PAPER

An innovative alcohol-solution combustion-calcination process for the fabrication of NiFe$_2$O$_4$ nanorods and their adsorption characteristics of methyl blue in aqueous solution

Qi Xu$^1$, Yueyang Xu$^{1,2}$, Jianming Xue$^1$, Fahua Zhu$^1$, Zhaoping Zhong$^1$ and Ruijiang Liu$^{1,3}$$^{*,}$

$^1$ State Key Laboratory of Clean and Efficient Coal-fired Power Generation and Pollution Control, China Energy Science and Technology Research Institute Ltd, Nanjing 210031, People’s Republic of China

$^2$ School of Energy and Environment, Southeast University, Nanjing 210096, People’s Republic of China

$^3$ School of Pharmacy, Jiangsu University, Zhenjiang 212013, People’s Republic of China

* Authors to whom any correspondence should be addressed.

E-mail: yueyang_xu@163.com and luckystar_lrj@ujs.edu.cn

Keywords: magnetic NiFe$_2$O$_4$ nanorods, alcohol-solution combustion-calcination technique, methyl blue, adsorption characteristics

Abstract

Magnetic nickel ferrite (NiFe$_2$O$_4$) nanorods were prepared via an innovative alcohol-solution combustion-calcination technique and evaluated for removing methyl blue (MB), which has a significant negative effect on aquatic life because of its high toxicity and color. The nanorods were characterized by TEM, EDS, XRD, VSM, SAED, FTIR, XPS and BET, the results showed that the NiFe$_2$O$_4$ sample has high magnetic saturation (Ms) and soft superparamagnetic behavior, and these properties accounted for their facile separation from the aqueous solution when an external magnetic field was applied. To understand the adsorption mechanism, adsorption experiments were performed using adsorption kinetics and adsorption isotherms. The Temkin model and the pseudo-second-order kinetic model best described the adsorption characteristics of MB onto NiFe$_2$O$_4$ nanorods. The effect of pH on the adsorption process was investigated, when pH was 3–7, the maximum adsorption capacity was reached, which was about 62 mg g$^{-1}$. The recycling efficiency was also estimated, after 10 runs of regeneration, it remained 70.1% initial adsorption capacity, indicating the adsorbent could be efficiently reused for the adsorption of MB.

1. Introduction

Nanomaterials have many significant properties, such as large specific surface area, excellent electrical conductivity, tiny size, facile dispersion [1–3], and so on. Thus, they are widely applied in bioengineering [4], environmental science [5], pharmaceuticals [6], medical [7], wastewater treatment [8], etc. Recently, magnetic nanomaterials have showed outstanding performances for removing dyes from wastewater [9], which were generated by industrial effluents such as cosmetics, paper, textiles, etc. Dye polluted water was not only harmful to the health of human and aquatic lives but also difficult to be disposed, which has become a demanding prompt solved problem [10].

Many strategies were available for purifying dye pollutions, including adsorption [11, 12], photocatalysis [13] and biological treatment [14], electrochemical processes [15], etc. Although photocatalysis and other methods could achieve good treatment of dye-polluted water, these methods were either more complex steps, or higher requirements for equipment [16, 17]. The adsorption method was regarded as one of the most competitive and effective techniques because of its convenient operation, high efficiency, low cost and it would cause no further damage to the water resources [18, 19]. Selecting an appropriate adsorbent has great significance for the adsorption of dye wastewater. Conventional adsorbents suffered from drawbacks like high cost, difficulty in regeneration, low adsorption ability, and limits in terms of separation [20], which have restricted their potential application in the environmental field.
Adsorption combined with magnetic nano-adsorbents has drawn great attention [21, 22]. Nano ferrites as one of the most important magnetic nano-adsorbents would be promising for removing dye from wastewater [23–25]. They are proved to be superior as magnetic nanomaterials due to their unique advantages of excellent magnetic performance, easy operation process, and higher adsorption capacity for dyes [26, 27]. There were many methods to prepare magnetic nanomaterials, such as hydrothermal method [28], co-precipitation [29], sol-gel [30], micro-emulsion [31], etc. However, in the preparation process of magnetic nano ferrite, these methods often have some disadvantages, such as long preparation cycle, complex operation, high equipment requirement, uncontrollable condition, introduction of impurity, and so on. So, the preparation of monodispersed ferrite particles with controlled synthetic conditions remains a challenge to date.

To overcome these drawbacks, we introduced a facile alcohol-solution combustion-calcination technique with characteristics such as a less preparation time, no need for dispersants, homogeneous products, and magnetism easily controlled for the preparation of magnetic NiFe$_2$O$_4$ nanorods. Magnetic NiFe$_2$O$_4$ nanorods prepared via the technique showed many excellent properties, such as enhancing adsorption area, excellent stability, reducing costs, high saturation magnetization, environmentally benign, and facile separation by an external magnet.

![Figure 1](image.png)

**Figure 1.** SEM morphology (A), TEM image (B), EDS pattern (C), SAED pattern (D), XRD pattern (E), and hysteresis loops (F) of magnetic NiFe$_2$O$_4$ nanorods calcined at 400 °C for 2 h with absolute alcohol of 30 ml.
In this project, magnetic NiFe₂O₄ nanorods were fabricated for the adsorption of methyl blue (MB) and they were characterized by TEM, EDS, XRD, VSM, SAED, FTIR, XPS and BET. Besides, their adsorption parameters, the effect of pH, and the recycling efficiency were discussed. This paper provided a novel and effective adsorbent for the adsorption treatment of dyes and furnished basic experimental data for the adsorption of MB by NiFe₂O₄ nanorods.

2. Experimental Details

2.1. Preparation and characteristics of magnetic NiFe₂O₄ nanorods
Magnetic NiFe₂O₄ nanorods were prepared via a facile alcohol-solution combustion-calcination technique. A certain amount of analytical grade iron nitrate [Fe(NO₃)₃·9H₂O] and nickel nitrate [Ni(NO₃)₂·6H₂O] were dissolved in 30 ml absolute ethanol. Then, the mixed solution was stirred with a magnetic stirrer for 1 h at room temperature. After that, the homogeneous solution was transferred to the crucible and ignited. When the flame went out, a gel-like substance was obtained. The crucible with intermediates gel inside was placed in a series resistance furnace, calcined at 400 °C for 2h with a heating rate of 3 °C·min⁻¹. Finally, the solid substance was cooled down to room temperature and ground to obtain the samples. For each group of adsorption experiments, three parallel experiments were carried out, and the corresponding results were reflected in the error bars.

2.2. Adsorption of MB onto NiFe₂O₄ nanorods
Firstly, the MB solutions with the variable concentrations from 100 to 400 mg·L⁻¹ were configured and stood still for 24 h. Subsequently, 2 ml MB aqueous solution and about 5 mg of magnetic NiFe₂O₄ nanorods were added into each centrifuge tube. All the tubes were sonicated for 5 min and then placed on the tube revolver to rotate. After 5–160 min, the tubes were taken down. Besides, by keeping the tube revolver spinning and shaking at room temperature for 24 h, the adsorption equilibriums were performed. Then, the magnetic NiFe₂O₄ nanorods were separated by an external magnetic field. The supernatant in the tubes was collected and the adsorption amount of MB was determined by a UV–vis spectrophotometer.

3. Results and Discussion

3.1. Characteristics of magnetic NiFe₂O₄ nanorods
The microstructures of the as-prepared nickel ferrite were investigated by SEM and TEM analysis. As shown in figures 1(A) and 1(B), the prepared magnetic NiFe₂O₄ nanomaterials were surface morphology of agglomerated nanorods ranging from 106.2 nm to 273.5 nm with an average size of 185.0 nm and average diameter of 25.7 nm. Also, the agglomeration may be due to the magnetic properties of the nanorods, which were further tested in the magnetic section. Besides, Peaks for NiFe₂O₄ nanorods could be visualized in EDS spectra of nickel ferrite in figure 1(C), which indicated the existence of Ni, O, and Fe elements in the composed material, the atomic percentage (atomic %) of nickel, iron, and oxygen was almost corresponded with the supposed content of the material preparation, confirming the successful synthesis of NiFe₂O₄. Figure 1(D) showed the nanorods had a NiFe₂O₄ polycrystalline structure. The crystal structure of the as-prepared samples was analyzed with XRD and the result was shown in figure 1(E). All the diffraction peaks of the product at 2θ crystal planes matched well to
spinel nickel ferrite standard PDF card (JCPDS No. 86–2267), which verified that the product was NiFe₂O₄. The hysteresis loops of NiFe₂O₄ were recorded via the vibrating sample magnetometer (VSM) at room temperature and presented in figure 1(F), which revealed that it was a typical narrow magnetic curve for ferromagnetic nanomaterials. The saturation magnetization (Ms) of the rod-structure NiFe₂O₄ was 86.0 Am²·kg⁻¹, revealing a clear soft superparamagnetic behavior of these nanorods [32], which allowed it to be applied in magnetic separation thus benefiting the dye removal and metal resources recycling in wastewater treatment.

3.2. Adsorption of MB onto NiFe₂O₄ nanorods
The magnetic properties of NiFe₂O₄ nanorods adsorbed MB were detected and shown in figure 2(A), after the adsorption of MB, the saturation magnetization of NiFe₂O₄ nanorods reduced 6.3 Am²·kg⁻¹. Although the magnetic properties of the nanomaterials had been slightly weakened, the overall magnetic properties of NiFe₂O₄ nanorods were still very superior, and the material recovery after adsorption could be realized through magnetic separation. Figure 2(B) displayed the SEM morphology of NiFe₂O₄ nanorods after the adsorption of MB. It could be seen that the adsorbed NiFe₂O₄ nanorods had a slight aggregation phenomenon compared with NiFe₂O₄ nanorods without adsorption, which was because when NiFe₂O₄ nanorods with positive surface charge...
was covered by MB with negative surface charge, the electrostatic repulsion of the neighboring nanomaterial was slightly reduced. Although the magnetic properties of NiFe₂O₄ nanorods were also reduced a little bit at this time, their saturation magnetization remained at a high level, so the aggregation of NiFe₂O₄ nanorods after adsorption was more obvious than before adsorption. However, the overall morphology of NiFe₂O₄ nanorods did not change significantly, which indicated that the effect of NiFe₂O₄ nanorods on MB would not damage the structure of nanomaterials themselves, indicating the possibility of recycling.

Fourier transform infrared spectroscopy (FTIR) was performed to detect the adsorption of MB on magnetic NiFe₂O₄ nanorods. The figure 3 showed the FTIR spectra of NiFe₂O₄, MB, the NiFe₂O₄ nanorods after adsorption of MB and the NiFe₂O₄ nanorods after re-calcination. It could be clearly concluded that there was a Fe-O bond peak (560 cm⁻¹, 482 cm⁻¹) in the infrared spectrum of bare NiFe₂O₄ nanorods (curve a). In the infrared spectrum of MB (curve b), the peak located at 1577 cm⁻¹ belonged to the stretching vibration peak of the benzene ring skeleton, the peak located at 1338 cm⁻¹ belonged to the C-N peak, and the C-H bond on the benzene ring were located at the peaks in the low frequency region. Curve c was the peak of the NiFe₂O₄ nanorods after the adsorption of dye, from which it could be clearly seen that there were both characteristic peaks of ferrite and MB in the spectrum, which could be confirmed that the removal of MB by NiFe₂O₄ nanorods belonged to adsorption rather than degradation. Curve d was the infrared spectrum after calcination of the NiFe₂O₄ nanorods with adsorption effect. There was only the peak of Fe-O bond in the figure, indicating that MB was completely removed by the calcination process. Therefore, the recycling of the nanomaterials could be realized by the calcination program.

Figure 4 displayed the removal amounts of MB from aqueous solution by a certain amount of NiFe₂O₄ nanorods with various initial concentrations of MB which varied from 100 mg·L⁻¹ to 400 mg·L⁻¹ at different times. In the first 20 min, the adsorbance of MB onto NiFe₂O₄ nanorods rapidly rose from 34.80% to 96.12%, and then gradually decreased over time until the equilibrium of adsorption-desorption, implying that there was a strong affinity between MB and the surface of magnetic NiFe₂O₄ nanorods. Therefore, the rapid adsorption of MB in the first 20 min could be attributed to the low total occupancy of the available surface sites of the NiFe₂O₄ surface [33]. The adsorption rate gradually decreased during the adsorption process until the constant plateau.
was reached. Moreover, the removal amount of MB dye increased with the concentration of MB increasing because more dye molecules could be adsorbed to the surface of the adsorbent in a higher initial concentration solution.

To estimate adsorption capability, three following models have been widely applied: the pseudo-first-order, pseudo-second-order, and intra-particle diffusion models [34]. Herein, the adsorption kinetics of MB onto NiFe$_2$O$_4$ samples (displayed in figure 5) were investigated to examine the potential mechanism.

The pseudo-first-order is presented as (equation (1)):

$$\ln q_e - q_t = \ln q_e - k_1 t$$

The pseudo-second-order model is presented as (equation (2)):

$$t \over q_t = (1 \over k_2q_e^2) + t \over q_e$$

The intra-particle diffusion model is presented as (equation (3)):

$$q_t = C_i + k_i t ^ {1 \over 2}$$

Where, $q_e$ (mg·g$^{-1}$) is the adsorbed amount at equilibrium. $k_1$ (min$^{-1}$), $k_2$ (g·mg$^{-1}$·min$^{-1}$), and $k_i$ (mg·g$^{-1}$·min$^{-0.5}$) denote the rate constant of pseudo-first-order model, pseudo-second-order model, and intra-particle diffusion model kinetic adsorption, respectively. A constant depicting resistance in the boundary layer is described as $C_i$ value (mg·g$^{-1}$).

The kinetic parameters for MB adsorption onto NiFe$_2$O$_4$ nanorods were summarized in table 1. According to the pseudo-second-order kinetic model, the adsorbed amount of MB at equilibrium ($q_e$) was almost the same as the experimental $q_e$, and the correlation coefficient was highest ($R^2 > 0.982$); while, the correlation
The coefficient of the pseudo-first-order model and intraparticle diffusion model were both under 0.80, indicating the pseudo-second-order model fitted well with the adsorption process [35]. Hence, the pseudo-second-order model was optimal to explain the MB adsorption mechanism onto the rod-structure NiFe$_2$O$_4$ samples, which meant adsorption efficiency was related to chemisorption [36], and electron transfer between the adsorbent and MB happened. In figure 6, the linearized plots of the adsorption amount versus pseudo-second-order kinetics model in the initial MB concentration range of 100–400 mg·L$^{-1}$ were obtained.

Figure 7 showed the equilibrium isotherms for adsorption of MB onto NiFe$_2$O$_4$, and the equilibrium adsorption characteristics were analyzed. To describe how MB interacted with NiFe$_2$O$_4$ and evaluate the mechanism of adsorption, the equilibrium adsorption isotherm models [37], were the most important tools. The experiments were performed by varying the initial MB concentration from 100 to 400 mg·L$^{-1}$.

The Langmuir isotherm model is represented as (equation (4)):

$$ q_e = \frac{q_{\text{max}} K_L C_e}{1 + K_L C_e} \quad (4) $$

Where $C_e$ (mg·L$^{-1}$) is the equilibrium concentration of the MB aqueous solution, $q_{\text{max}}$ is the maximum MB adsorption amount onto adsorbent (mg·g$^{-1}$), and $K_L$ is the Langmuir adsorption equilibrium constant (L·mg$^{-1}$), $q_e$ (mg·g$^{-1}$) is the mass of dye adsorbed per unit mass of adsorbent.

![Figure 7. Modeling of MB adsorption isotherms for Langmuir, Freundlich and Temkin models.](image)

![Figure 8. Effect of pH (A) on the adsorption of MB onto NiFe$_2$O$_4$ nanorods with initial MB concentration of 200 mg·L$^{-1}$ and the recycle efficiency (B) of NiFe$_2$O$_4$ nanorods for the removal of MB.](image)
The Freundlich isotherm described the heterogeneous surface, which assumes adsorption not only occurs in monolayer but also multilayer formations. A linear form of the Freundlich model can be expressed as the following equation (equation (5))

\[ q_e = K_F C_e^\frac{1}{n} \]

Where \( K_F \) is the Freundlich constant when the adsorption reaches equilibrium, \( 1/n \) measures the adsorption intensity.

The Temkin isothermal model takes the adsorbate-adsorbent interaction into account. The isotherm is given by a linearized expression as (equation (6)):

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

Where, \( A_T \) (L·mg⁻¹) is an equilibrium fitting parameter and \( B \) (J·mol⁻¹) is a constant related to the energy of sorption corresponding to the binding energy.

As could be seen from table 2, Temkin isotherm showed the highest correlation coefficient of 0.9957, suggesting that the adsorption value has a linear relationship with temperature, and the adsorption of MB onto NiFe₂O₄ nanorods was monolayer and multilayer combined mechanism [38].

### 3.3. Effect of pH value on MB adsorption and recycle of NiFe₂O₄ nanorods

The pH might affect the adsorption of MB. Figure 8(A) illustrated the adsorption capacity of MB onto NiFe₂O₄ nanorods within the pH range of 3–11 in the control of 0.1 M NaOH and HNO₃ solutions. The initial MB concentration was 200 mg L⁻¹ in these experiments. When the pH was 3–7, the adsorption capacity was the maximum, which was about 62 mg g⁻¹. When the pH was 7–9, it dropped slightly, but it remained at a relatively high value (>55 mg g⁻¹). The adsorption capacity continued to decrease at the pH of 10. It might because pH has a significant effect on the surface charge of adsorbent and the structure of dyestuffs. In a word, the maximum adsorption value was reached when the pH was 3–7.

The adsorption capacity of MB remained close to the initial adsorption capacity up to 10 runs, and the data were calculated in figure 8(B). The removal amount of dye decreased slightly with the increase of the number of experiments. When the number of experiments reached 10, the removal amount declined from 99.9% to 70.1%.

| Table 2. Evaluated model parameters of adsorption isotherms for MB onto magnetic NiFe₂O₄ nanoparticles at room temperature. |
|--------|--------|--------|--------|
| Model  | Equation | Adj. r-square | Parameters | Parameters value |
|--------|--------|--------|--------|--------|
| Langmuir | \( q_e = \frac{q_{\text{max}} K L C}{1 + K L C} \) | 0.9469 | \( q_{\text{max}} \) | 97.7268 |
| Freundlich | \( q_e = K_F C_e^\frac{1}{n} \) | 0.9527 | \( K_F \) | 40.7201 |
| Temkin | \( q_e = B \ln(A_T C_e) \) | 0.9957 | \( B \) | 16.4172 |
|        |        |        | \( A_T \) | 10.8763 |

Figure 9. XPS spectra of NiFe₂O₄ nanorods before (A) and after (B) adsorption.
However, the adsorption removal efficiency of MB was still at a relatively high value. Furthermore, the recycled NiFe$_2$O$_4$ was washed with deionized water three times to eliminate impurities. These data indicated that the as-prepared NiFe$_2$O$_4$ nanorods have high recycled efficiency for the adsorption of dye.

3.4. XPS analysis
X-ray photoelectron spectroscopy (XPS) was a technique for analyzing the properties of surfaces, XPS could measure the elemental composition, chemical and electronic states of elements in materials. In this paper, the XPS technology was employed to characterize the magnetic NiFe$_2$O$_4$ nanorods before and after the adsorption of MB (figure 9). The results revealed that the elements of the nanomaterials without adsorption were only Ni, Fe and O, which was a further characterization for the successful preparation of nickel ferrite. The XPS spectra of the magnetic NiFe$_2$O$_4$ nanorods after adsorption have increased N, C and S elements compared with those before adsorption. These elements were the components of MB dye, which indicated the presence of MB on the surface of the nanomaterials, and also confirmed the adsorption of MB on magnetic NiFe$_2$O$_4$ nanorods.

3.5. BET detection
BET specific surface area test was an effective strategy to determine the surface information of nanomaterials. The figure 10 described the nitrogen adsorption and desorption isotherms of magnetic NiFe$_2$O$_4$ nanorods before and after adsorption. This figure illustrated that according to International Union of Pure and Applied Chemistry (IUPAC) classification, the isotherms of magnetic NiFe$_2$O$_4$ nanorods before and after adsorption both belonged to type IV isotherms accompanied by type III hysteresis loops, indicating that adsorption did not change the mesoporous structure of NiFe$_2$O$_4$ nanorods. However, according to the relevant detection data, the specific surface area of the magnetic NiFe$_2$O$_4$ nanorods was 123.0 m$^2$ g$^{-1}$ when NiFe$_2$O$_4$ nanorods did not adsorb MB. When NiFe$_2$O$_4$ nanorods adsorbed MB, the surface area of the NiFe$_2$O$_4$ nanorods decreased to 94.4 m$^2$ g$^{-1}$, indicating that the surface sites of the magnetic NiFe$_2$O$_4$ nanorods were occupied by MB, and the specific surface area decreased, which confirmed the adsorption of MB on the nanomaterials.

3.6. Comparison of adsorbance with various adsorbents
Adsorption method had aroused wide interest among researchers in the field of dye treatment. Table 3 compared the adsorption capacity of different adsorbents for different dyes. It could be seen that the adsorption capacity of the adsorbent designed in this project was at a medium level among many adsorbents. The NiFe$_2$O$_4$ nanorods was magnetic and facile to recycle, and the magnetic NiFe$_2$O$_4$ nanorods could be regenerated by a

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Table 3. Adsorption capacity comparison between different adsorbents and magnetic NiFe$_2$O$_4$ nanorods.

| Adsorbent                                | Adsorbate       | $q_{\text{max}}$ (mg g$^{-1}$) | References |
|------------------------------------------|-----------------|---------------------------------|------------|
| Brown macroalga                          | Methylene blue  | 95.45                           | [39]       |
| Activated lignin-chitosan extruded pellets | Methylene blue  | 36.25                           | [40]       |
| Modified red mud                         | Crystal violet  | 38.16                           | [41]       |
| Polyaniline emeraldine salt              | Eosin yellow    | 335                             | [42]       |
| magnetic NiFe$_2$O$_4$ nanorods          | Methyl blue     | 62                              | Present work |

However, the adsorption removal efficiency of MB was still at a relatively high value. Furthermore, the recycled NiFe$_2$O$_4$ was washed with deionized water three times to eliminate impurities. These data indicated that the as-prepared NiFe$_2$O$_4$ nanorods have high recycled efficiency for the adsorption of dye.
simple calcination process and the adsorption dye level was still at a high level after being reused for 10 times. Therefore, this magnetic NiFe$_2$O$_4$ nanorods was of great value for development.

4. Conclusions

(1) Magnetic NiFe$_2$O$_4$ nanorods were successfully fabricated by the facile alcohol-solution combustion-calcination technique. The nanorods showed an average length of 185.0 nm and average diameter of 25.7 nm and displayed a high removal amount of MB (with initial concentration ranging from 100–400 mg L$^{-1}$), which was up to 96.12%.

(2) Temkin model and the pseudo-second-order kinetic model fitted well with the adsorption process, the NiFe$_2$O$_4$ nanorods exhibited high magnetic saturation and superparamagnetic behavior, making it easy for separating the adsorbent from the aqueous solution when a magnetic field was applied, which might not cause secondary damage to the environment in the process of adsorbing dye pollution. Besides, the adsorption value could reach maximum when the pH was 3–7.

Acknowledgments

The work was supported by the Science and Technology Innovation Project of CHN Energy (Grant No. GJNY-20–109).

Data availability statement

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

ORCID iDs

Ruijiang Liu  https://orcid.org/0000-0002-0716-832X

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