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Adsorption performances and electrochemical characteristics of methyl blue onto magnesium-zinc ferrites

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Abstract

A novel and facile rapid combustion approach was developed for the controllable preparation of small size and easy recovery magnesium–zinc ferrites for methyl blue (MB) removal in dye solution. The effects of prepared criteria of x value, calcination temperature, and the amount of ethanol on the average grain sizes and magnetic property were reviewed. The characterization results displayed that Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles met the expectations of the experiment at the calcination temperature of 400 °C with absolute ethanol volume of 20 ml, and they were selected to remove MB. The adsorption process belonged to chemical adsorption on the basis of the pseudo-second-order model. The electrochemical characteristics of MB onto the prepared nanoparticles were analyzed by cyclic voltammetry (CV). The influences of pH and cycle times on the removal efficiency were investigated. When the pH went beyond 3, the removal efficiency of MB onto the magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles maintained above 99%, the maximum adsorption capacity was 318.18 mg g$^{-1}$. After seven cycles, the relative removal rate of MB remained 96% of the first one.

1. Introduction

With the development of human society, dyes were widely applied in textiles, paper, leather, and pharmaceutical industries, etc [1–3]. A large amount of dye wastewater was generated, which has caused serious environmental pollution problems and posed a serious threat to human health [4–6]. Therefore, there is an urgent need for economical and efficient dye wastewater treatment technology.

Up till now, for the removal of dyes, many effective methods such as advanced oxidation [7], microbial degradation [8], adsorption [9], photocatalytic oxidation degradation [10–12] have been probed. Among these methods, the adsorption method was considered to be the most promising technology for the treatment of dyes in wastewater on account of its inexpensive, high efficiency, good recovery, and facile manipulation [13–15]. With the development of dyes removal methods based on adsorption technology, and all sorts of dyes adsorption materials have been exploited, such as activated carbon [16], molecular sieve [17], activated alumina [18], zeolite [19], etc. However, these adsorbents had the difficult problem of regeneration [20, 21], which was easy to cause secondary pollution to the water system, and restricted their industrial development.

Over the last few years, nanotechnology has emerged as an important research area because of its massive applications and high application value. The application of nanomaterials is an indispensable part of nanotechnology. Nanomaterials have a large specific surface area, allowing them to have more active sites to adsorb pollutants in the water [22]. However, nonmagnetic nanomaterials were difficult to separate from the solutions [23]. Compared with various traditional adsorbents, magnetic nanomaterials had the advantages of small size, large specific surface area, superparamagnetic properties, good adsorption performance, and easy recovery [24, 25], and they could be separated from solutions under the action of a magnetic domain. Therefore, magnetic nanomaterials were employed for the removal of dyes. In many studies, magnetic nanomaterials were mainly prepared by hydrothermal [26], co-precipitation [27], sol-gel [28], and microemulsion [29] methods. However, many of these approaches were complicated, had long production cycles, and the purity of the
products could not be easily controlled. To overcome these demerits, the rapid combustion approach was
developed with advantages of short processing time, short preparation cycle, and low cost. And it can achieve
the purpose of controlling nanoparticles size by controlling the dosage of the solvent and the calcination mechanism
[30]. It is a relatively simple and ideal method for preparing magnetic nanomaterials.

Hence, in this work, a one-step rapid combustion approach was applied to prepare magnetic magnesium-
zinc ferrites nanomaterials with nitrates and absolute ethanol as ingredients and solvents. By investigating the
amount of ethanol and the calcination temperature, the optimum conditions for the element were obtained and
applied to remove MB. Kinetic models, infrared, and electrochemical of adsorbent before and after adsorption
were applied to deduce the mechanism of MB removal over magnetic Mg0.5Zn0.5Fe2O4 nanomaterials.

2. Experimental details

2.1. Preparation and characterization of magnesium–zinc ferrites

The rapid combustion approach was adopted to prepare magnetic magnesium–zinc ferrites. Ferric nitrate
nonahydrate, magnesium nitrate hexahydrate, and zinc nitrate hexahydrate were used due to their
stoichiometric proportions. Metal nitrates were dissolved in different volumes of absolute ethanol with
magnetic stirring. The prepared solutions were placed in crucible and ignited. The acquired precursors had
undergone the calcination for 2 h at the temperature of 400 °C. The final, magnetic Mg0.5Zn0.5Fe2O4
nanomaterials were obtained.

The surface texture and elemental compositions of the products were analyzed via the scanning electron
microscopy (SEM) and the x-ray energy dispersive spectrometer (EDS); the FT-IR spectra of pure samples, MB,
the samples adsorbed MB, and recycling samples were also examined; the x-ray diffraction was going on test the
crystalline structure of the products; the saturation magnetizations of power specimens were measured by the
vibrating sample magnetometer (VSM).

2.2. Adsorption experiment

Firstly, magnetic Mg0.5Zn0.5Fe2O4 nanoparticles were placed in MB solution with concentrations of 500–1000
g L−1 to analyze the adsorption behavior. After being dispersed for 5 min by ultrasonic wave, the adsorption
process was performed for 10–180 min. When the adsorption process was finished, magnetic field was applied to
separate the adsorbent, and the remnant liquid was taken to measure the absorbances on UV. The absorbance
(q) and the removal efficiency (η) of the nanomaterials were calculated on the basis of equations (1) and (2) [31].

\[ q = \frac{V(C_0 - C)}{m} \]  
\[ \eta = \frac{(C_0 - C)}{C_0} \times 100\% \]  

where \(C_0\) (mg L−1), \(C\) (mg L−1), \(V\) (mL), and \(m\) (g) respectively represented original concentration, post-
adsorption concentration, the volume of solution, and the mass of adsorbent.

Subsequently, 1 M HCl along with NaOH solutions were applied to coordinate the pH (pH = 1, 3, 5, 7, 9,
and 11) of MB solutions, and the influence of different pH values on the removal rate was investigated. Finally, in
order to research the cycle property of magnetic Mg0.5Zn0.5Fe2O4 nanoparticles, 75 mg of the product was
placed in MB solution, and after adsorption, it was separated, and the absorbance of MB was measured according
to formula (1), and the solid adsorbed MB was put into the crucible to calcine again 2 h at 400 °C, the
above steps were cycled six times.

3. Results and discussion

3.1. Characterization of Mg0.5Zn0.5Fe2O4 nanoparticles

SEM morphology, EDS, XRD, and VSM spectra of Mg0.5Zn0.5Fe2O4 nanomaterials were revealed in figure 1.
Figure 1(A) indicated that the prepared magnetic Mg0.5Zn0.5Fe2O4 nanomaterials were the spherical shape, and
the average diameter of them was 19.35 nm. Figure 1(B) displayed the EDS pattern of the magnetic
Mg0.5Zn0.5Fe2O4 nanoparticles, which indicated the presence of Mg, Zn, Fe, and O, and further verified that
Mg0.5Zn0.5Fe2O4 nanoparticles were successfully prepared. The recorded x-ray diffraction patterns for the
prepared samples at 400 °C for 2 h were displayed in figure 1(C), the plane with crystal planes values of (220),
(311), (400), (422), and (440) could be matched correctly with Mg0.5Zn0.5Fe2O4 standard x-ray diffraction
pattern (JCPDS No. 04–001–9289) [32]. It was observed from figure 1(D) that the saturation magnetization of
the magnetic Mg0.5Zn0.5Fe2O4 nanoparticles was 16.6 e m u /g.
The results of XRD and magnetic property patterns of magnetic Mg\textsubscript{x}Zn\textsubscript{1-x}Fe\textsubscript{2}O\textsubscript{4} nanomaterials at various conditions were revealed in figure 2. Figure 2(A) showed the XRD patterns of Mg\textsubscript{x}Zn\textsubscript{1-x}Fe\textsubscript{2}O\textsubscript{4} nanoparticles, it could be seen that as the content of Mg increased, the crystallinity of the nanomaterials gradually decreased. When the x value increased from 0.6 to 0.9, the characteristic peak was almost invisible, so it was considered that the nanomaterials did not form a crystallinity, and when x equaled to 0.5, the characteristic peak was the widest and lowest. Because the characteristic peak was wider and lower, the average grain size was smaller, and its specific surface area was larger, which would lead to better adsorption performance. In summary, magnetic Mg\textsubscript{0.5}Zn\textsubscript{0.5}Fe\textsubscript{2}O\textsubscript{4} nanoparticles were the optimum adsorbent. As revealed in figure 2(B), when the mole variable of magnesium was 0.5, its magnetic property was medium level, indicating that the nanoparticles had good magnetic property, which could contribute to the recycling of the nanomaterials. According to figure 2(C), it could be seen that as the calcination temperature for the nanomaterials rose, the crystallinity of the nanomaterials gradually rose. This reason was that too high temperature would cause the agglomeration of the nanoparticles, the agglomeration would cause the crystal size to become larger; at the same time, the magnetic property increased (figure 2(D)). Figures 2(E) and (F) displayed the XRD patterns and hysteresis loops of Mg\textsubscript{0.5}Zn\textsubscript{0.5}Fe\textsubscript{2}O\textsubscript{4} nanoparticles formed with different amounts of ethanol. Apparently, with the increase of absolute ethanol amount, the characteristic peak was first enhanced and then reduced. When the amount of absolute ethanol was 20 ml, the characteristic peak was the widest and least, and the average diameter of the nanoparticles also reached the minimum. As depicted in figure 2(F), the magnetic property rose from 21.3 emu g\textsuperscript{-1} to 50.4 emu g\textsuperscript{-1} with absolute ethanol increasing from 30 to 50 ml. Then, the further increase of ethanol resulted in the decrease of the magnetic property. Based on the above results, x value of 0.5, calcination temperature of 400 °C, and 20 ml ethanol were chosen as the best conditions for the later experiments.

The average crystalline size of the magnetic Mg\textsubscript{x}Zn\textsubscript{1-x}Fe\textsubscript{2}O\textsubscript{4} nanoparticles was calculated by using Scherer’s equation (3) [33].

$$D = \frac{0.89\lambda}{\beta \cos \theta}$$

Figure 1. SEM morphology (A), EDS spectrum (B), XRD pattern (C), and VSM (D) of magnetic Mg\textsubscript{0.5}Zn\textsubscript{0.5}Fe\textsubscript{2}O\textsubscript{4} nanoparticles.
where $\lambda$, $\beta$, and $\theta$ were wavelength, full width at half maximum (FEWH), and the angle of XRD, respectively. $D$ (nm) represented the average crystallite size of the nanoparticles.

The average grain sizes of magnetic $\text{Mg}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ nanoparticles under diverse conditions were revealed in figure 3. The average grain size of $\text{Mg}_0.5\text{Zn}_0.5\text{Fe}_2\text{O}_4$ nanoparticles was on the rise as the temperature went up based on figure 3(A). This reason was that the high calcination temperature contributed to the crystal growth. The impact of ethanol amount from 10–100 ml on the average grain size of nanoparticles was shown in figure 3(B). When the amount of ethanol was 20–50 ml, as the burning time increased, the sintering degree of the nanomaterials also increased, which resulted in a rise of the average grain size. Therefore, the sintering degree was the main factor affecting the average grain size. At the same time, the further rise of ethanol amount resulted in the better dispersibility of the solute, and reduced grain size of the nanomaterials. In summary, When the calcination temperature was maintained at 400 °C, the nanomaterials prepared with 20 ml of ethanol best met the experimental expectations, which conformed with the explain in figure 2.
3.2. Adsorption kinetics

500–1000 mg L\(^{-1}\) \(\text{B}^{-1}\) solutions were used to run the MB adsorption experiments of the nanoparticles. The pseudo-first-order, pseudo-second-order, together with intraparticle diffusion kinetic models were applied to investigate the adsorption process. The MB adsorption capacity and removal efficiency curves at different MB concentrations were revealed in figure 4. According to figure 4(A), as the solution concentration improved, the adsorption capacity increased and attained the maximum adsorption capacity of 397.73 mg g\(^{-1}\) at 1000 mg L\(^{-1}\). The reason was that the concentration difference between the adsorbent surface and the solution increased, and the adsorption power enhanced, that resulted in the adsorption capacity increased. The MB removal efficiency of magnetic Mg\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) nanoparticles rose from 90% to 99% over time. The sustained increase of MB concentration would lead to the decrease of MB removal efficiency in the first 10 min, such as 91% of MB removal efficiency at 1000 mg L\(^{-1}\) (figure 4(B)). The removal efficiency gradually achieved balance at about 60 min. The reason was that there were many adsorption and binding sites inside the nanomaterials, as the adsorption progressed, the internal pores were gradatim took up MB molecules and reached saturation. When the nanomaterials reached the maximum adsorption capacity, the rate did not change. The adsorption process was segmented into three parts, the movement of MB molecules from the fluid body to the outer surface of magnetic Mg\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) nanoparticles, it enters the pore size through external diffusion, then diffuses to the inner surface of nanoparticles, and finally was adsorbed on the inner surface of nanoparticles. The three kinetics models were represented by the following three equations (4)–(6), which could generally illustrate the mechanism and behavior of adsorption [34–36].
where $q_e$ and $q_t$ were the equilibrium state and adsorption capacities (mg g$^{-1}$); $k_1$, $k_2$, and $k_i$ were rate constants of three kinetic models; $C_i$ was constant.

The fitting results of three kinetics models at diverse MB concentrations were displayed in figure 5. Table 1 displayed the obtained parameters. As revealed in graph and parameters, the obtained experimental results were perfectly fitted in pseudo-second-order model than another two models, indicated that the adsorption process of MB onto Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles conformed to the chemical adsorption mechanism [37]. The regression coefficients ($R^2$) exceeded 0.98.
3.3. Electrochemical performance of MB onto Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles

Electrochemical characterizations in different states were achieved using cyclic voltammetry [23, 38]. Figure 6 showed the CV curves of bare MGCE electrode, MGCE/magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles, and MGCE/Post-adsorption magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles. As revealed in figure 6, bare MGCE was adopted to discern redox peaks. When the magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles were modified onto the electrode surface, the redox peak current decreased because of steric hindrance effect after modification. Hence, the contact between electrode and [Fe(CN)$_6$]$_{3/-4}$ in electrolytic solution could be validly controlled. Afterwards, the surface of the electrode was treated with the adsorbed magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles, and this resulted in a prominent reduction in electricity peak. This peak descend might be attributed to the rejection resistance of [Fe(CN)$_6$]$_{3/-4}$ and MB molecule. These results proved that MB had been adsorbed onto magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles.

3.4. Influence of pH and recycle of nanoparticles

The removal efficiencies at various pH values were depicted in figure 7(A). The removal efficiency of the removal of MB for Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles was low of 83% with pH of 1, indicated that the strong acid condition were not conductive to MB adsorption. With the pH value increasing from 1 to 3, the MB removal efficiency of magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles enhanced from 83% to 99%. When pH overtopped 3, the removal efficiency held steady, and the adsorbance of MB for the prepared nanoparticles was 318.18 mg g$^{-1}$. The charge of the MB molecules changed with pH. The MB molecules were positively charged when pH was less than 3, and the magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles were also positively charged, which would cause strong franklinic.

Table 1. Kinetic parameters of MB adsorbed by Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles.

| Kinetic model                      | Equation | Parameter | Initial MB concentration (mg l$^{-1}$) |
|-----------------------------------|----------|-----------|----------------------------------------|
| Pseudo-first-order model          | $q_t = q_e (1 - e^{-kt})$ | R$^2$ | 0.6910 0.7850 0.7470 0.7725 0.6947 0.6931 |
|                                   | $k_t$    | 0.4605 0.4684 0.1667 0.2529 0.2833 0.2643 |
|                                   | $q_e$    | 198.3701 238.3688 277.1693 314.9203 353.1641 392.3152 |
| Pseudo-second-order model         | $q_t = q_e^2t/k_2 + k_2t$ | R$^2$ | 0.9800 0.9900 0.9825 0.9906 0.9880 0.9869 |
|                                   | $k_2$    | 0.0395 0.1107 0.6912 0.0031 0.0036 0.0027 |
|                                   | $q_e$    | 198.8205 238.8475 279.0497 320.1339 359.8408 398.5581 |
| Intraparticle diffusion model     | $q_t = C_i + kt^{1/2}$ | R$^2$ | 0.7153 0.6050 0.6210 0.6645 0.7248 0.7706 |
|                                   | $k_i$    | 0.0395 0.1667 0.6835 1.9411 1.8160 2.4477 |
|                                   | $C_i$    | 0.5 0.5 0.5 0.5 0.5 0.5 |

Figure 6. Cyclic voltammograms of bare MGCE electrode, MGCE/magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles, and MGCE/Post-adsorption magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles.
resistance between them and affect the removal process, resulting in low removal efficiency. However, With the pH value increasing from 1 to 11, the number of positive charges carried by the MB molecules gradually decreased, so the electrostatic resistance decreased, the adsorption process would become easier, and the adsorption capacity would increase accordingly. The cyclic utilization revealed in figure 7(B) indicated that the relative removal rate could maintain above 99% in the first six cycles of experiments. After the seventh calcination, the removal of MB dropped to 96%. While the reason was that the repeated calcination caused the pore collapse of the magnetic Mg0.5Zn0.5Fe2O4 nanoparticles, and the specific surface area of the nanomaterials would be also decreased, resulting in a smaller contact area between the MB molecules and the magnetic Mg0.5Zn0.5Fe2O4 nanoparticles. The adsorption capacity decreased accordingly. Overall, after seven cycles, the adsorption capacity and relative removal rate remained at a high level, indicating that magnetic Mg0.5Zn0.5Fe2O4 nanoparticles had good recycling performance.

3.5. FT-IR spectra certification for MB onto Mg0.5Zn0.5Fe2O4 Nanoparticles
Figure 8 displayed the infrared spectra of fresh Mg0.5Zn0.5Fe2O4 nanoparticles (a), MB (b), the Mg0.5Zn0.5Fe2O4 nanoparticles after absorbing MB (c), and the Mg0.5Zn0.5Fe2O4 nanoparticles desorbed after calcination at 400 °C for 2 h (d).

![Figure 7. Influence of pH and recycle of Mg0.5Zn0.5Fe2O4 nanoparticles.](image)

![Figure 8. FT-IR spectra of fresh Mg0.5Zn0.5Fe2O4 nanoparticles (a), MB (b), the Mg0.5Zn0.5Fe2O4 nanoparticles after absorbing MB (c), and the Mg0.5Zn0.5Fe2O4 nanoparticles desorbed after calcination at 400 °C for 2 h (d).](image)
Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles and MB in the infrared spectra could be clearly seen, which suggested that MB was adsorbed onto Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles. Besides, it could be seen from the infrared spectroscopy of the Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles recalcined at 400 °C for 2 h that the characteristic peaks of MB onto Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles completely disappeared, which illustrated that the recalcined Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles could be recycled.

4. Conclusions

In present investigation, a one-step rapid combustion approach was applied to prepare magnetic magnesium-zinc ferrites. Through the analysis of XRD and VSM, magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles were employed for MB removal. The adsorption process belonged to chemical adsorption on the basis of the pseudo-second-order models by fitting the experimental results. The removal efficiency of MB adsorbed by Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles maintained above 99% when pH went beyond 3. After seven cycles of recalcination, the relative removal rate remained 96% of the first one. Hence, magnetic Mg$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles exhibited excellent reconstruction performance.

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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