 (d) 4mL FeCl_2

Fig.3 shows SEM images of the $\text{MgO-MgFe}_2\text{O}_4$ composites annealed under different temperatures, it can be seen that all the products can maintain the hierarchical microstructure of the template (Fig.S1) without collapsing, which implies that the subsequent heating temperature have little impact on the morphology. Moreover, Fig.4 presents that with the increase of the addition amount of FeCl_2 , and the $\text{MgO-MgFe}_2\text{O}_4$ composites begin to lose the shape of MCH template and collapse. Thus, proper addition amount of FeCl_2 will play a vital role in maintaining the flowerlike architecture.

3.3 Magnetic property

Magnetic properties of the as-synthesized $\text{MgO-MgFe}_2\text{O}_4$ composites were further studied by magnetic hysteresis measurements under room temperature. Magnetic hysteresis

loops can be observed in Fig.5 and Fig.6. It can be seen that no sample has evident hysteresis, i.e. both remanence and coercivity are zero, indicates that the MgO-MgFe₂O₄ composites are soft magnets. To be specific, saturation magnetization intensity of product annealed under 400 °C is relatively low (4.02 emu/g), which could be due to the reason that MgFe₂O₄ is not fully formed. However, when the temperature increases to 500 °C, the MgO-MgFe₂O₄ composite begins to show strong magnetic behaviour with a saturation magnetization intensity of 11.4 emu/g. Otherwise, when the annealing temperature continues to rise, the magnetism of MgO-MgFe₂O₄ composites increases, but the increasing trend is not obvious. Fig.6 shows the room temperature magnetization curves of MgO-MgFe₂O₄ composites with different MgFe₂O₄ content, it can be deduced that due to the increase of mass contribution from magnetic MgFe₂O₄, the magnetic property of the adsorbent strengthens. The Magnetic property of the MgO-MgFe₂O₄ composites is related to their grain size. The size of the MgFe₂O₄ particle is comparatively small and the atom on surface is not coordination unsaturated, resulting in the strong heat disturbance and inconsistency of magnetic moment with external magnetic field.²³

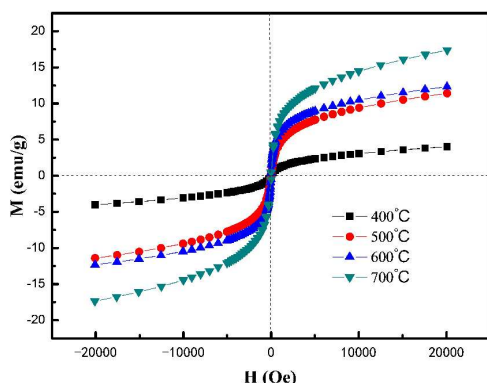


Fig.5 Magnetization curves of MgO-MgFe₂O₄ composites under different annealing temperatures

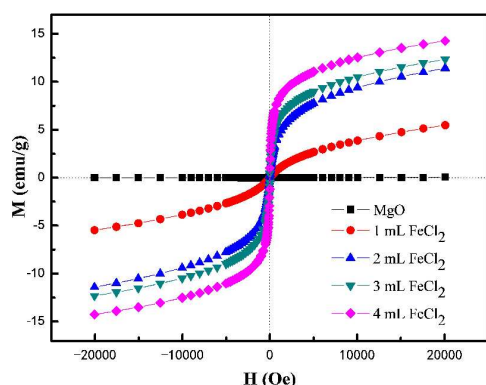


Fig.6 Magnetization curves of MgO-MgFe₂O₄ composites with different MgFe₂O₄ content and magnetization curve of MgO

3.4 Adsorption performance

To investigate the adsorption performance of MgO-MgFe₂O₄ composite, adsorption experiment was conducted by adding 0.02g adsorbent to 50 mL Congo red (CR) solution of 200 mg L⁻¹, after fully dispersing and shaking for predetermined time to ensure the adsorption equilibrium, the magnetic adsorbents were

magnetically separated (as show in Fig. S2). Then the corresponding dye concentrations in the solutions were measured by UV-Vis spectrometer. Fig.7 shows the UV-Vis absorption spectra of remained CR solutions after using MgO-MgFe₂O₄ composites annealed under different temperatures. It reveals that with the increase of calcination temperature, higher concentration of CR remained in the solution, representing a declination of adsorption performance. So rising annealing temperature may not be favourable to the adsorptive property of MgO-MgFe₂O₄ composite. As is seen in Fig.8, the UV-Vis spectra of remaining CR solutions after using adsorbents with different MgFe₂O₄ content are discussed, it demonstrates that with increase of the mass ratio of MgFe₂O₄ to MgO increased, resulting in decrease the adsorptive property of MgO-MgFe₂O₄ composite.

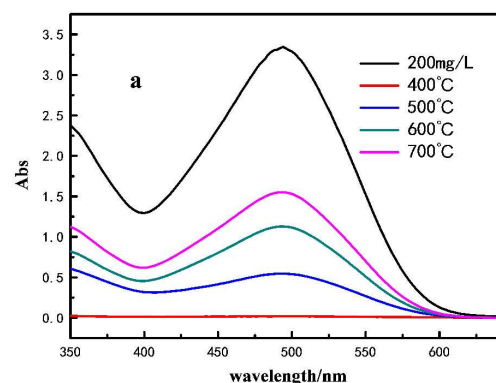


Fig.7 UV-Vis absorption spectra of CR solutions after treated by MgO-MgFe₂O₄ composites annealed under different temperatures.

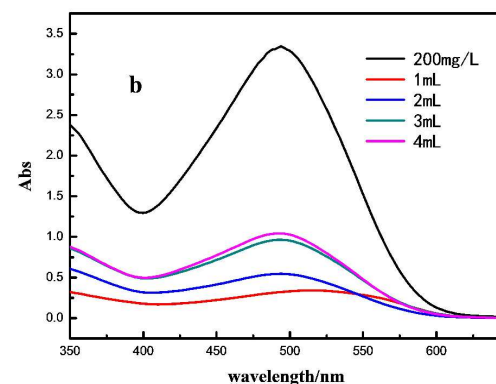


Fig.8 UV-Vis absorption spectra of CR solutions after treated by MgO-MgFe₂O₄ composites with different MgFe₂O₄ content

3.5 Adsorption isotherms and kinetic reactions of the optimized MgO-MgFe₂O₄ composite

3.5.1 Adsorption isotherms study

Adsorption experiment of adding 0.08g adsorbent to 80mL CR solutions (ranging from 100 mg L⁻¹ to 700 mg L⁻¹) was taken to investigate the removal capacity of the MgO-MgFe₂O₄ composite prepared under optimal condition (adding 2mL FeCl₂, annealing at 500°C). To investigate how CR molecules interact with the MgO-MgFe₂O₄ composite, two well know models, the Freundlich¹⁶ and Langmuir¹⁷ isotherms, were selected to analyze equilibrium adsorption data. The Freundlich isotherm describes the non-ideal adsorption of a heterogeneous system and reversible adsorption,¹⁸ and the model can be expressed as formula (4). The Langmuir isotherm is based on the assumption of a structurally

homogeneous adsorbent and the monolayer coverage. The equation is given as formula (5). q_e is the amount of dye adsorbed per unit of adsorbent (mg g^{-1}), C_e is the concentration of dye solution at adsorption equilibrium (mg L^{-1}), K_f and n is the Freundlich adsorption isotherm constant in formula (4), where q_m is the maximum amount of adsorption which completes monolayer coverage on the adsorbent surface (mg g^{-1}) and K_L is the Langmuir constant (L mg^{-1}) in formula (5).

$$q_e = K_f C_e^{1/n} \quad (4)$$

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (5)$$

Non-linear-regression of the two adsorption isotherms were employed to investigate the dye-adsorbent interaction and the results of fitting Langmuir and Freundlich equations to isotherm curves are summarized in Table 2. Fig.9 shows that the adsorption of CR onto the optimized MgO-MgFe₂O₄ composite can be better described by the Langmuir model for the entire concentration range, whereas, fitting to the Freundlich isotherms may not be applicable. Also, correlation coefficient (R^2) of Langmuir isotherm is 0.969, while R^2 of Freundlich isotherm is only 0.714, which further indicates that the adsorption conforms perfectly to Langmuir model. Significantly, the maximum

Table 2 Langmuir and Freundlich isotherm parameters for adsorption of CR on the MgO-MgFe₂O₄ composite

model	Langmuir			Freundlich			
	parameter	$q_m(\text{mg g}^{-1})$	$K_L(\text{Lmg}^{-1})$	R^2	K_f	n	R^2
CR		498	3.871	0.969	293.3	8.659	0.714

adsorption capacity (q_m) calculated from the Nonlinear simulation equation was 498mg g^{-1} , it was notable in Table 3 that the maximum adsorption capacity of the optimized MgO-MgFe₂O₄ composite is much higher than those of many other magnetic composites.^{10,12,19-22} This value is even higher than some hierarchical or porous adsorbents without magnetic property,²⁴⁻²⁷ clearly demonstrating the magnetic composite presented in this work is excellent candidate in designing magnetically separable super-adsorbents for removing organic dye from wastewater.

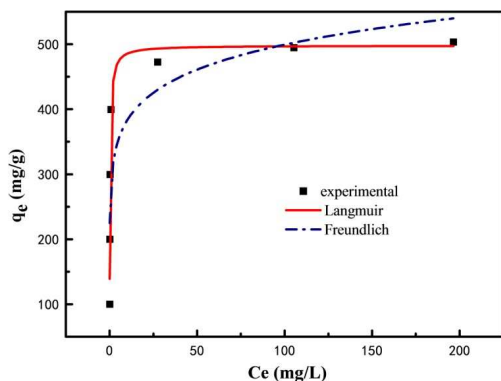


Fig.9 Adsorption isotherm curves of CR adsorption on the optimized MgO-MgFe₂O₄ composite.

Table.3 Adsorption capacities of CR dye on various adsorbents

adsorbent	Adsorption capacity (mg/g) references	
Hierarchical MgO	2409	[13]
Hierarchical hollow FeOOH	275	[19]
Maghemite nanoparticles	208	[20]
Mesoporous $\gamma\text{-Fe}_2\text{O}_3\text{-TiO}_2$	125	[10]
$\text{Co}_3\text{O}_4\text{-Fe}_3\text{O}_4$ hollow spheres	125	[21]
Fe_3O_4 /activated carbon	66	[22]
Fe_3O_4 @graphene	34	[12]
MgO-MgFe ₂ O ₄ composite	498	This paper
Hierarchical hollow NiO	440	[24]
Mesoporous activated carbons	189	[25]
MgO nanoplates	131	[26]
Mesoporous Fe_2O_3	53	[27]

3.5.2 Adsorption kinetic study

To understand the adsorption kinetics, the effect of contact time on the adsorption of CR onto MgO-MgFe₂O₄ composite was investigated by adding 0.08g of optimized sample to 80mL CR solutions (ranging from 200 mg/L to 400 mg/L). The amount of CR adsorbed by MgO-MgFe₂O₄ composite as a function of time is presented in Fig10. It can be seen that approximately 99% of the CR can be adsorbed within the first 2 min and adsorption equilibrium could be achieved in a short time.

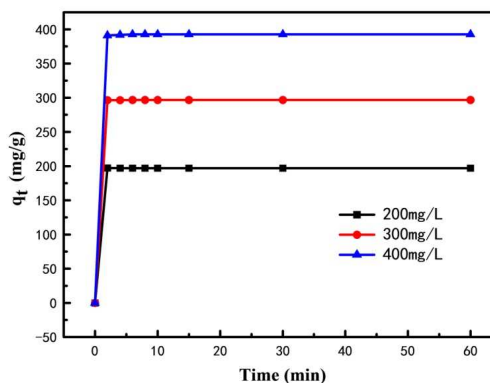


Fig.10 Time profiles of CR adsorption on the MgO-MgFe₂O₄ composite

The adsorption kinetics was obtained by fitting the experimental data with pseudo-second-order kinetic model,²⁸ and the equation is given below:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

where q_e (mg g^{-1}) is the amount of CR adsorbed at equilibrium, q_t (mg/g) is the amount of CR adsorbed at time t , t (min) is the adsorption time, and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the rate constant of pseudo-second-order adsorption. The curves of pseudo-second-order kinetics of CR adsorption onto the composite is shown in Fig.11, From Fig.11, it can be deduced that the adsorption process fits quite well with pseudo-second-order model. Also, The obtained correlation coefficients ($R^2 > 0.9999$) further suggest that the adsorption followed the pseudo-second-order kinetic model perfectly, indicating that the chemical adsorption was the rate-limiting step.²⁹

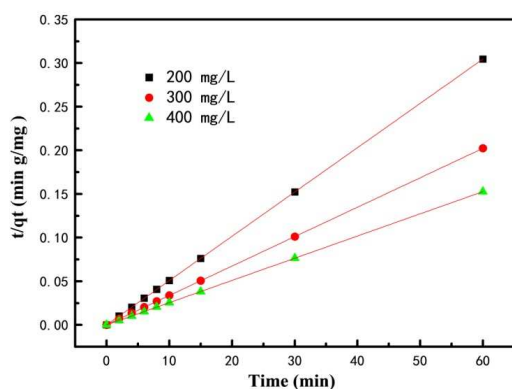


Fig.11 The pseudo-second-order kinetics plots of CR adsorption on the MgO-MgFe₂O₄ composite

Conclusions

In summary, we have demonstrated a facile method to prepare a series hierarchical MgO-MgFe₂O₄ composite with both excellent adsorption performance and good magnetic property for removing Congo red from water. The MgO-MgFe₂O₄ composite prepared under optimal condition shows attractive merits as follows: (a) High adsorption capacity. The MgO-MgFe₂O₄ composite shows an excellent adsorption performance of 498 mg g⁻¹ for CR, which is much higher comparing to many previous works including magnetic and non-magnetic adsorbing materials. (b) Rapid adsorption equilibrium. Adsorption kinetic study shows that adsorption equilibrium of CR molecules onto MgO-MgFe₂O₄ composite can be achieved only after 2 min, which allows fast processing in industrial application. (c) Easy separation. The strong magnetization property favors the MgO-MgFe₂O₄ composite to be separated by an external magnetic field, and it is verified that the MgO-MgFe₂O₄ composite that adsorbed CR can be easily separated by magnet. These attractive merits of MgO-MgFe₂O₄ composite can meet the need of practical application for fast treatment of large volume CR contaminated water.

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Notes

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†Electronic Supplementary Information (ESI) available: Preparation and SEM images of MCH template and Illustration of the application process of MgO-MgFe₂O₄ composites. See DOI:10.1039/b000000x/

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A table of contents entry

MgO-MgFe₂O₄ composites have been synthesized by a facile method, and showed enhanced adsorption capacity and good magnetic separation performance.

