Preparation of magnetic cobalt-cuprum-zinc ferrite nanoparticles and their adsorption mechanism of methyl blue

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Abstract

Magnetic cobalt-cuprum-zinc ferrites were prepared from anhydrous ethanol using the combustion method, and their structure and properties were characterized using the XRD, SEM, EDS, and VSM techniques, and its formation mechanism was discussed. The magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles calcined at 400 °C with 25 ml anhydrous ethanol were used for the removal of methyl blue (MB). The results showed that the pseudo-second-order kinetic model best agreed with the adsorption method. In addition, analysis of the adsorption isotherms using the Freundlich, Langmuir, and Temkin models showed that the Temkin model was most consistent with experimental results, which revealed that the adsorption of MB onto the Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles was a multi-molecular layer chemisorption. Further, the influence of pH on the adsorption capacity was evaluated and was highest at pH 11. The cyclability and removal rate of the nanoparticles were explored. The removal rate was approximately 80% after 7 cycles, revealing that the magnetic Co\(_x\)Cu\(_y\)Zn\(_{1-x-y}\)Fe\(_2\)O\(_4\) nanoparticles are important for wastewater treatment.

1. Introduction

The improvement in societal development has brought about better living conditions and an increase in environmental pollution, especially water pollution. Wastewater contains huge amount of nondegradable substances; when disposed into the environment, animals and plants, and humans absorb toxic substances from wastewater, which affects public health [1]. Water is an indispensable resource for life; thus, resolving water pollution is critical. Dyestuff, plastics, chemical plants, and other industrial wastewater are important sources of water pollution [2]. In addition, dye pollution has high organic content and many harmful substances, which affects the growth of microorganisms in water, damages water structure, and pollutes the environment. Therefore, it is also essential to resolve dye pollution from the environment. Dye wastewater treatment methods include adsorption, membrane separation, chemical oxidation, and photocatalytic oxidation methods [3, 4]. Among them, the adsorption method has some advantages such as simplicity of operation, high adsorption ability and stability, and no secondary pollutions [5, 6]; therefore, the adsorption technology is widely applied in the removal of dyes from solution.

Adsorbents play an important role in the adsorption process. Common adsorbents are activated carbon and alumina, and silicone [7]. With the development of nanotechnology, nanoparticles are employed to adsorb dyes with large specific surface area. However, nanoparticles are difficult to separate from solutions owing to their nanosizes. Magnetic nanoparticles can effectively overcome this weakness; they have advantages such as large specific surface area, selective adsorption ability, high chemical stability, and low cost. After adsorption of dyes,
magnetic nanoparticles can be separated from solutions under the effect of an applied magnetic field [8]. Therefore, magnetic nanoparticles are often used to treat polluted waters.

As one of the significant magnetic nanomaterials, magnetic cobalt-cuprum-zinc ferrites has attracted much attention. Approaches used for the preparation of magnetic cobalt-cuprum-zinc ferrites include the gas-phase condensation, sol-gel, coprecipitation, and the combustion methods [9–11]. These methods have some disadvantages: The gas-phase condensation method requires expensive equipment, which are difficult to operate; the sol-gel method requires high-temperature calcination; and the product purity of the coprecipitation method is low. On the other hand, the combustion method has simple operation requirements and a low cost. In addition, it requires a short production cycle and only one solvent for the process, and the properties of its product can be easily controlled by the solvent volume. Therefore, the combustion method is often used for the preparation of magnetic nanoparticles [12, 13].

In this study, magnetic cobalt-cuprum-zinc ferrites were prepared using the combustion method and characterized by relevant instruments, and its adsorption mechanism was investigated using methyl blue (MB) as the adsorption model.

2. Experimental detail

2.1. Materials and chemicals

Anhydrous ethanol (99.7%, AR), ferric nitrate nonahydrate [Fe(NO3)3·9H2O, 98.5%, AR], zinc nitrate hexahydrate [Zn(NO3)2·6H2O, 99.0%, AR], cobalt nitrate hexahydrate [Co(NO3)2·6H2O, 98.5%, AR], copper nitrate hydrate [Cu(NO3)2·3H2O, 99.0%, AR], and methyl blue (MB, BS) were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China).

2.2. Preparation and characteristics of magnetic cobalt-cuprum-zinc ferrites

Magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles were prepared using the combustion method. At room temperature, anhydrous ethanol (25 ml) was poured into a beaker, and Fe(NO3)3·9H2O, Zn(NO3)2·6H2O, Co(NO3)2·6H2O, and Cu(NO3)2·6H2O were added successively. The solution was then agitated in a magnetic field until a uniform solution was formed and set for combustion. After combustion, the burnt products were calcined at 400°C for 2 h, and ground to get the final product.

The phase identification of magnetic cobalt-cuprum-zinc ferrites was performed by x-ray diffraction (XRD, Philips APD-10 x-ray diffractometer with Cu Kα radiation, Japan); the magnetic measurement was taken on a vibrating sample magnetometer (VSM, HH-15, Physicence Opto-electronics Co., Ltd, Beijing, China). Moreover, the morphology and composition were investigated through scanning electron microscopy (SEM, JSM-TOOIF, JEOL Ltd, Japan) and energy dispersive spectroscopy (EDS, JSM-TOOIF, JEOL Ltd, Japan).

2.3. Adsorption behaviors of MB onto the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles

To investigate the mechanism of MB adsorption onto the magnetic cobalt-cuprum-zinc ferrites, the kinetics experiments were imperative. After preliminary experiments, it was observed that the adsorption rate of MB was highest when the concentration of Co0.4Cu0.2Zn0.4Fe2O4 in solution reached 2.5 mg mL−1 while the concentration of MB wastewater was often within the range of 200–500 mg L−1. At room temperature, the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles (5 mg) were added into the centrifuge tubes containing 2 ml MB solutions (200–500 mg mL−1), dispersed using ultrasonic waves for 5 min, and the liquid was stirred and rotated for corresponding certain time. To enhance the adsorption capacity of cobalt-cuprum-zinc ferrites for MB, 1 M sodium hydroxide (NaOH) and hydrochloric acid (HCl) solutions were used to change the pH values of the MB solutions. To reveal the interactive relationship between magnetic cobalt-cuprum-zinc ferrites and MB, adsorption isotherms were analyzed using an initial MB solution (300 mg L−1). At the end of the adsorption process, the magnetic cobalt-cuprum-zinc ferrites were separated from the solution under the effect of an applied magnetic field, and the absorbance of the liquid supernatant was measured using an ultraviolet spectrometer. For the cyclic utilization of the nanoparticles, the used magnetic cobalt-cuprum-zinc ferrites were recalcined at 400 °C for 2 h, and reused in the subsequent adsorption process. The adsorption capacities (qt) of MB onto the magnetic cobalt-cuprum-zinc ferrites were calculated using the equation (1) [14].

\[
q_t = \frac{(C_0 - C_t) \cdot V}{m}
\]  

where \(C_0\) and \(C_t\) (mg L−1) are the concentrations of MB before and after the adsorption at time (t, min), respectively. V (L) is the volume of the MB solution, and \(m\) (g) is the weight of the magnetic cobalt-cuprum-zinc ferrites.
3. Results and discussion

3.1. Characterization of the Magnetic CoxCuyZn1-x-yFe2O4 Nanoparticles

Figure 1 shows the Fourier-transform infrared (FTIR) spectra of the precursor gel and cobalt-cuprum-zinc ferrites prepared at various temperatures. The prepared magnetic cobalt-cuprum-zinc ferrites were further analyzed using FTIR spectroscopy within the wavelength range 400–4000 cm\(^{-1}\). Magnetic cobalt-cuprum-zinc ferrites were not generated in the precursor; the FTIR showed that the stretching vibration of the hydrogen bond (O–H) in water was found at 3400 cm\(^{-1}\). Moreover, the stretching vibrations of the double bond for the carbonyl (C=O) and nitro (–O–N=O) groups were observed at 1630 and 1384 cm\(^{-1}\), respectively. The vibration of the hydroxyl group (–OH) was observed at 1045 cm\(^{-1}\), and the flexural oscillation of the carbon-hydrogen (C–H) bond was observed at 829 cm\(^{-1}\). Increasing the calcination temperature to 400 °C caused the bonds detected in the precursor to be invisible under the FTIR; however, new substances could be found at 573 cm\(^{-1}\). Further, when the calcination temperature was increased to 500 and 600 °C, no chemical bonds were observed under the FTIR spectrometer, indicating that no other chemical bonds were formed; nonetheless, substances with wavelength 573 cm\(^{-1}\) could be seen. This might be because higher temperatures break chemical bonds, prompting new ones to form. It was confirmed that the new peak formed at 573 cm\(^{-1}\) was because of the existence of the Fe–O bond, which indicated the formation of magnetic cobalt-cuprum-zinc ferrites. The results also showed that magnetic cobalt-cuprum-zinc ferrites could be formed at 400 °C. When magnetic cobalt-cuprum-zinc ferrites were produced, the calcination temperature could be directly raised to 400 °C [15, 16].

Figure 2 shows the characteristics of the Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles prepared at 400 °C for 2 h using anhydrous ethanol (25 ml). The Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles were spherical with an average diameter of approximately 20 nm (figure 2(A)), and their elemental composition is shown in figure 2(B). It was observed that the proportion of each element in the nanoparticles was approximately consistent with the pre-experimental hypothesis. Figure 2(C) shows the XRD pattern of the Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles calcined at 400 °C for 2 h. It could be seen that, the peaks of the as-prepared product completely conformed to the standard PDF card of the Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles. The hysteresis loop of the Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles are shown in figure 2(D). This showed that increasing the magnetic field, increased the magnetization of the Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles until saturation (the magnetic saturation intensity was approximately 50 emu·g\(^{-1}\)), indicating that these are soft magnetic particles.

The average particle size, peak height, and peak width were important factors influencing the degree of crystallization of the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles. In addition, when the x and y values of the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles changed, their peak and average particle sizes also changed (figure 3). The y value was varied from 0.1 to 0.8 (figure 3(A)) and the highest peak of the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles was observed at y = 0.2. Moreover, the x value was varied from 0.1 to 0.7 (figure 3(C)) and the widest peak of the magnetic Co0.4Cu0.2Zn0.4Fe2O4 nanoparticles was observed at x = 0.4. In figures 3(B) and (D), the average grain size increased as the y value was changed from 0.1 to 0.8, and the x value from 0.1 to 0.7; however, as the y value was changed from 0.1 to 0.2 and the x value from 0.1 to 0.4, the average grain size of the nanoparticle decreased. Further, at y = 0.2 and x = 0.4, the degree of crystallization of the
magnetic Co_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2}O_{4} nanoparticles was lowest. Combining figures 3 and 4 showed that as the average grain size decreased, the magnetic properties also decreased and the peak became wider, which influenced the crystallization effect. It was shown that the larger the peak, the smaller the average grain size, the lower the magnetization, and the lower the degree of crystallization of the magnetic Co_{0.4}Cu_{0.2}Zn_{0.4}Fe_{2}O_{4} nanoparticles.

When the material content was changed by varying the x and y values, the magnetic value was determined. The average size of the nanoparticle (d) could be calculated by the Scheller equation (2) [17].

\[
d = \frac{0.89 \lambda}{B \cos \theta}
\]

where B is the maximum half-width of the diffraction, \( \theta \) is the Bragg diffraction angle, and \( \lambda \) is the wavelength of x-ray.

The magnetism of magnetic cobalt-cuprum-zinc ferrites was also an important factor affecting the adsorption process. Factors influencing the magnetic performance include material content, concentration of particles, surfactant, particle size, and temperature [18, 19]. The effect on the changes in the x and y values on the magnetic properties of magnetic cobalt-cuprum-zinc ferrites was shown in figure 4. At \((x, y) = 0.1\), the magnetization of the magnetic cobalt-cuprum-zinc ferrites was weak, exhibiting a superparamagnetic phenomenon. In addition, as the particle size decreased, the magnetic moment decreased, and the magnetic property weakened. However, as the particle size increased, the magnetic moment increased, and the magnetic properties strengthened [20]. Temperature also affects the Curie temperature of the magnetic cobalt-cuprum-zinc ferrites, affecting their magnetic properties [21].

The relationship between magnetization and magnetic field strength of the magnetic cobalt-cuprum-zinc ferrites with changes in x and y of the material content was shown in figure 4. It was obvious that magnetic cobalt-cuprum-zinc ferrites would eventually reach magnetic saturation regardless of the change in the x or y values. Moreover, as y varied from 0.1 to 0.5, the magnetization of magnetic cobalt-cuprum-zinc ferrites gradually became stronger, and the coercivity gradually became weaker (figure 4(A)). On the other hand, as y varied from 0.5 to 0.8, the magnetization of magnetic cobalt-cuprum-zinc ferrites became weaker and the coercivity became stronger. At \( y = 0.5 \), the maximum magnetic saturation intensity was approximately 75 emu g\(^{-1}\), and the hysteresis loss and coercivity were insignificant. Further, when the x value changed, the magnetization of the magnetic cobalt-cuprum-zinc ferrites did not change.

Figure 2. SEM morphology (A), EDX spectrogram (B), XRD pattern (C) Hysteresis loop (D) of magnetic cobalt-cuprum-zinc ferrites calcined at 400 °C for 2 h using 25 ml anhydrous ethanol.
significantly (figure 4(B)). At $x = 0.3$, the maximum magnetic saturation intensity was approximately 52 emu g$^{-1}$, and the hysteresis loss and coercivity were insignificant. Magnetic cobalt-cuprum-zinc ferrites are soft magnetic materials. The magnetization saturation was due to the change of most magnetic domains in the magnetic cobalt-cuprum-zinc ferrites.

### 3.2. Adsorption kinetics of MB onto Co$_{0.4}$Cu$_{0.2}$Zn$_{0.4}$Fe$_{2}$O$_{4}$ nanoparticles

To study the relationship between time and the adsorption capacity of the Co$_{0.4}$Cu$_{0.2}$Zn$_{0.4}$Fe$_{2}$O$_{4}$ nanoparticles for MB, three dynamic models were applied: the pseudo-first-order, pseudo-second-order, and intraparticle models [22].
The pseudo-fist-order kinetics was suitable for the adsorption process in the liquid phase (formula (3)). The pseudo-second-order kinetics was suitable for electron transfer between the adsorbents and adsorbates (formula (4)). Intraparticle kinetics model was used to describe the channel diffusion mechanism (formula (5)) [23, 24].

\[
q_t = q_e(1 - e^{-k_1t}) \tag{3}
\]

\[
q_t = \frac{q_e^2k_1t}{1 + q_ek_2t} \tag{4}
\]

\[
q_t = x_i + k_i t^{1/2} \tag{5}
\]

where \(q_t\) and \(q_e\) (mg·g\(^{-1}\)) are the adsorbed MB amount at any time \((t, \text{min})\) and at equilibrium, respectively. \(k_1\) (min\(^{-1}\)), \(k_2\) (g·mg\(^{-1}\)·min\(^{-1}\)), and \(k_i\) (mg·g\(^{-1}\)·min\(^{-0.5}\)) are the adsorption rate constants of the three models, respectively, and \(x_i\) (mg·g\(^{-1}\)) is the boundary layer thickness.

The adsorption capacities of MB on magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles at different concentrations were correlated with time (figure 5). With an initial concentration for MB, the adsorption capacity first increased and reached saturation with time. With a further increase in the initial concentration of MB, the adsorption capacity gradually increased and reached saturation in a shorter time. The reaction rate slowed down, finally stabilized, and reached saturation. The adsorption capacity of MB on the Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles could not reach saturation because the surface-active sites were occupied and the adsorption was stopped.

The results collected in the experiment were described using \(q_t\) and \(t\), and the experimental data were analyzed using these three models (figure 6). Among these three models, the graph of the pseudo-second-order model was most consistent with the experimental data, while the graph of the other two models showed a large deviation from experimental data. According to data graph analysis, the pseudo-second-order model was most suitable for describing the adsorption process of MB on the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles. The parameters of these three kinetic models were listed in table 1. The association coefficient \((R^2)\) of the pseudo-second-order model was larger than those of the other two models. In addition, the closer the association coefficient of the model was to 1, the closer the model was to the experimental data. The association coefficient of the pseudo-second-order model was 0.9978, and the theoretical values of \(q_e\) were similar to those of the experimental data, a process coherent with the pseudo-second-order equations. These results showed that the adsorption performance of the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles was consistent with the pseudo-second-order model.

3.3. Adsorption isotherms of MB onto Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles

The experimental data were analyzed separately using the Freundlich, Langmuir, and Temkin models to study the effect of different initial MB concentration on the adsorption of the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles [25].

The Langmuir model assumed the monolayer adsorption, where all the points had the same energy in a homogeneous adsorption plane, yielding an adsorption effect in the gap. The expression for the Langmuir model
model is as follows [26]:

\[ q_e = \frac{q_{max} K_I C_e}{1 + K_I C_e} \]  

where \( q_{max} \) (mg g\(^{-1}\)) is the maximum adsorption capacity of magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles for MB, \( K_I \) (L·mg\(^{-1}\)) is the rate constant of the Langmuir model and \( C_e \) is the equilibrium concentration.
On the other hand, the Freundlich adsorption isothermal assumed an uneven adsorption at the surface, the expression for the Freundlich model is equation (7) [27].

\[ q_e = \frac{q_{\text{max}} K_F C_e}{1 + K_F C_e} \]

where \( K_F \) (L·mg\(^{-1}\)) is the rate constant of the Freundlich model and \( C_e \) is the equilibrium concentration.

The Temkin adsorption isothermal assumed chemisorption, and the expression for the Temkin model is equation (8) [28].

\[ q_e = B \ln(A_T C_e) \]

where \( A_T \) (L·mg\(^{-1}\)) is the rate constant of the Temkin model, and \( C_e \) is the equilibrium concentration.

The results collected from the experiment were described by \( C_e / q_e \), and three hypothetical models were presented in the graph (figure 7). Among the three models, the Temkin model agreed best with the group of data; all points were well distributed on the Temkin model. It was observed from the graph that the Temkin model best matched the MB adsorption process of the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles. The parameters for the three kinetic models are listed in table 2. The association coefficient \( R^2 \) of the Temkin model was 0.9984, while the association coefficients of the other two models were 0.9641 and 0.9394, respectively. The closer the association coefficient of the model was to 1, the better the model agreed with the experimental data and findings of the adsorption mechanism. Based on the assumption of the Temkin model, the adsorption mechanism of the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles was a multi-molecular layer adsorption.

### 3.4. Influence of pH and removal rate

The pH value also affected the adsorption capacity of MB by the magnetic Co\(_{0.4}\)Cu\(_{0.2}\)Zn\(_{0.4}\)Fe\(_2\)O\(_4\) nanoparticles, influencing not only the nature of the adsorbents, but also the form of pollutants in the wastewater. To evaluate the pH value that is required to achieve the best adsorption capacity, pH values were tested from 1 to 11. It was observed that changing the pH value resulted to a corresponding change in the adsorption capacity (figure 8(A)). In addition, a gradual increase in the pH value from 1 to 5, substantially increased the adsorption capacity.
increase in the pH from 5 to 11, gradually increased the adsorption capacity, but the adsorption capacity decreased slightly as the pH was changed from 8 to 9. A low pH was beneficial to the protonation of the adsorbent surface, producing more positive charge sites. At high pH, the surface of the adsorbent became negatively charged, enhancing a strong repulsive force, which decreased the adsorption capacity [29].

Recyclability was an important index for product inspection, analyzing whether the magnetic Co$_{x}$Cu$_{y}$Zn$_{(1-x-y)}$Fe$_2$O$_4$ nanoparticles had production value [30]. In figure 8(B), the removal rate was approximately 100% in the first cycle; a significant decline occurred after 3 cycles, but was approximately 80% after 7 cycles. There was no significant change in the removal rate during the 3 to 7 cycles, and the removal rate was approximately 78%. The experimental data showed that the development of magnetic Co$_{x}$Cu$_{y}$Zn$_{(1-x-y)}$Fe$_2$O$_4$ nanoparticles is substantially important.

4. Conclusions

(1) Magnetic cobalt-cuprum-zinc ferrites were prepared using an anhydrous ethanol combustion method, which were formed at 400 °C. The Magnetic cobalt-cuprum-zinc ferrites nanoparticles had an average diameter of 20 nm and a magnetic saturation intensity of approximately 52 emu·g$^{-1}$.

(2) The pseudo-second-order kinetics and Temkin models described the adsorption process and the adsorption relationship between MB and the magnetic Co$_{0.4}$Cu$_{0.2}$Zn$_{0.4}$Fe$_2$O$_4$ nanoparticles, which demonstrated that the adsorption process of MB onto the nanoparticles was a multi-molecular layer chemisorption mechanism.

(3) When the pH exceeded 5, the adsorption capacity of the magnetic Co$_{0.4}$Cu$_{0.2}$Zn$_{0.4}$Fe$_2$O$_4$ nanoparticles for MB maintained a high value. In addition, the magnetic Co$_{0.4}$Cu$_{0.2}$Zn$_{0.4}$Fe$_2$O$_4$ nanoparticles demonstrated a higher recycling capacity, and the removal rate for MB was approximately 80% after seven cycles.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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