Combustion process for magnetic copper–cobalt ferrite and its Congo red adsorption property

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Keywords: Cu0.5Co0.5Fe2O4 nanoparticles, Congo red, adsorption mechanism, copper–cobalt ferrites

Abstract

A rapid combustion process was introduced for the preparation of magnetic copper–cobalt ferrite, which was characterized using X-ray diffraction (XRD), vibrating sample magnetometer (VSM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and Brunauer–Emmett–Teller techniques (BET). The prepared magnetic copper–cobalt ferrite showed a large specific surface area (104.6 cm² g⁻¹) and nanoscale particle size (55.4 nm), with a saturation magnetization of 19.6 Am² kg⁻¹. The nanoparticles were used to adsorb and remove Congo red (CR) from dyestuff wastewater, and the adsorption mechanism was revealed. Compared with the pseudo-first-order kinetic model and intraparticle diffusion kinetic model, the pseudo-second-order kinetic model was better at describing the CR adsorption process on the Cu0.5Co0.5Fe2O4 nanoparticles, while the Temkin isotherm best fitted the CR adherence on the Cu0.5Co0.5Fe2O4 nanoparticles. All data suggested that the adsorption of CR on the Cu0.5Co0.5Fe2O4 nanoparticles followed the monomultilayer hybrid chemisorption mechanism. In addition, as the pH increased from 2 to 10, the adsorption capacity of the Cu0.5Co0.5Fe2O4 nanoparticles for CR decreased, indicating that an acidic environment was beneficial for the adsorption of CR on the Cu0.5Co0.5Fe2O4 nanoparticles. When recycling the Cu0.5Co0.5Fe2O4 nanoparticles after adsorbing CR, the relative adsorption rate was still 62.5% of the initial adsorption capacity after five cycles, revealing the reusability and promising applicability of Cu0.5Co0.5Fe2O4 nanoparticles in sewage treatment.

1. Introduction

It is believed that water pollution will be a global threat over the next decade [1]. One primary source of water contamination is dyes. If dyestuff wastewater is directly released into the environment without treatment, it can result in many environmental problems and health issues [2]. In particular, diazo dyes are more toxic because of their difficult degradation and possible secondary pollution after the first treatment [3]. Congo red (CR), which is a diazo dye, possesses an intricate aromatic structure [4] and is a potent mutagen that results in diseases of the blood, nerves, respiratory system, intestines, stomach, and reproductive system [5]. Therefore, it is crucial to treat CR wastewater before it is discharged into the environment.

Many approaches have been applied to remove dyes from wastewater, including physicochemical methods, biological methods, and comprehensive treatments that combine physicochemical and biological methods [6]. Among these, adsorption is considered one of the most effective methods because of its low cost and lack of secondary pollution [7].

The adsorbent is a crucial factor in the adsorption process. In recent years, nanomaterials have been used for adsorption because of their large specific surface areas and adsorption capacities. However, non-magnetic nanomaterials have a significant weakness; they can be difficult to separate because of their nanosize [8]. Thus,
magnetic nanomaterials have been applied in the adsorption process because they can be separated under an applied magnetic field [9]. Therefore, research on using magnetic nanomaterials for adsorption has become a hotspot.

As one of the most important magnetic iron-oxide-based nanomaterials, magnetic Cu0.5Co0.5Fe2O4 nanoparticles have been prepared using various methods [10, 11]. Among these, the rapid combustion process is favored because of its short previous preparation process, short production cycle, uniform product particle size, and the absence of a protective gas requirement [12–16].

Therefore, in this study, magnetic Cu0.5Co0.5Fe2O4 nanoparticles were prepared by a rapid combustion method and were characterized and employed to adsorb CR. The adsorption behavior of CR on the Cu0.5Co0.5Fe2O4 nanoparticles was also examined.

2. Experimental details

2.1. Preparation and characteristic of Cu0.5Co0.5Fe2O4 nanoparticles

A rapid combustion process was used in the preparation experiments for magnetic Cu0.5Co0.5Fe2O4 nanoparticles. Typically, 6.92 g of ferric nitrate ninhydrine (Fe(NO3)3·9H2O, 98.5%), 1.03 g of copper nitrate trihydrate (Cu(NO3)2·3H2O, 99.0%), and 1.25 g of cobalt nitrate hexahydrate (Co(NO3)2·6H2O, 98.5%) were placed into 20 ml of anhydrous ethanol. After their dissolution, the dissolved solution was burned in a crucible. The solid obtained after burning was calcined at 400 °C for 2 h in a programmed temperature-controlled furnace. The calcination process was complete when the furnace naturally cooled below 50 °C. The obtained product was ground to produce the magnetic Cu0.5Co0.5Fe2O4 nanoparticles. The phase identification and particle size characteristics of the product were determined using XRD. The composition and morphology of the Cu0.5Co0.5Fe2O4 nanomaterial were analyzed using EDS and SEM, respectively. The magnetic properties were detected using a vibrating sample magnetometer (VSM). The specific surface area and pore size distribution of the Cu0.5Co0.5Fe2O4 nanomaterial were measured using the BET method.

2.2. CR adsorption experiments on Cu0.5Co0.5Fe2O4 nanoparticles

All of the adsorption experiments of kinetic study were performed in centrifuge tubes (50 ml), with 20 ml of the CR solution and 50 mg of the Cu0.5Co0.5Fe2O4 nanoparticles used in every experiment. CR solutions of 100–400 mg l⁻¹ with pH values of 2–10 were prepared for the adsorption experiments, and the pH values were changed using 0.01 M HCl and NaOH solutions. After the adsorption experiments were concluded, the centrifuge tubes were removed at specific intervals, and the Cu0.5Co0.5Fe2O4 nanoparticles were separated from the solutions using an external magnetic field. Adsorption equilibrium experiments were carried out in the same way, and the centrifuge tubes were shaken at room temperature for 24 h. A UV spectrophotometer (UV2550) was employed to measure the residual concentration of the CR solution, and the adsorption capacity and equilibrium were calculated according to the following equation (1) [17–19]:

\[
q = \frac{V(C_0 - C)}{m}
\]

where \(q\) is the amount of dye adsorbed at a specific time in mg g⁻¹, \(V\) is the CR solution volume in L, \(C_0\) is the initial CR concentration in mg l⁻¹, \(C\) is the CR concentration at a specific time in mg l⁻¹, and \(m\) is the mass of Cu0.5Co0.5Fe2O4 nanoparticles in g.

3. Results and discussion

3.1. Characterization of magnetic Cu0.5Co0.5Fe2O4 nanoparticles

Figure 1 shows the characteristics of the magnetic Cu0.5Co0.5Fe2O4 nanoparticles. The SEM image (figure 1(A)) showed that the magnetic Cu0.5Co0.5Fe2O4 nanomaterial had a spherical structure, and the particle sizes were homogeneous. A particle size distribution histogram was obtained using Nanomeasure software and fitted using a log-normal distribution function (figure 1(B)) [20, 21]. The total number (N) of particles was 109, and the mean size was 55.4 nm, the polydispersity parameter (\(\sigma\)) was 7.3527 nm. These results demonstrated the successful preparation of nanoscale particles with uniform morphology and dispersity. Figure 1(C) shows an EDS spectrogram. The element contents of Cu, Co, Fe, and O in the magnetic Cu0.5Co0.5Fe2O4 nanoparticles were 11.46%, 11.41%, 38.54%, and 38.59%, respectively. The elemental composition and percentages suggested the successful preparation of magnetic Cu0.5Co0.5Fe2O4 nanoparticles. Figure 1(D) shows the XRD pattern, where the diffraction peaks were indexed to the standard PDF cards of CuFe2O4 (ICPDS. No. 34–0425) and CoFe2O4 (ICPDS. No. 22–1086) [10], revealing the formation of Cu0.5Co0.5Fe2O4 nanoparticles. The hysteresis loops of the Cu0.5Co0.5Fe2O4 nanoparticles are shown in figure 1(E). The results indicated that the saturation
magnetization of the Cu0.5Co0.5Fe2O4 nanoparticles was up to 19.6 Am2 kg−1, and they had low coercivity and weak hysteresis, showing classical soft magnetism [22–24]. This made it easy for them to disperse during dye adsorption, making them easy to remove and recycle by magnetic separation after adsorption. The pore size distribution and N2 adsorption–desorption isotherms of the Cu0.5Co0.5Fe2O4 nanoparticles are shown in figure 1(F). The N2 adsorption–desorption isotherms for the Cu0.5Co0.5Fe2O4 nanoparticles revealed typical type IV curves, suggesting their porous structure. In addition, they had a specific surface area of 104.6 cm2 g−1. Their pore radii were mainly distributed from 5 nm to 12 nm, which resulted in a large adsorption capacity for CR.

3.2. CR adsorption on Cu0.5Co0.5Fe2O4 nanoparticles
3.2.1. Adsorption kinetics
The effect of the initial CR concentration on the adsorption capacity of the magnetic Cu0.5Co0.5Fe2O4 nanoparticles for the removal of CR is shown in figure 2. The curves in figure 2 reveal that the adsorption capacity increased with the initial CR concentration. For all initial CR concentration, the initial adsorption rate
was very fast because there were numerous available adsorption sites on the surface of the Cu0.5Co0.5Fe2O4 nanoparticles. In addition, the adsorption capacity of the Cu0.5Co0.5Fe2O4 nanoparticles reached a maximum after approximately 30 min, and the adsorption reached equilibrium. Moreover, although the adsorption increased with the initial concentration, the increment decreased, possibly because the adsorption sites of the Cu0.5Co0.5Fe2O4 nanoparticles were occupied due to the high concentration of CR molecules.

To reveal the effect of the contact time on the CR adsorption of the Cu0.5Co0.5Fe2O4 nanoparticles, three models (the pseudo-first-order kinetic model, pseudo-second-order kinetic model, and intraparticle diffusion kinetic model) \[10, 25\] were employed to fit the adsorption experimental data. The formulas for these models are listed in table 1 \[26, 27\].

The adsorption fitting curves of the pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models for CR on Cu0.5Co0.5Fe2O4 nanoparticles with various initial CR concentrations are shown in figure 3, and the related fitting parameters are presented in table 1. It was not hard to see that the pseudo-second-order kinetic model best coincided with all the adsorption data for CR solutions with concentrations of 100–400 mg L\(^{-1}\). The fitting variances (R\(^2\)) of the pseudo-second-order kinetic model were relatively the largest (R\(^2\) > 0.98). The smallest variance was also close to 0.99, suggesting that the pseudo-second-order kinetic model best fit the adsorption of CR on the Cu0.5Co0.5Fe2O4 nanoparticles compared with the other models. The results revealed that the adsorption of CR on the Cu0.5Co0.5Fe2O4 nanoparticles followed the chemisorption mechanism, which indicated that electron sharing or electron transfer occurred between the Cu0.5Co0.5Fe2O4 nanoparticles and CR. In other words, the valence force resulted in the attraction of Cu0.5Co0.5Fe2O4 nanoparticles and CR \[24, 28\]. The linear relationships of the pseudo-second-order kinetic model for CR adsorption with various initial CR concentrations on the Cu0.5Co0.5Fe2O4 nanoparticles are displayed in figure 4, revealing good linear relations.

**Table 1.** Adsorption kinetic parameters of magnetic Cu0.5Co0.5Fe2O4 nanoparticles for adsorption of CR at room temperature.

| Kinetic model                  | Equation | Parameter | Initial CR concentration (mg L\(^{-1}\)) |
|-------------------------------|----------|-----------|-----------------------------------------|
|                               |          |           | 100          | 200         | 300         | 400          |
| Pseudo-first-order model      | ln(q\(_e\) - q\(_t\)) = ln(q\(_e\)) - k\(_1\)t | k\(_1\) | 0.1764 | 0.2038 | 0.2092 | 0.1393 |
|                               | ln(q\(_e\)) | q\(_e\) | 37.2676 | 81.8445 | 121.1471 | 145.2731 |
|                               | R\(^2\)   | R\(^2\)   | 0.7354 | 0.7727 | 0.7103 | 0.7927 |
| Pseudo-second-order model     | q\(_t\) = \(\frac{q\(_e\)^2k_2t}{1 + q\(_e\)k_2t}\) | k\(_2\) | 0.0122 | 0.0077 | 0.0054 | 0.0020 |
|                               |           | q\(_e\) | 38.6896 | 84.2355 | 124.6009 | 152.8471 |
|                               | R\(^2\)   | R\(^2\)   | 0.9899 | 0.9904 | 0.9903 | 0.9897 |
| Intraparticle diffusion model  | q\(_t\) = I + k\(_t\)t\(^{1/2}\) | k\(_1\) | 0.5118 | 0.8328 | 1.1929 | 2.8457 |
|                               |           | I\(_c\) | 32.5598 | 74.2516 | 110.3381 | 118.3335 |
|                               | R\(^2\)   | R\(^2\)   | 0.7861 | 0.7446 | 0.7558 | 0.7941 |
Figure 3. The fitting curves of pseudo-first-order, pseudo-second-order, and the intraparticle diffusion kinetic models for CR adsorption with initial concentration of 100 mg l$^{-1}$ (A), 200 mg l$^{-1}$ (B), 300 mg l$^{-1}$ (C), and 400 mg l$^{-1}$ (D) on Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles.

Figure 4. Linear relationships of the pseudo-second-order kinetic model for the CR adsorption with various initial CR concentrations on Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles.
3.2.2. Adsorption isotherms

The adsorption capacities of the Cu0.5Co0.5Fe2O4 nanoparticles under various CR concentrations were estimated by fitting the Langmuir, Freundlich, and Temkin models [29, 30], and the equations of these three models are listed in Table 2 [31, 32].

The fitting curves of the three adsorption isotherms for CR on the Cu0.5Co0.5Fe2O4 nanoparticles at room temperature are shown in Figure 5, and the related parameters are listed in Table 2. The Temkin isotherm model had the largest variance ($R^2 = 0.9991$) of the three, while the errors of some of the experimental data for the Langmuir and Freundlich models were relatively larger. Therefore, the Temkin isotherm best fitted the CR adsorption equilibrium data on the Cu0.5Co0.5Fe2O4 nanoparticles. Based on the assumptions of the three models, the CR adsorption state on the Cu0.5Co0.5Fe2O4 nanoparticles was not monolayer adsorption or multilayer adsorption. The adsorption of CR on the magnetic Cu0.5Co0.5Fe2O4 nanoparticles was the result of a hybrid mono-multilayer mechanism.

![Figure 5. Fitting curves of Langmuir, Freundlich and Temkin isotherm models for CR on Cu0.5Co0.5Fe2O4 nanoparticles at room temperature.](image)

### Table 2. Adsorption isotherm parameters of CR on magnetic Cu0.5Co0.5Fe2O4 nanoparticles at room temperature.

| Isotherms model | Equation | $R^2$ | Parameter | Parameter value |
|-----------------|----------|-------|-----------|-----------------|
| Langmuir        | $\frac{C_q}{q_e} = \frac{1}{q_{\text{max}}K_L} + \frac{C_e}{q_{\text{max}}}$ | 0.9803 | $K_L$     | 0.4849          |
| Freundlich      | $q_e = K_F C_e^{1/n}$ | 0.9794 | $1/n$     | 0.3104          |
| Temkin          | $q_e = \frac{RT}{B} \ln(A_T C_e)$ | 0.9991 | $A_T$     | 7.0244          |

3.2.3. Influence of pH on adsorption and recycling of Cu0.5Co0.5Fe2O4 nanoparticles

Figure 6 shows the influence of the pH on the CR adsorption capacity of the Cu0.5Co0.5Fe2O4 nanoparticles at room temperature, along with the adsorption recycling of the Cu0.5Co0.5Fe2O4 nanoparticles for CR removal. With an increase in pH, the CR adsorption capacity of the Cu0.5Co0.5Fe2O4 nanoparticles decreased (as shown in Figure 6(A)). This occurred because, for ferrite nanomaterials, with an increase in pH, the transfer of protons ($H^+$) makes the surface of the nanomaterials negatively charged and the zeta potential changes from positive to negative [33]. However, the anionic dye CR has lone electron pairs and amine functional groups and is negatively charged [28]. Thus, the electrostatic repulsion between the CR and Cu0.5Co0.5Fe2O4 nanoparticles caused a reduction in the adsorption capacity. Therefore, an acidic environment promoted the adsorption of CR onto the Cu0.5Co0.5Fe2O4 nanoparticles. Moreover, the CR adsorption capacity of the Cu0.5Co0.5Fe2O4 nanoparticles could be controlled by adjusting the pH value. In other words, changing the pH value could alter the adsorption...
capacity of the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles for CR. Thus, we could realize the desorption of CR on the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles by adjusting the pH value. From figure 6 (B), it can be seen that after recycling five times, the relative adsorption rate was still 62.5% of the initial adsorption capacity. Thus, the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles exhibited a good recycling capacity, suggesting potential applications in sewage treatment areas.

4. Conclusions

(1) Magnetic Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles were successfully prepared via a rapid combustion process. These magnetic Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles had a particle size of approximately 55.4 nm, saturation magnetization of 19.6 Am$^2$ kg$^{-1}$, and specific surface area of 104.6 cm$^2$ g$^{-1}$.

(2) The magnetic Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles exhibited a good adsorption capacity for the removal of CR. The Temkin isotherm and pseudo-second-order kinetic model best fitted the adsorption state of CR on the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles, suggesting that the adsorption was a result of a mono-multilayer hybrid chemisorption mechanism.

(3) An acidic environment was beneficial for the adsorption of CR on the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles, and the adsorption capacity was greatly affected by the pH value. After five cycles, the relative adsorption rate was still 62.5% of the initial adsorption capacity of the Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticles for CR. Furthermore, Cu$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ showed promising applicability to sewage treatment.

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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