Adsorption performance of reactive red 2BF onto magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles

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Abstract

The facile solution combustion and gel calcination process was applied to prepare magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles, and the characterization of the product was analyzed by SEM, TEM, XRD, and VSM techniques. The magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles prepared with absolute alcohol volume of 15 ml at 600 °C were expressed with the average particle size of approximately 60 nm and the saturation magnetization of 39.2 emu g$^{-1}$. Where after, Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles were applied to remove reactive red 2BF (RR-2BF), and at room temperature, the adsorbance of RR-2BF onto them was examined. The results demonstrated that pseudo-second-order kinetics could well express the adsorption process, and Temkin isotherm conformed to experimental data. The adsorption capacity of the sample could reach 130 mg g$^{-1}$, which was greatly affected by pH. The acidic environment was conducive to adsorption while the alkaline environment was conducive to desorption. Moreover, the material had excellent cycling performance.

1. Introduction

Dye plays an extremely important role in our daily life, and our life is becoming increasingly colorful because of the existence of dyes [1, 2]. With the expansion of aesthetic demand and the progress of economy and technology, various synthetic dyes emerge in endlessly. While people enjoy the colorful life brought by dyes, there are also a lot of dye wastewater on the earth. Dye wastewater contains a lot of harmful substances, such as potentially toxic metals, organic matter and so on. These refractory molecules and functional groups cause serious harm to human body, water and soil [3]. The random discharge of synthetic dyes will also have a serious impact on the growth and reproduction of microorganisms in the water body, which may destroy the self-purification ability of water and cause eutrophication [4, 5]. Therefore, the effective treatment of dye wastewater is very important to prevent environmental pollution. Dye wastewater treatment technology is constantly developing and updating, such as photocatalytic degradation, ion exchange technology, biodegradation technology, adsorption method, etc [6, 7]. Compared with other technologies, the adsorption process has low cost, simple and efficient operation, and low requirements on equipment, thus it has attracted the attention of many researchers [8, 9].

Small size nanomaterials, especially nanoparticles, play a very unique role in adsorption. Due to their small size, only in the range of 1–100 nm, these nanomaterials exhibit different properties from other macroscopic materials, such as surface effect and small size effect [10, 11]. The unique properties of these nanoparticles enable them to absorb more pollutants with the large specific surface areas and the plentiful adsorption sites for dyes during the adsorption process. In particular, magnetic iron oxide nanoparticles have good magnetic properties. They not only have large specific surface area of adsorption, but also have excellent magnetic recovery and reuse performance, which further reduces the harm of the adsorbed particles to the environment [12, 13]. In fact, the mechanism for almost all ferric oxide-based nanomaterials is the improvement of Fe$_3$O$_4$ nanomaterials, because Fe$^{2+}$ in Fe$_3$O$_4$ is facile to be oxidized to Fe$^{3+}$, Fe$_3$O$_4$ turns onto Fe$_2$O$_3$, and the magnetism sharply decreases. To avoid the oxidization of Fe$^{2+}$, Fe$^{2+}$ in Fe$_3$O$_4$ is replaced by 2-valent metal ions, which results that the magnetism...
of nanomaterials maintains a large value. Among them, low price Zn$^{2+}$ is a better selection, however, the magnetism of ZnFe$_2$O$_4$ nanomaterials is also small, then Cu$^{2+}$ is selected to be added into the system. Thus, Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles, as one of the ferric oxide-based nanoparticle materials, is an important nanostructured dye adsorption material. There are many methods to prepare Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles, such as the simultaneously reduction-reoxidation technique [14], solid-state reaction [15], microwave combustion method [16], and so on. However, these methods generally require higher experimental equipment and the experimental process is complex. Therefore, a simple and feasible combustion method is proposed in this paper to prepare the powder material. Solution combustion and gel calcination approach adopted unique solution combustion and calcination process is a convenient technology. The entire program with short pretreatment time is operational and no other dispersants are required. Meanwhile, the magnetism was readily adjusted by changing the calcination temperature and solvent volume [17].

Reactive red 2BF (RR-2BF), whose structure was displayed as scheme 1, is a red pigment type. Due to its strong ability to absorb electrons of B and F elements, RR-2BF can make the colored cloth more crimson, therefore it is widely applied in industrials.

In this paper, magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles were successfully prepared via the novel solution combustion and gel calcination process, their composition, structure, and properties were disclosed. In the process mechanism of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles for dyes removal was investigated with RR-2BF as model.
2. Experimental

2.1. Preparation and characterization of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles

Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles were prepared via the solution combustion and gel calcination process using absolute alcohol as solvent \[18\]. The zinc nitrate, cupric nitrate, and iron nitrate were employed to prepare Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles, according to the molar ratios of zinc, copper and iron of 3:7:20. 2.48 g Zn(NO_3)\_2\cdot6H_2O, 2.01 g Cu(NO_3)\_2\cdot3H_2O, 3.36 g Fe(NO_3)\_3\cdot9H_2O, and 15 ml absolute alcohol were added in beaker at room temperature, the mixture was continuously stirred for 1 h. Subsequently, the mixture solution was placed into a crucible and ignited. When the fire caused by alcohol was natural out, the intermediate was formed and calcined in controlled temperature furnace at 600 °C for two hours to form nanopowders.

The composition analysis of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles were executed by x-ray diffractometer (XRD); the morphology was surveyed with the scanning electron microscopy (SEM) and the transmission electron microscopy (TEM); the magnetic behavior was investigated on vibrating sample magnetometer (VSM).

2.2. Adsorption of RR-2BF onto Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles

The concentrations of RR-2BF solutions were determined by visible spectroscopy (UV-2550) with the absorbances at 240 nm. The absorbance of RR-2BF solution versus the concentration was revealed in figure 1.
and the relationship and the correlation coefficient were set in figure 1. The adsorption kinetics experiments were completed by constantly stirring RR-2BF solutions of 20 ml with concentrations of 100, 200, 300, 400 mg l$^{-1}$ and 0.05 g Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles for different periods of time. At room temperature, adsorption isotherm experiments at various RR-2BF concentrations of 100–600 mg l$^{-1}$ for a sufficient length of time were designed. The effects of pH on RR-2BF adsorption of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles and the zeta potential of the nanoparticles were measured at pH of 1–11, the pH value was changed by 1 M HCl or NaOH [7]. Magnetic nanoparticles of the same quality were applied for adsorbing RR-2BF solution of the same concentration but different pH values. The residual RR-2BF concentration in the solution was determined by separating the Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles with an external magnetic field. The experiments were repeated three times, and according to the relationship of the average absorbance and the concentration of RR-2BF, the adsorbance of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles might be calculated as the following formula [19].

$$q = \frac{m_{RR-2BF}(A_{RR-2BF} - A_{mag})}{m_{mag}A_{RR-2BF}}$$

(1)

Wherein $q$ was the RR-2BF amount adsorbed by Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles (mg g$^{-1}$), $m_{RR-2BF}$ was the initial weight of RR-2BF (mg), $m_{mag}$ was Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles weight (g), $A_{RR-2BF}$ and $A_{mag}$ were the UV absorbance values of blank RR-2BF solution and RR-2BF solution after adsorption, respectively.

3. Results and discussion

3.1. Characterization of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles

The characterization of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles prepared with 15 ml absolute alcohol at 600 °C was displayed in figure 2. The SEM morphology of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles was evinced in figure 2(a), obviously, their particle size was about 60 nm. The TEM image of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles was revealed in figure 2(b), obviously, Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles had a polycrystalline structure, their average particle size was consistent with the conclusion of SEM image shown in figure 2(a), also around 60 nm. According to figure 2, the particle size was performed statistical analysis, and the size histogram was drawn as figure 2(c). The data accorded with Gauss function as equation (2), and the variance for the Gauss function was up to 0.9928, which suggested that the particle size distribution of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles prepared with 15 ml absolute alcohol at 600 °C conformed to Gaussian distribution.

$$\text{Percent} = 11.30 + 89.60 \times e^{-\frac{(d-61.53)^2}{953.64}}$$

(2)

The XRD pattern for Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles was displayed in figure 2(d), and the peaks were matched well with (220), (311), (222), (400), (422), (511), (440), (620), and (622) planes, which correspond to single phase spinel Zn-Cu ferrite (ICPDS No.51-0386) crystal structure. Figure 2(e) revealed the hysteresis loops of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles with the soft magnetization behavior and hysteresis coercivity ($Hc$), the Ms and $Hc$ were 39.2 emu g$^{-1}$ and 93.9 Oe, respectively.
3.2. Adsorption kinetics of RR-2BF onto Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles

Exposing the rate change of RR-2BF removal from the solution was of great significance in the whole adsorption process. To reveal the adsorption mechanism for RR-2BF from solution onto Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles, the pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetics models were applied to analyze behavior of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles for RR-2BF adsorption [18].

Three formulas for the kinetics models were exhibited as equations (3)–(5) [20–22].

\[
q_t = q_e (1 - e^{-k_1 t}) 
\]  

(3)

\[
q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} 
\]  

(4)

\[
q_t = q_i + k_i t^{1/2} 
\]  

(5)

Wherein \(q_t\) and \(q_e\) (mg·g\(^{-1}\)) were the adsorbances of RR-2BF (mg·g\(^{-1}\)) at a given time and equilibrium; \(k_1\) (min\(^{-1}\)), \(k_2\) (g·mg\(^{-1}\)·min\(^{-1}\)), and \(k_i\) (mg·g\(^{-1}\)·min\(^{-1}\)) were the kinetics constants for three models; \(x_i\) was the boundary layer thickness for intraparticle diffusion model.

The adsorbances \(q_t\) versus time \(t\) under different initial RR-2BF solution concentrations were exhibited in figure 3. It could be seen that the adsorbance of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles obtained after calcination at 600 °C increased rapidly at the initial contact stage. As time went on, the adsorption rate increased until it reached equilibrium. Figure 3 revealed that the initial RR-2BF concentration had significant influence on the adsorption process. At a lower RR-2BF concentration, the adsorption reached equilibrium at 30 min. However, at a higher concentration of RR-2BF, it took about 60 min for the adsorption to keep equilibrium. This phenomenon revealed that the physical adsorption was present throughout the process of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles for RR-2BF removal. Moreover, when the concentration of RR-2BF likewise was 100 mg l\(^{-1}\), the adsorbance of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles for RR-2BF achieved 38.4 mg g\(^{-1}\), which was as about 2 times as that of RR-2BF onto Ni_{0.5}Zn_{0.5}Fe_{2}O_{4} nanoparticles [23]. However, the preparation process of Zn_{0.3}Cu_{0.7}Fe_{2}O_{4} nanoparticles made great progress compared with that of Fe_{2}O_{4} nanoparticles, the protection of inert gases could be removed.

Figure 4. Fits of the pseudo-first-order kinetics model, pseudo-second-order kinetics model and intraparticle diffusion kinetics model at initial RR-2BF concentrations of 100 mg l\(^{-1}\) (A), 200 mg l\(^{-1}\) (B), 300 mg l\(^{-1}\) (C), and 400 mg l\(^{-1}\) (D).
in preparation of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles. All above results suggested that Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles were significant in removal of dye adsorption field.

When RR-2BF concentration ranged from 100 to 400 mg l$^{-1}$, the fitting curve of the adsorption kinetic model for Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles to RR-2BF was revealed in figure 4, the corresponding kinetics parameters were calculated and listed in table 1. By comparing with each other, it could not be hard to find that the pseudo-second-order kinetics well matched the kinetics data, and their correlation coefficients ($R^2$) were the largest. However, the $R^2$ values of other models was lower, which indicated that the fitting effect was not as good as that of the pseudo-second-order kinetics model. Thus, the pseudo-second-order kinetics could well describe the adsorption process of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles. The relations of $t/q_t$ versus $t$ were displayed in figure 5, those presented very good linear relations, which also revealed that the adsorption process could be traced by the pseudo-second-order kinetics.

### Table 1. Fitted kinetics parameters for adsorption of RR-2BF in aqueous solution onto magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles at room temperature.

| Kinetics model                      | RR-2BF concentration (mg l$^{-1}$) |
|------------------------------------|------------------------------------|
|                                    | 100  | 200  | 300  | 400  |
| Pseudo-first-order model           |      |      |      |      |
| $q_e$ (mg g$^{-1}$)                | 36.9485 | 70.7391 | 93.4651 | 108.0598 |
| $k_1$                              | 0.1662 | 0.1538 | 0.1494 | 0.1734 |
| Adj. R-Square                      | 0.8658 | 0.8924 | 0.7190 | 0.6776 |
| Pseudo-second-order model          |      |      |      |      |
| $q_e$ (mg g$^{-1}$)                | 38.3763 | 73.6537 | 98.0184 | 112.4003 |
| $k_2$                              | 0.0115 | 0.0052 | 0.0035 | 0.0040 |
| Adj. R-Square                      | 0.9889 | 0.9855 | 0.9835 | 0.9838 |
| Intraparticle diffusion model      |      |      |      |      |
| $x_i$                              | 32.1729 | 60.4027 | 77.6455 | 93.6528 |
| $k_i$                              | 0.5055 | 1.0709 | 1.6922 | 1.5711 |
| Adj. R-Square                      | 0.6563 | 0.6365 | 0.7905 | 0.7912 |

**Figure 5.** Linear fits of the pseudo-second-order kinetics model at initial RR-2BF concentrations of 100–400 mg l$^{-1}$.

3.3. Adsorption isotherm of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles

Adsorption isotherm could reflect the interrelationship of the adsorbent and the adsorbate. In this paper, three isotherm models, including Langmuir, Freundlich and Temkin [18], had been applied to disclose mechanism of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles for RR-2BF adsorption. The following three equations were respectively expressed as three isotherm models [24–26].

\[
q_e = \frac{q_{\text{max}} K_t C_v}{1 + K_t C_v} \quad (6)
\]

\[
q_e = K_F C_v^{\frac{1}{n}} \quad (7)
\]
Wherein $q_e$ was the equilibrium adsorbance of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles (mg·g$^{-1}$); $C_e$ was RR-2BF concentration at the time of equilibrium (mg·l$^{-1}$); $K_L$ (l·mg$^{-1}$), $K_T$ (mg·l$^{-1}$·mg$^{-1}$/$g$), and $K_T$ (l·g$^{-1}$) were the constants for three models; $q_{\text{max}}$ (mg·g$^{-1}$) was $q_t$ (mg·g$^{-1}$) for the complete adsorption; 1/$n$ and $B_T$ were the constants for Freundlich model and Temkin model, respectively [18].

The adsorption isotherms of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles were examined with various initial RR-2BF concentration in 100–600 mg·l$^{-1}$. Figure 6 exhibited the adsorption data at equilibrium had been analyzed through isotherms models, and the parameters for them were displayed in Table 2. Compared with the correlation coefficients of Freundlich and Langmuir model, Temkin model yielded the largest variance ($R^2 = 0.9981$). On the basis of the theory of Temkin model, the performance of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles belonged to the monolayer-multilayer hybrid adsorption mechanism [18].

### 3.4. Effect of pH on adsorbance

To disclosed the pH influence of RR-2BF solution on the adsorption of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles, the adsorption with different pH values were executed at room temperature for 24 h at 200 mg·l$^{-1}$ of RR-2BF concentration [7]. The effect rules of pH on the adsorbance and the zeta potential of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles for RR-2BF were shown in figures 7(A) and (B). When pH was less than 3, the adsorbance of the material was large, and the adsorbance was basically kept at a high level under acidic environment. When the pH increased, the adsorbance of the nanomaterials decreased rapidly, and if the pH was greater than 9, the

$$q_t = B_T \ln(K_F C_e) \quad (8)$$

![Figure 6. The Langmuir, Freundlich, and Temkin adsorption isotherms of RR-2BF onto magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles at room temperature.](image)
adsorbance dropped below 10 mg g⁻¹. The causes of this phenomenon were that with the pH increases, the number of hydroxide ions in the solution increased, the ions and methylene blue on the material of the adsorption sites had competition effect. As in acidic environment, the competition was weak, resulting in the material to dye adsorption capacity was large, and when the solution gradually shifted towards the direction of alkaline solution, the attachment of hydroxide ions onto the material enhanced gradually, while the adsorption of dyes reduced. This phenomenon showed that by controlling pH value, RR-2BF on Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles could be desorbed. At the same time, the zeta potential of Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles decrease with the increase of pH as shown in Figure 7(B), that is, the surface charge of Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles also changed from positive charge to negative charge with the increase of pH. According to the structure of RR-2BF, RR-2BF showed negative charge, thus in the acidic environment, Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles with the positive surface charge facilely combined with RR-2BF, therefore, the adsorbance of RR-2BF onto Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles was larger in the acidic environment. Which agreed with the conclusion of the pH effect on the RR-2BF adsorption.

3.5. Recycle of Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles
The magnetic Zn₀.₃Cu₀.₇Fe₂O₄ nanoparticles could be recycled many times and maintain a high relative dye removal rate. As demonstration of figure 7(C), the relative removal rate of the materials to the dye gradually decreased but with a small extent with the increase of cycle times. As the number of cycles increased, the nanoparticles tended to agglomerate, and the adsorption sites would gradually collapse. There is also a loss in the amount of materials during recycling, resulting in the successive decrease of adsorbance. After 10 cycles, the materials could still maintain a relative removal rate of 80%, indicating that the materials had stable properties, excellent cycling performance, little impact on the environment, and had practical application value.
4. Conclusions

The solution combustion and gel calcination process with absolute alcohol as solvent was successfully applied to prepare magnetic Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles. By characterization, the average particle size of Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles was about 60 nm, and the saturation magnetization was 39.2 emu g$^{-1}$. The adsorption of RR-2BF onto Zn$_{0.3}$Cu$_{0.7}$Fe$_2$O$_4$ nanoparticles was monolayer-multilayer hybrid physical absorption mechanism. The pseudo-second-order kinetics and Temkin isotherm could well fit the adsorption process at room temperature. The adsorbance of nanomaterials, which reached 130 mg g$^{-1}$, was greatly affected by pH. The adsorption and desorption could be realized by adjusting pH, and the recycling effect of the material was excellent, after 10 cycles, the relative removal rate can remain above 80%.

Data availability statement

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

Conflicts of interest

The authors declare no conflict of interest.

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