Plant-mediated synthesis of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles using Minidium leavigatum and their applications as an adsorbent for removal of reactive blue 222 dye

Mahsa Shojaei Yeganeh, Ali Reza Kazemizadeh, Ali Ramazani, Parvin Eskandari, and Hossein Rabbi Angourani

1 Department of Chemistry, Zanjan Branch, Islamic Azad University, P O Box 49195-467, Zanjan, Iran
2 Department of Chemistry, University of Zanjan, P O Box 45195-313, Zanjan, Iran
3 Research Institute of Modern Biological Techniques (RIMBT), University of Zanjan, P O Box 45195-313, Zanjan, Iran
4 Authors to whom any correspondence should be addressed.

E-mail: alirezakazemizadeh@yahoo.com, aliramazani@gmail.com and aliramazani@znu.ac.ir

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Abstract

Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ magnetic nanoparticles were synthesized using Minidium leavigatum extract and required metal salts by a green method. This method has some benefits such as nontoxic, economic viability, ease to scale up, less time consuming and environmental friendly approach for the synthesis of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ magnetic nanoparticles without using any hazardous organic chemicals. The sample was characterized by powder x-ray diffraction (XRD), fourier transform infrared spectroscopy (FTIR), vibrating sample magnetometer (VSM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), energy dispersive x-ray analysis (EDX), and Brunauer–Emmett–Teller (BET) surface area analysis. The environmentally friendly synthesized nanoparticles were used as adsorbent for removal of reactive blue 222 (RB 222) dye from aqueous solutions. The effects of initial dye concentration and nanoparticle dosage on dye adsorption were assessed. Adsorption equilibrium studies were investigated to determine the adsorption capacity of the adsorbents by using the Langmuir and Freundlich isotherm models. The Langmuir model yielded more suitable than the Freundlich model for the adsorption of RB 222 on Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ magnetic nanoparticles. Adsorption kinetics obeyed a pseudo second-order kinetic model.

1. Introduction

Nowadays, synthetic dyes are widely used in the textile, rubber, printing, leather, cosmetics, and plastics industries. One of the basic environmental problems of these industries is the pollution of wastewater with hazardous organic dyes. The presence of organic dyes in water resources causes a critical damage to aquatic species and prevents sunlight exposure for aquatic plant and animal species, thus it makes changes in aquatic ecosystem. In addition, these dyes can be very toxic and even carcinogenic to human and mammalian animals; therefore, the removal of these contaminations from wastewater is essential [1–5]. There are various techniques for dye removal from wastewater such as ozonation [6], electrochemical destruction [7], photodegradation [8], biological methods include aerobic degradation, anaerobic degradation, biosorption, etc [9–12], adsorption [13], coagulation [14], membrane filtration [15], and ion exchange [16] methods. Adsorption process is a simple, low cost, and quite effective method compared to the other mentioned procedures. Since the nanoparticles have high surface area, they can act as an efficient adsorbent for the removal of dyes from wastewater [17]. The removal of organic dyes by using magnetic nanoparticles as adsorbents is an interesting field for wastewater treatment, because the magnetic nanoparticles that adsorbed the organic dye are easily separated from the solution by using a magnet at the end of process [18].

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In recent years, the spinel ferrite nanoparticles have received a lot of attention due to their unique magnetic and electrical properties. These nanoparticles have been used as catalysts for the synthesis of organic compounds \[19\], sensor \[20\], permanent magnet \[21\], wastewater treatment \[22–24\], microwave devices \[25, 26\], electronic devices \[27\], and biomedical agents \[28\]. There are various ways to synthesize these nanoparticles \[29–35\], but plant-mediated synthesis of them has been attracted by chemists, because of their cost-effective, environmental friendly synthesis, milder conditions, feasibility, easier and faster procedure respect to the other chemical and physical methods. In addition, the plant-mediated nanoparticle synthesis is proper for the large scale synthesis of nanoparticles and due to its biocompatibility, it is suitable for the medical applications. In this method, the aqueous extract is used to make nanoparticles at normal temperatures and pressures, thus saving a lot of energy is obtained. The phytoconstituents which existed in the plants such as alkaloids, flavonoids, saponins, steroids, tannins and other nutritional compounds have two essential roles as reducing and stabilizing agents. \[36–38\].

Mindium laevigatum (Vent.) Rech. f. & Schiman-Czeika is one of the useful medicinal plants that belong to Campanulaceae family and it is endemic plant of Iran and Turkey. It is widely used as blood purifier, anti-asthma, anti-dyspnea, and especially as an anti-hemorrhoid drug in traditional medicine \[39, 40\]. The Persian name of the plant is Gholeshekafteh and in Zanjan province, Iran is called Gengedasi. Narnar is its former scientific name: Michauxia laevigata Vent \[40\]. These families have important medicinal activities for treatment of various diseases such as tonsillitis, laryngitis, bronchitis, and warts because of their strong gel and flavonoids that obtained from its seed, flowers and stem gum \[41\]. There have been no reports on the use of Mindium laevigatum in the plant-mediated synthesis of nanoparticles in literature.

Reactive blue 222 (RB 222) is an azo dye and it is used for coloring cotton, wool, silk, polyamide textiles, and ink color \[42\]. The structure and characteristics of RB 222 are shown in scheme 1.

In this research, we discuss the plant-mediated synthesis of Cu0.5Zn0.5Fe2O4 NPs using Mindium laevigatum as an economy, simple and eco-friendly approach and its application as an adsorbent for removal of RB 222 dye. This is the first report on the plant-mediated synthesis of nanoparticles using Mindium leavigatum.
2. Materials and methods

2.1. Materials and methods

RB 222 was obtained from Merck and used without further purification. Cupper (II) acetate tetrahydrate (Cu(OAc)2, 4H2O), zinc chloride (ZnCl2) and iron (III) nitrate nonahydrate (Fe(NO3)3, 9H2O) were purchased from Merck company. UV–vis absorption spectra were prepared on a Metrohm (Analytical Jena–Specord 205) double beam instrument. IR spectra were measured on KBr pellets with a Mattson (Unicam Ltd, Cambridge, UK) 1000 Fourier transform infrared spectrophotometer. The structural properties of Cu0.5Zn0.5Fe2O4 nanoparticles were identified by x-ray powder diffraction (XRD) with a Philips PW1730 x-ray diffractometer using Cu (Kα) radiation (wavelength: 1.5406 Å), operated at 40 kV and 30 mA at room temperature in the range of 2θ from 10 to 80. The particle size and morphology of the sample surfaces was studied by a Tescan Mira III FESEM scanning electron microscope equipped with an energy dispersive x-ray spectrometer (EDX). EDX analysis was performed to further confirmation of the nanoparticles composition. A Philips EM208s–100 Kv transmission electron microscopy (TEM) was used. Atomic force microscopy (AFM) were performed by a Bruker ICON AFM. The magnetic properties of sample were measured at room temperature using vibrating sample magnetometer (VSM, Meghnatis Kavir Khashan Co., Khashan, Iran).

2.2. Preparation of aqueous extract

The stems of Minidium leavigatum were washed, crushed and powdered. For preparation of the extract, 20 grams of the powdered plant material is loaded into the thimble, which is placed inside the soxhlet extractor. Then 300 ml of distilled water is added to a 500 ml round-bottom flask which is attached to a 250 ml soxhlet extractor and a condenser on a heating mantle. The solvent is heated to reflux and the extraction process is continued for 12 h.

2.3. Synthesis of Cu0.5Zn0.5Fe2O4 nanoparticles using minidium leavigatum

35 ml of distilled water, 2 mmol of Fe(NO3)3·9H2O, 0.5 mmol of ZnCl2 and 0.5 mmol of Cu(OAc)2·4H2O were added to a ceramic vessel and the mixture was stirred and heated on a sand bath until 60 °C. Then 15 ml of the extract was added to it and heated in temperature between 60 °C–80 °C for 12 h. The solid residue was heated in an air-heated furnace at 750 °C for 3 h to obtain the Cu0.5Zn0.5Fe2O4 NPs.

2.4. RB 222 adsorption in the presence of Cu0.5Zn0.5Fe2O4 NPs

The dye adsorption experiments were performed by using various amounts of Cu0.5Zn0.5Fe2O4 NPs (20, 30 and 40 mg) in 50 ml solution of RB 222 (20 mg l⁻¹) at the room temperature. The optimum amount of Cu0.5Zn0.5Fe2O4 NPs was 40 mg for removal of RB 222 (20 ppm). Then for investigation of the effect of initial dye concentration, 40 mg of Cu0.5Zn0.5Fe2O4 NPs was added to 50 ml of different concentrations of RB 222 (10, 20, 30 and 40 mg l⁻¹). For studying the progress of the adsorption, 5 ml of solution were removed from the reaction medium at regular time intervals. The nanoparticles were separated from solution using a magnet and the solution was centrifuged. The change on the absorbance at a maximum wavelength (λmax = 612 nm) of dye was monitored by UV–vis spectrophotometer. The adsorption performance of the process was defined as % adsorption = (A0 – A)/A0 × 100, where A0 is the initial absorbance and A is the final absorbance at λmax = 612 nm.

3. Results and discussion

3.1. Characterization of Cu0.5Zn0.5Fe2O4 nanoparticles

IR spectrum was measured by using KBr pellet in the range of 400–4000 cm⁻¹. The IR spectrum of Cu0.5Zn0.5Fe2O4 NPs was shown in figure 1. Based on literature, the IR peaks in the range of 100–1000 cm⁻¹ are related to vibration of ions in solid crystal compounds [43, 44]. Two absorption bands for Fe–O are observed at 566 and 436 cm⁻¹. These two frequencies in the spinel ferrite NPs have been related to the long bond length of metal–oxygen ions in the tetrahedral (Mtetra-O) and shorter bond length of metal–oxygen ions in the octahedral (Moctra-O) positions. The bands at 1546 cm⁻¹ and 1029 cm⁻¹ related to C–O and C–O–C stretching vibrations. The bands at 3442 cm⁻¹ and 1438 cm⁻¹ are related to the O–H stretching and C–O–H bending vibrations, respectively.

The crystal structure analysis was carried out by the x-ray diffraction patterns. The XRD pattern of Cu0.5Zn0.5Fe2O4 NPs was shown in figure 2. The XRD analysis confirmed the cubic structure of the compound. The average crystallite size of nanoparticles was determined by the full width at half maximum of the XRD patterns using the Scherrer formula (D = 0.9λ/βcos θ). In the formula D is the crystallite size (nm), λ is the...
x-ray wavelength of Cu Kα = 0.154 nm, β is the full width at half maximum of the peak and θ is the Bragg angle [45]. By using the formula, the average size of Cu0.5Zn0.5Fe2O4 NPs was 23.5 nm.

In figure 3 the SEM image of the Cu0.5Zn0.5Fe2O4 NPs was shown. An overview of the image of the synthesized compound demonstrated that the sample consist of small nanoparticles. In figure 4, the TEM image of Cu0.5Zn0.5Fe2O4 NPs was shown. The TEM image indicates that the average size of the nanoparticles is about 20 nm.

The AFM images of Cu0.5Zn0.5Fe2O4 NPs was shown in figure 5. The AFM images show that the size of the nanoparticles is about 20 nm. The particle size determined by TEM, and AFM images is in accordance with those of Scherrer’s equation.

EDX was carried out for compositions confirmation of the nanoparticles (figure 6). The EDX showed that Cu0.5Zn0.5Fe2O4 NPs consists of Cu, Zn, Fe and O and no other elements were detected in the sample, thus the synthesized sample has high purity. The weight and atomic fractions of all elementary constituents in the Cu0.5Zn0.5Fe2O4 NPs determined by EDX are presented in table 1. The results of XRD and FT-IR studies are in accordance with EDX analysis.

The magnetic behavior was investigated by VSM for Cu0.5Zn0.5Fe2O4 NPs calcined at 750 °C (figure 7). The specific saturation magnetization (Ms), remanent magnetization (Mr), and coercivity (Hc) values were
43.67 emu g$^{-1}$, 3.56 emu g$^{-1}$, and 22.15 Oe, respectively. The sample exhibited a magnetic property in the presence of a magnetic field.

From the BET results, the pore volume, BET surface area, and average pore diameter for Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs were calculated 0.06847 (cm$^3$/g), 5.0189 m$^2$/g$^{-1}$, and 54.57 nm, respectively.

### 3.2. Effect of adsorbent dosage

The adsorption of various organic dyes such as reactive blue 222, reactive blue 21, reactive yellow 145, reactive red 195, and direct blue 129 by Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs were checked. The results showed that the best adsorption of organic dyes was for reactive blue 222 and the other dyes are not well adsorbed by Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs. The effect of adsorbent dosage on the adsorption of reactive blue 222 by Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs at room temperature was investigated by varying the nanoparticle dosage (20, 30 and 40 mg) at a constant concentration of RB 222 (30 ppm). The figure 8 shows the decolorization of RB 222 (30 ppm) for 45 min by using different amounts of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs (20, 30 and 40 mg). The maximum removal occurred at an adsorbent dose of 40 mg. As
Figure 5. The AFM images of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs.

Figure 6. The EDX micrograph of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs.

Table 1. The elementary constituents of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ powder measured by EDX.

| Elements | Weight percentage (%) | Atomic percentage (%) |
|----------|-----------------------|-----------------------|
| Cu       | 8.80                  | 4.05                  |
| Zn       | 9.13                  | 4.08                  |
| Fe       | 44.52                 | 23.29                 |
| O        | 37.55                 | 68.58                 |
| Total    | 100                   | 100                   |

Figure 7. The Magnetization curve for Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs.
can be shown in figure 8, the removal efficiency of RB 222 is increased by increment of adsorbent dosage. It is because of raising the number of vacant sites toward the RB 222 molecule at constant dye concentration.

### 3.3. Effect of initial dye concentration

The effect of initial concentration of RB 222 (20, 30, and 40 mg l$^{-1}$) in the presence of 0.04 g of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs at room temperature was investigated as shown in figure 9. According to these results, the removal efficiency of RB 222 is decreased by increasing the initial concentration of RB 222.

### 3.4. Adsorption of organic dyes at different time

The effect of contact time on the removal of RB 222 (30 mg l$^{-1}$) in the presence of 0.04 g of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs was studied at room temperature and the results were presented in figure 10. The removal of RB increases with passing the time and reaches over 90% at about 45 min. These results confirmed that the primarily broad accessibility of vacant sites on the Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs surface gradually decreases as the sites are filled up over the time in the adsorption process.

### 3.5. Adsorption isotherms

The adsorption isotherms show the quantity of solute adsorbance per unit weight of adsorbent as a function of concentration in the bulk solution. There are various isotherm models such as Freundlich [46], Langmuir [47], Dubinin-Radushkevich [48], Harkins-Jura and Temkin [49]. The Freundlich isotherm is based on multilayer adsorption on the surface of the adsorbent and is depicted in equation (1).

\[
\text{Ln} q_e = \text{Ln} K_f + \frac{1}{n} \text{Ln} C_e
\]  

where $q_e$ (mg g$^{-1}$) is the amount of RB 222 adsorbed per unit mass of Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs at equilibrium, $K_f$ (mg$^{(1-1/n)}$ l$^{1/n-1}$ g$^{-1}$) is the Freundlich constant, $n$ (g l$^{-1}$) is the heterogeneity factor, and $C_e$ (mg l$^{-1}$) is the equilibrium liquid concentration of RB 222. A plot of Ln$ q_e$ versus Ln$ C_e$ yields a straight line with slope $= 1/n$ and y-intercept $= \text{Ln} K_f$.

The Langmuir equation is based on monolayer adsorption on the adsorbent surface to estimate the maximum adsorption capacity and is depicted in equation (2).
where \( C_e \) (mg l\(^{-1}\)) is the equilibrium liquid concentration of RB 222, \( q_e \) (mg g\(^{-1}\)) is the amount of RB 222 adsorbed per unit mass of Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs at equilibrium, \( K_L \) is the equilibrium constant (1 mg\(^{-1}\)) and \( q_{\text{max}} \) is the amount of adsorbate required to form monolayer (mg g\(^{-1}\)). A plot of \( C_e/q_e \) versus \( C_e \) yields a straight line with slope \( = 1/q_{\text{max}} \) and y-intercept \( = K_L/q_{\text{max}} \).

The Freundlich and Langmuir isotherm plots are shown in figures 11 and 12, respectively for the adsorption of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs. The isotherm parameters for the adsorption of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs are shown in table 2. The correlation values (\( R^2 \)) showed that the Langmuir model yields more suitable than the Freundlich model for the adsorption of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs and thus there is a monolayer adsorption on the surface of adsorbent.

3.6. Kinetic study

The adsorption kinetics of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs was investigated by using the pseudo first-order, the pseudo second-order, and the intraparticle diffusion models. The equations of investigated kinetic models were shown in table 3 [50].

The pseudo first-order, pseudo second-order, and intraparticle diffusion model plots for adsorption of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs were shown in figures 13–15, respectively. The adsorption reached equilibrium after 45 min without remarkable increment in RB 222 adsorption after that time. The kinetic parameters for the models were tabulated in table 4. The correlation values (\( R^2 \)) for the kinetic models showed that pseudo second-order model yields more suitable than the other models for the adsorption of RB 222 on Cu\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) NPs.
Figure 12. The Langmuir isotherm plot for the adsorption of RB 222 on Cu_{0.5}Zn_{0.5}Fe_{2}O_{4} NPs (amount of NPs = 40 mg, C_0 = 20, 50, 60, 70 mg l^{-1}).

Table 2. The isotherm parameters for the adsorption of RB 222 on Cu_{0.5}Zn_{0.5}Fe_{2}O_{4} NPs.

| Model                  | Parameters          | Values              |
|------------------------|---------------------|---------------------|
| Freundlich             | K_f (mg^{(1/n)} l^{-1} g^{-1}) n (g l^{-1}) R^2 | 18.1632 6.9156 0.9410 |
| Langmuir               | q_{max} (mg g^{-1}) K_L (l mg^{-1}) R^2          | 32.26 0.4952 0.9858 |

Table 3. The Kinetic equation models.

| Model           | Equation                                                                 | Parameters                                                                                       |
|-----------------|--------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------|
| Pseudo first-order | Log (q_t - q_e) = Log q_e + \frac{k_1}{q_e} t                       | q_e: the amount of adsorbed per unit mass of adsorbent at equilibrium (mg/g)                   |
| Pseudo second-order | \frac{t}{q_t} = \frac{1}{q_e k_2} + \frac{1}{q_e} \frac{t}{k_2} | q_e: the amount of adsorbed per unit mass of adsorbent at equilibrium (mg/g) k_2: The rate constant of Pseudo second-order adsorption (g/mg.min) |
| Intraparticle diffusion | q_t = k_I \sqrt{t} + 1                                                | 1: boundary layer thickness effect (mg/g), the larger 1, the greater the boundary layer effect k_I: the intraparticle diffusion rate constant (mg.g^{-1}.min^{-0.5}) |

Figure 13. Plot of pseudo first-order equation for adsorption of RB 222 on Cu_{0.5}Zn_{0.5}Fe_{2}O_{4} NPs (amount of NPs = 0.040 g, and C_0 = 30 mg l^{-1}).
4. Conclusion

Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ magnetic nanoparticles have been prepared using Minidium leavigatum extract and appropriate metal salts by a green method. These magnetic nanoparticles have excellent adsorption capacity for removal RB 222 from water. Equilibrium data were investigated by Freundlich and Langmuir models. The results showed that the Langmuir model yields more suitable than the Freundlich model for the adsorption of RB 222 on Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ NPs. The maximum adsorption capacity was determined as 32.26 mg g$^{-1}$.

Adsorption kinetic data for pseudo first-order, pseudo second-order, and intraparticle diffusion models were obtained and the results showed that pseudo second-order kinetic model is more suitable than the other two models.

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ORCID iDs

Ali Reza Kazemizadeh 𝗄 diarr https://orcid.org/0000-0003-3085-5125
Ali Ramazani 𝗄 diarr https://orcid.org/0000-0003-3072-7924
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