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Adsorption performance of magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for reactive red 2BF

Hezhong Ouyang$^{1*}$, Shuyan Liu$^{1,2}$, Zhou Wang$^3$, Aihua Liu$^1$, Dandan Liu$^{1*}$ and Shuping Xu$^{1*}$

$^1$ The People’s Hospital of Danyang, Affiliated Danyang Hospital of Nantong University, Zhenjiang 212300, People’s Republic of China
$^2$ College of Vanadium and Titanium, Panzhihua University, 617000, People’s Republic of China
$^3$ These authors contributed equally.

* Authors to whom any correspondence should be addressed.

E-mail: dyliudandan@163.com and dyxushuping@163.com

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Abstract

A novel nitrate solution combustion process for formation of magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles was introduced, and XRD, VSM, SEM, TEM, and BET techniques were employed to characterize the nanoparticles. For Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles prepared at 400 $^\circ$C for 2 h with 20 ml absolute ethanol, the average size and the saturation magnetization were approximately 22 nm and 8.1 A·m$^2$ kg$^{-1}$, respectively. Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanomaterials were subjected to reactive red 2BF adsorption, and the adsorption performances were investigated. The results revealed that the experimental data fit the Temkin isotherm model and the pseudo-second-order kinetics model, suggesting that the RR-2BF adsorption process was a monolayer-multilayer-associated chemisorption mechanism. The effects of pH on the adsorption capacity and cycle capacity of the magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for the adsorption of reactive red 2BF were revealed.

1. Introduction

The rapid development of human society has improved people’s material and cultural life, which has led to increasing environmental pollution, especially water pollution [1, 2]. Among different pollution sources, dye wastewater pollution has attracted extensive attention in dye applications in the textile industry [3]. Dye wastewater discharged from these industries can prevent visible light necessary for flora and fauna living under water; moreover, some of these dyes are toxic and even hypertoxic [4]. Accidental consumption of these dyes in large quantities is poisonous, and prolonged exposure can cause cancer [5]. Therefore, the removal of dyes from wastewater is imperative.

Contemporary methods for dye removal include chemical, physical, and microbiological methods [6, 7]. The adsorption method is considered the most effective owing to thorough-paced removal, low cost, and no secondary pollution [8, 9]. In this method, the key factor is the adsorbent selected [10]. Activated carbon is an ideal adsorbent owing to its high adsorption capacity [11]. For large specific surface areas, many researchers have applied nano-activated carbon and obtained good removal effects [12]. However, separating nanomaterials is challenging owing to their nano size [13]. For facile separation, some researchers have used magnetic nanomaterials for adsorption, since these can be separated using an external magnetic field, greatly simplifying the separation after the adsorption process [14, 15].

Contemporary techniques for synthesizing magnetic nanomaterials include the coprecipitation process [16], sol-gel process [17], hydrothermal process [18], nitrate solution combustion process [19, 20] and so on. Among these, the nitrate solution combustion process is preferred owing to its short production cycle, uniform production particle size, and facile operation [21–24].

Therefore, in this study, we prepared magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles via a novel nitrate solution combustion process. Using reactive red 2BF (RR-2BF) as the model of adsorbate, we investigated the adsorption performance of Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for RR-2BF, which may have applications in sewage treatment.
2. Experimental

2.1. Preparation and characteristic of Mg0.5Ni0.5Fe2O4 nanomaterials

The nitrate solution combustion process was applied to prepare Mg0.5Ni0.5Fe2O4 nanomaterials. Magnesium nitrate hexahydrate (99.0%, 1.19 g), nickel nitrate hexahydrate (98.0%, 1.37 g), and ferric nitrate nonahydrate (98.5%, 7.55 g) were added to 20 ml pure ethanol and dissolved to obtain a homogeneous solution. The solution was transferred into a crucible and fired; after the fire was quenched, the crucible and intermediate were placed in a programmed temperature-control furnace and calcined at 400 °C for 2 h. After calcination, the furnace was naturally cooled to room temperature, and the product was taken out, ground in an agate mortar, and magnetic Mg0.5Ni0.5Fe2O4 nanoparticles were obtained.

Scanning electron microscopy (SEM) and field-emission transmission electron microscopy (TEM) were used to observe the morphology; x-ray diffraction (XRD) was used for phase identification and particle characterization; a vibrating sample magnetometer (VSM) was used to measure the magnetism of the nanoparticles [6]; and the BET method was applied to measure and calculate the specific surface area of magnetic Mg0.5Ni0.5Fe2O4 nanoparticles.

2.2. Adsorption experiments

Mg0.5Ni0.5Fe2O4 nanoparticles (0.05 g) were placed in a 2 ml centrifuge tube. Then, 2 ml RR-2BF solution of a predetermined concentration (100–400 mg L\(^{-1}\)) was placed in a centrifuge tube, dispersed for 5 min using ultrasonic centrifugation, and the tube was slowly rolled for a certain time. When the adsorption process was terminated, Mg0.5Ni0.5Fe2O4 nanoparticles were separated from the solution by applying an external magnetic field [25]. The absorbance of the RR-2BF solution after adsorption was examined using ultraviolet spectrophotometry, and the residual concentration of RR-2BF was calculated on the basis of the linear relationship between the absorbance and the concentration, and then, the adsorption capacity and the removal efficiency (η) of Mg0.5Ni0.5Fe2O4 nanoparticles was obtained according to equations (1), (2) [26]:

\[
q = \frac{V(C_0 - C)}{m} \quad (1)
\]

and

\[
\eta = \frac{(C_0 - C)}{C_0} \times 100\% \quad (2)
\]

where q is the adsorption capacity per gram of adsorbent (mg g\(^{-1}\)), V is the volume of RR-2BF solution (L), and \(C_0\) and \(C\) are the initial concentration and the concentration of RR-2BF at determination time (mg L\(^{-1}\)), respectively. \(m\) is the quantity of the Mg0.5Ni0.5Fe2O4 nanoparticles (g).

A 0.01 M solution of either HCl and NaOH solution was used to adjust the pH value of the RR-2BF solution, and NaCl solution of 0.01 M and congo red solution of 500 mg L\(^{-1}\) were added into RR-2BF solutions to investigate the effects of ionic strength, coexisting ions and the presence of DOM on adsorption capacity of Mg0.5Ni0.5Fe2O4 nanoparticles for RR-2BF, the equilibrium absorption capacity was measured after 24 h of adsorption [27]. The experimental data was analyzed using the Origin software employing a non-linear fit.

3. Results and discussion

3.1. Characteristic of magnetic Mg0.5Ni0.5Fe2O4 nanomaterials

Figure 1 shows the characteristics of magnetic nanomaterials prepared at 400 °C for 2 h with 20 ml pure ethanol. As shown in the SEM morphology scans (figure 1(A)), the nanomaterials had a spherical structure and inhomogeneity, which suggested that the nanoparticles had larger magnetism; the average diameter was approximately 22 nm. Figure 1(B) shows a TEM image of the magnetic Mg0.5Ni0.5Fe2O4 nanoparticles, confirming that the average diameter of the particles is approximately 22 nm, in agreement with the SEM results, and from the gray level of the nanoparticles, the interiors of the nanoparticles were homogeneous. Figure 1(C) shows the XRD pattern. The diffraction peaks of magnetic Mg0.5Ni0.5Fe2O4 nanoparticles corresponded to (220), (311), (400), (511), (440), (622); all the diffraction peaks could be indexed to NiFe2O4 standard PDF (JCPDS No. 10–0325) and MgFe2O4 standard PDF (JCPDS No. 17–0464) card. XRD analysis results revealed that the Mg0.5Ni0.5Fe2O4 crystallized into cubic crystal structure with space group Fd-3m. The lattice parameter of Mg0.5Ni0.5Fe2O4 was only one of 8.35 Å and the average grain size was 23 nm. The hysteresis loop of the as-prepared product is displayed in figure 1(D), where the magnetic Mg0.5Ni0.5Fe2O4 nanoparticles exhibited soft and strong magnetic behavior, with a saturation magnetization of 8.1 A m\(^{-1}\) kg\(^{-1}\). The N2 adsorption-desorption isotherms of Mg0.5Ni0.5Fe2O4 nanoparticles was shown in figure 1(E), and the pore size distribution was revealed as inset of figure 1(E). It was seen that the adsorption-desorption isotherms belonged to IV type isotherm model, their specific surface area was calculated by the BET method, and the value was...
While, the adsorption isotherm was not coincident with the desorption isotherm, which revealed the existence of the internal surface. The pore size distribution of the nanoparticles was as the inset of figure 1(E), the pore size mainly distributed 2–4 nm, this provided the possibility of larger specific surface area. All the data benefited for the adsorption of RR-2BF.

3.2. Adsorption performances of Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for RR-2BF

3.2.1. Adsorption kinetics

The pseudo-first-order kinetics model, pseudo-second-order kinetics model, and intraparticle diffusion kinetics model were applied to reveal the adsorption mechanism of magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for RR-2BF removal. Details of these models [28] are presented in table 1.
Figure 2 shows the adsorption process and the removal efficiency of RR-2BF onto Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles prepared at 400 °C for 2 h with 20 ml pure ethanol.

### Table 1. Adsorption kinetic parameters of RR-2BF onto magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles at room temperature.

| Kinetic model                  | Equation                        | Parameter  | 100    | 200    | 300    | 400    | $R^2$  |
|-------------------------------|---------------------------------|------------|--------|--------|--------|--------|--------|
| Pseudo-first-order model      | $q_t = q_e(1 - e^{-kt})$        | $q_e$      | 31.9527| 72.4185| 98.1256| 115.3819| 0.6814 |        |
|                               |                                 | $k_1$      | 0.3489 | 0.4077 | 0.3672 | 0.2345 | 0.7006 | 0.7154 | 0.7088 |
| Pseudo-second-order model     | $q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t}$ | $q_e$      | 32.1560| 72.6946| 98.6794| 117.8003| 0.9881 |        |
|                               |                                 | $k_2$      | 0.0784 | 0.0639 | 0.0315 | 0.0067 | 0.9851 | 0.9903 | 0.9879 |
| Intraparticle diffusion model  | $q_t = x_i + k_1 t^\frac{1}{2}$ | $x_i$      | 31.1323| 71.4217| 96.0904| 106.4129| 0.7426 |        |
|                               |                                 | $k_1$      | 0.0866 | 0.1050 | 0.2143 | 0.9352 | 0.6963 | 0.7242 | 0.7517 |

$q_e$ and $q_t$ were the qualities of adsorbed RR-2BF at equilibrium and a sampling time (mg·g$^{-1}$), respectively [10]; $k_1$ (min$^{-1}$), $k_2$ (g·mg$^{-1}$·min$^{-1}$), and $x_i$ (mg·g$^{-1}$·min$^{-1}$) were the rate constants [29]; and $x_i$ was associated to the thickness of the boundary layer.

Figure 2 shows the adsorption process and the removal efficiency of RR-2BF onto the magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles prepared at 400 °C for 2 h with 20 ml pure ethanol and various concentrations of RR-2BF. As shown in figure 2(A), the adsorption process reached equilibrium after approximately 20 min; that is, the adsorption of Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for the adsorption of RR-2BF only required 20 min in the industrial sewage treatment process. While, the removal efficiency of RR-2BF reached maximum of 90.8% (figure 2(B)) when RR-2BF concentration was 200 mg l$^{-1}$. Thus, magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles are promising adsorbents for sewage treatment. The kinetics simulation analysis for RR-2BF onto magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles is shown in figure 3, and table 1 lists the related parameters. According to the variances ($R^2$), the pseudo-second-order kinetics was more suitable for reflecting the adsorption process compared with the other two models, which demonstrated that the adsorption mechanism of Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for RR-2BF belonged to chemical adsorption, and the linear relationships are displayed in figure 4.

#### 3.2.2. Adsorption isotherm

The Langmuir, Freundlich, and Temkin isotherm models were used to evaluate the adsorption equilibrium state of RR-2BF on magnetic Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles at room temperature [7]. Their equations [13] are presented in table 2.

| Equation                        | Parameter  | 100    | 200    | 300    | 400    | $R^2$  |
|---------------------------------|------------|--------|--------|--------|--------|--------|
| Langmuir model                  | $\frac{q_e}{C} = \frac{1}{K_L} + \frac{1}{q_m}$   | $K_L$  | 0.5321 | 0.5321 | 0.5321 | 0.5321 | 0.9999 |        |
| $q_m$ (mg·g$^{-1}$)             | $q_e$      | 31.9527| 72.4185| 98.1256| 115.3819|        |        |
| Freundlich model                | $q_e = \frac{1}{K_F} + \frac{1}{n K_F C}$        | $K_F$  | 0.5321 | 0.5321 | 0.5321 | 0.5321 | 0.9999 |        |
| $n$                             | $q_e$      | 31.9527| 72.4185| 98.1256| 115.3819|        |        |
| Temkin model                    | $q_e = \frac{1}{x_i} + \frac{1}{k_T C}$          | $k_T$  | 0.5321 | 0.5321 | 0.5321 | 0.5321 | 0.9999 |        |
| $x_i$                           | $q_e$      | 31.9527| 72.4185| 98.1256| 115.3819|        |        |

$q_e$ and $q_t$ were the qualities of adsorbed RR-2BF at equilibrium and a sampling time (mg·g$^{-1}$), respectively [10]; $k_1$ (min$^{-1}$), $k_2$ (g·mg$^{-1}$·min$^{-1}$), and $x_i$ (mg·g$^{-1}$·min$^{-1}$) were the rate constants [29]; and $x_i$ was associated to the thickness of the boundary layer.

The Langmuir model assumes that the adsorption of the adsorbate on the adsorbent is a monolayer, its adsorption capacity depends on the occupation rate of the active sites on the adsorbent surface, and the interface energy is uniform [29]. However, the Freundlich model assumes that the adsorption of adsorbate on the adsorbent is heterogeneous, the active sites are first occupied, and then the binding energy decreases with an increase in the number of occupied sites [10]. Whereas the Temkin model considers the adsorbent and the
adsorbate to interact, and the adsorption heat linearly decreases with an increase in the coverage [31], the binding energy is uniformly distributed, and it increases to a maximum binding energy.

According to the adsorption equilibrium data at room temperature, the fitting models are displayed in figure 5, and table 2 lists the adsorption isotherm parameters. Based on the determination coefficient ($R^2$) values and the relative errors of adsorption capacity ($q_{\text{max}}$), the Temkin model was found to be the best fitting model at

Figure 3. Adsorption kinetics simulation of RR-2BF onto Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles.

Figure 4. Linear relationships of the pseudo-second-order kinetics model for the adsorption of RR-2BF onto Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles.
room temperature, indicating that the adsorption state of Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles for RR-2BF was a multi-monolayer associative mechanism [26].

### 3.2.3. Effect of pH on adsorption capacity and adsorption cycle capacity

Figure 6(A) shows the influence of pH on the adsorption capacity of Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles for RR-2BF at room temperature; the adsorption capacity of Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles for RR-2BF at room temperature decreased with an increase in the pH value. This suggests that hydroxyl ions could compete with the adsorption sites of the adsorbent with RR-2BF molecules in the solution, and the hydrogen ion could exclude RR-2BF molecules in the solution. Therefore, the adsorption capacity of Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles for RR-2BF was larger in acidic environments, but smaller in alkaline environments. The cycle capacity is shown in figure 6(B), which shows that the adsorption capacity of RR-2BF continually decreased with the increase in cycle number because the magnetic Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles were always calcined for cycle application. The calcination may reduce the porosity of the magnetic Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles, causing the specific surface area to decrease, resulting in a decrease in the adsorption capacity. However, magnetic Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles demonstrate excellent cycle capacity, and the adsorption capacity retained at 65% after eight cycles. This implies that a promising application of Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles is for dye sewage treatment.

### Table 2. Adsorption isotherm parameters of RR-2BF onto magnetic Mg₀.₅Ni₀.₅Fe₂O₄ nanoparticles at room temperature.

| Isotherms model | Equation | R² | Parameter | Parameter’s value |
|-----------------|----------|----|-----------|-------------------|
| Langmuir        | \( q_e = \frac{q_{\text{max}} K_C C_e}{1 + K_C C_e} \) | 0.9586 | \( q_{\text{max}} \) | 95.8637 |
| Freundlich      | \( q_e = K_f C_e^{1/n} \) | 0.9839 | 1/n | 0.2860 |
| Temkin          | \( q_e = B \ln(A T C_e) \) | 0.9927 | \( A_T \) | 19.4851 |

\( q_e \) was the quality of adsorbed RR-2BF (mg g⁻¹) [29], \( C_e \) was the equilibrium concentration of RR-2BF in solution after adsorption (mg L⁻¹), \( q_{\text{max}} \) was the maximum adsorption capacity (mg g⁻¹) [30], \( K_C \) (L mg⁻¹) and \( K_f \) (mg L¹/n mg⁻¹) were the adsorption rate constants [31]. \( B = \frac{R T}{bT} \) was the constant, \( A_T \) was equilibrium binding constant for the maximum binding energy (L·g⁻¹), \( R \) was the universal gas constant (8.314 J·mol⁻¹·K⁻¹), \( T \) (K) was Kelvin temperature.
3.2.4. Effects of ionic strength, coexisting ions and the presence of DOM on adsorption capacity

Dye wastewater often contains some salts and dead organic matters (DOM), it is indispensable to evaluate their effects on the adsorption process. In this work, the effects of HCl, NaCl, NaOH and Congo red (CR) on the adsorption capacity of Mg0.5Ni0.5Fe2O4 nanoparticles for RR-2BF at room temperature were investigated. These usually existing substances in water solutions could provide common ions and organic matter, and the relative adsorption rates of Mg0.5Ni0.5Fe2O4 nanoparticles for RR-2BF were revealed in figure 7. With the increase of HCl in RR-2BF solution of 200 mg L−1, the relative adsorption rate increased. The reason might be that H+ greatly reduced the dissociation of RR-2BF in water solutions, resulting the solubility of RR-2BF decreased in water solutions, and then resulting the adsorption capacity of RR-2BF onto Mg0.5Ni0.5Fe2O4 nanoparticles increased. However, with the increase of NaOH in RR-2BF solution, the relative adsorption rate decreased. The reason might be that OH− greatly promoted the dissociation of RR-2BF in water solutions, increasing the solubility of RR-2BF decreased in water solutions, and then resulting the adsorption capacity of RR-2BF onto Mg0.5Ni0.5Fe2O4 nanoparticles decreased. While, when NaCl was dissolved in RR-2BF solution, the ionic strength was increased, because RR-2BF was sodium salt, Na+ decreased the dissociation and the solubility of RR-2BF in water solutions, so the relative adsorption rate increased, but the ascensional range was unapparent. When CR was also dissolved in RR-2BF solution, CR and RR-2BF belonged to the same type dye, therefore, the adsorptions of CR and RR-2BF onto Mg0.5Ni0.5Fe2O4 nanoparticles formed competition, resulting adsorption capacity of RR-2BF onto Mg0.5Ni0.5Fe2O4 nanoparticles decreased. Fortunately, the decrease of relative adsorption rate was non-significant, which suggested that the adsorption of Mg0.5Ni0.5Fe2O4 nanoparticles for RR-2BF had high selectivity.

3.2.5. FTIR for the adsorption process

To reveal the adsorption mechanism of RR-2BF on Mg0.5Ni0.5Fe2O4 nanoparticles, the FTIR spectra of Mg0.5Ni0.5Fe2O4 nanoparticles before adsorption, RR-2BF, Mg0.5Ni0.5Fe2O4 nanoparticles after adsorption, and the recalcined Mg0.5Ni0.5Fe2O4 nanoparticles were examined and revealed in figure 8, figure 8(a) showed the FTIR spectrum of Mg0.5Ni0.5Fe2O4 nanoparticles with only the Fe–O peaks appeared in 580 cm⁻¹ and 420 cm⁻¹, figure 8(b) revealed the FTIR spectrum of RR-2BF with the characteristic peaks of C–H bond, C=C bond on benzene ring and C–N bond of amine in RR-2BF molecules appeared in 1400–1800 cm⁻¹. When RR-2BF was adsorbed on Mg0.5Ni0.5Fe2O4 nanoparticles, their FTIR spectrum was displayed in figure 8(c), the peaks of C–H bond, C=C bond on benzene ring and C–N bond of amine in RR-2BF molecules appeared in 1400–1800 cm⁻¹ and the peaks of iron–oxygen bond were also displayed in the spectrum. Compared with figure 8(a), figures 8(b), and (c), it could be estimated that there was a chemical reaction in the adsorption of RR-2BF by Mg0.5Ni0.5Fe2O4 nanoparticles, which was mutually supported by the above research conclusions of adsorption kinetics. Figure 8(d) curve was the FTIR spectrum of the post-adsorbed Mg0.5Ni0.5Fe2O4 nanoparticles recalcined at 400 °C for 2 h, the characteristic peaks of RR-2BF disappeared, and only the characteristic peak of iron–oxygen bond was found, and the FTIR spectrum kept the same patterns, which suggested that Mg0.5Ni0.5Fe2O4 nanoparticles could be regained through the recalcination, in other words, except for adjusting pH, the adsorption capacity of Mg0.5Ni0.5Fe2O4 nanoparticles could be regained by the recalcination method. Therefore, the recalcination method could be applied to recycle the magnetic Mg0.5Ni0.5Fe2O4 nanoparticles.
4. Conclusions

This study is summarized as follows:

(1) Magnetic $\text{Mg}_0.5\text{Ni}_0.5\text{Fe}_2\text{O}_4$ nanoparticles were successfully prepared via a nitrate solution combustion process. The average particle size of the as-prepared product prepared at 400 °C for 2 h with 20 ml of pure ethanol was approximately 22 nm, and their saturation magnetization was 8.1 $\text{A} \cdot \text{m}^2 \text{kg}^{-1}$.

(2) The pseudo-second-order kinetics was best suited to reflect the adsorption process of $\text{Mg}_0.5\text{Ni}_0.5\text{Fe}_2\text{O}_4$ nanoparticles for RR-2BF, indicating chemical adsorption. The Temkin isotherm model was the most suitable for evaluating the adsorption state of RR-2BF on $\text{Mg}_0.5\text{Ni}_0.5\text{Fe}_2\text{O}_4$ nanoparticles at room temperature, and displayed that the adsorption was a multi-monolayer associative mechanism.
(3) The adsorption capacity of Mg$_{0.5}$Ni$_{0.5}$Fe$_2$O$_4$ nanoparticles for RR-2BF decreased with an increase in the pH value, but it still maintained 65% of the first adsorption capacity when the cycle number was eight times, revealing the promising application of the nanomaterials.

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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).

ORCID iDs

Hezhong Ouyang @ https://orcid.org/0000-0002-4173-6909
Dandan Liu @ https://orcid.org/0000-0001-9448-5560
Shuping Xu @ https://orcid.org/0000-0001-8832-1631

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