Glycine assisted synthesis of $ZnFe_2O_4$ nanoparticles by one pot microwave heating route and organic pollutant adsorption for water treatment

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Abstract

We synthesized magnetic $ZnFe_2O_4$ nanoparticles *via* one pot solvent free microwave assisted technique. Iron (III) nitrate nonahydrate and zinc nitrate hexahydrate as reactants were crushed in the presence of reactive reagents. Then, the prepared mixture was transferred into a domestic microwave oven with the power of 900 W for 20 minutes. Finally, the resulting compound was collected, washed several times with ethanol, dried at 80 °C for 24 h and analyzed. The constructed nano-sized zinc ferrite was characterized using powder x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and energy dispersion of x-ray spectrometry (EDX). This technique is a simple, fast, environmental friendly and can draw a usable viewpoint for the synthesis of nanomaterials. The ability of the prepared product was studied on the removal of organic pollutant (Congo Red) from aqueous solution through adsorption mechanism.

Keywords: Microwave, Glycine, Nanoparticles, Zinc ferrite, Solid state

1. Introduction

Spinel zinc ferrite in nano-sized structure is considered as a practical class of magnetic nanomaterials due to special magnetic, electrical and catalytic properties [1-3]. Whereas these materials are widely employed in the various scientific and industrial fields, their synthesis using facile and low-cost techniques plays a key role in the mass production processes. Up to now, many methods have been used to synthesize magnetic ferrite nanomaterials such as coprecipitation [1, 4], hydrothermal/solvothermal [2, 5], mechanochemical [6, 7], and sol-gel methods [8, 9]. Amongst the synthesis methods, microwave method has attracted a lot of

attention because of simplicity and short time reactions, which are usually performed in solution media [10]. Microwave irradiation as a heating source harnesses the chemical synthesis of nanomaterials by rapid heating rate. However, a few studies on the use of this method for the synthesis of nano-sized materials especially magnetic materials have been reported. In this work, we used this facile way to drive one pot synthesis reaction in solid state for the production of magnetic zinc ferrite nanomaterial. Then, the resulting product was used to evaluate decolorization of Congo red (CR) as a model pollutant from aqueous solution through adsorption mechanism.

2. Experimental

2.1. Initial materials

All of chemicals were purchased from Merck Co. and used without further purification.

2.2. Synthesis method

The nitrate salts of iron and zinc as metal sources with a stoichiometrical amount were mixed to each other in the presence of glycine as a fuel and also organic driving agent. The mixture was transferred to a domestic microwave oven with the power of 900W for 20 min. After the treatment, the product was collected, washed to remove the residual initial materials, dried and analyzed. The structural and morphological study was carried out by using FT-IR, XRD, SEM and EDX analyses.

2.3. Characterizations

The X-ray diffraction (XRD) pattern was recorded by a STOE powder diffraction system using Cu Kα radiation (wavelength, λ = 1.54060 Å). Scanning electron microscopy (SEM) images and energy-dispersive X-ray spectroscopy analysis (EDX) was taken on a Philips XL-30 ESEM with gold coating. Fourier transform infrared (FT-IR) spectrum was recorded on a Shimadzu-8400S spectrometer in the range of 400–4000 cm^{-1} using KBr pellets. The UV-Vis absorption study was performed at room temperature in the wavelength range of the 190–800 nm on a UV-Vis spectrometer (ShimadzuUV-1700).

2.4. Adsorption experiment

Adsorption behavior of CR dye was evaluated in the following conditions: 50 mL of CR aqueous solution with a initial concentration in the range of 10 to 200 mg.L^{-1} was agitated in the presence of the prepared zinc ferrite nanoparticles (0.02 g) as an adsorbent at room temperature for 2 h in the dark. After centrifuging, the concentration of the residual dye was measured by using UV–Vis spectrophotometer at appropriate wavelength corresponding to the maximum absorption of CR (498 nm). The removal efficiency of dye from the solution was calculated by equation(1):

$$\% \text{ Removal efficiency} = \frac{C_0 - C_t}{C_0} \times 100 \quad \text{(Eq. 1)}$$

Whereas, C_0 is the initial concentration of dye and C_t is the final concentration of dye after treatment time, t.

3. Results and discussion

Fig. 1 indicates the FT-IR spectrum of the resulting product, $ZnFe_2O_4$ nanoparticles, after microwave treatment. Due to microwave heating procedure, the organic sections were removed and no absorption band was observed in the FT-IR spectrum of the obtained product. The appeared peaks at 420 and 551 cm^{-1} are related to metal-oxygen vibration frequencies.

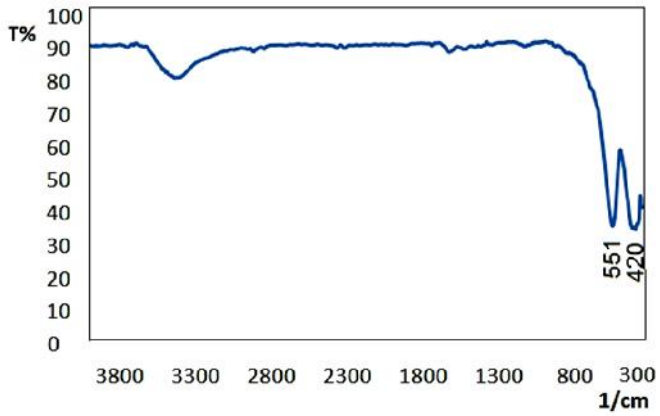

Fig. 1. FT-IR spectrum of the resulting product.

The XRD pattern of the product (Fig. 2) confirms the formation of pure phase of $ZnFe_2O_4$ matching with the crystalline system of cubic phase (ASTM card No. 01-079-1150).

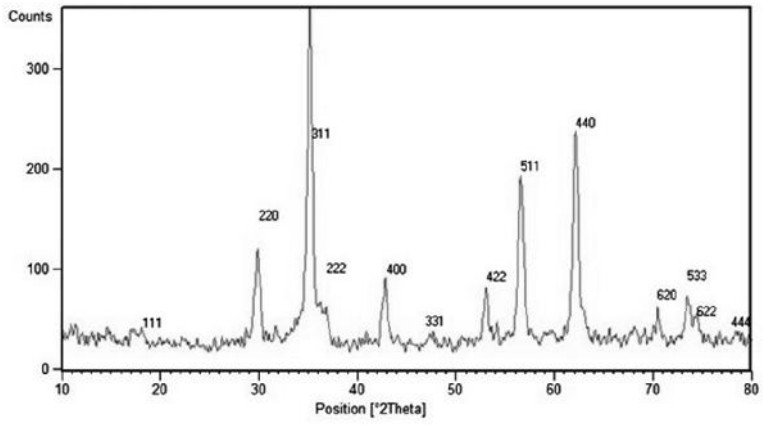

Fig. 2. XRD pattern of prepared magnetic nanopartciles.

FESEM images (Fig. 3) indicate a particulate morphology with average particle size of 32 nm for the synthesized product. The Elemental analysis by EDX revealed the presence of Zn, Fe, O elements (Fig. 4).

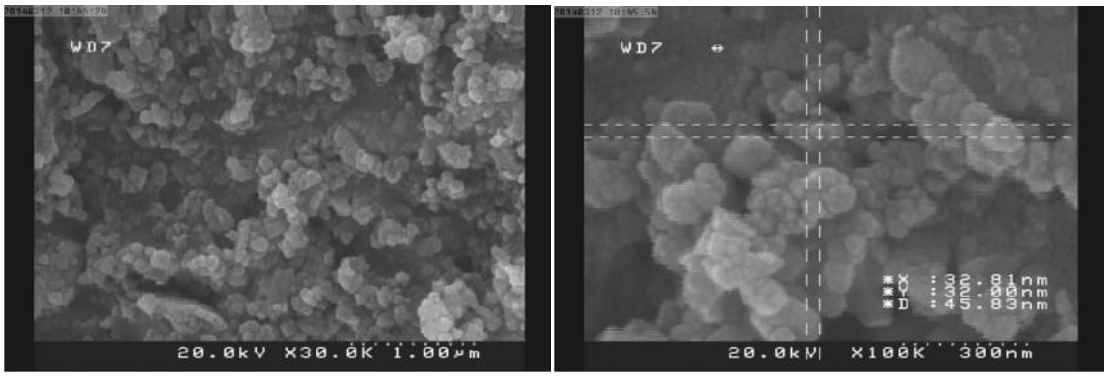

Fig. 3. FESEM images of $ZnFe_2O_4$ nanoparticles.

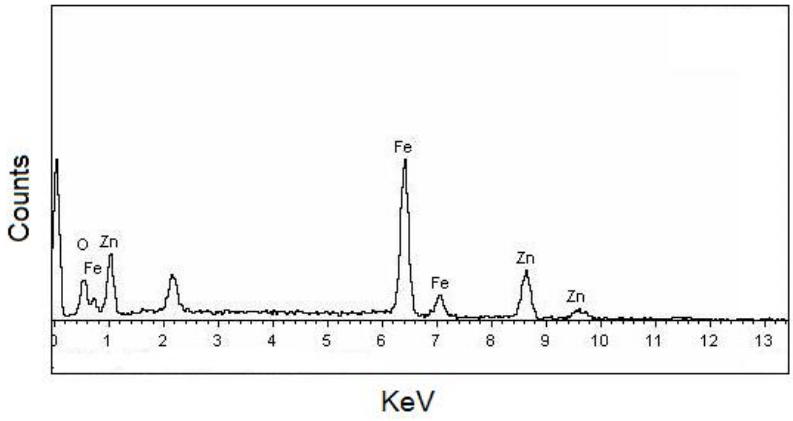

Fig. 4. EDX analysis of product.

The capability of the prepared zinc ferrite nanoparticles for the decolorization of CR from water by adsorption mechanism was studied and adsorption isotherms were determinded by Langmuir and Freundlich models as follow:

$$\frac{C_e}{q_e} = C_e\left(\frac{a_L}{K_L}\right) + \left(\frac{1}{K_L}\right) \quad \text{(Eq. 2)}$$

$$\text{Log } q_e = \text{Log } K_F + \frac{1}{n} \text{ Log } C_e \quad \text{(Eq. 3)}$$

Where, a_L (Lmg^{-1}) and K_L (Lg^{-1}) are the Langmuir constants (Fig. 5a). Meanwhile, K_F ($mg^{1-1/n}\ L^{1/n}\ g^{-1}$) and n are the Freundlich adsorption isotherm constants (Fig. 5b). These adsorption isotherms are employed to explain the interactions between dye molecules and adsorbent. In these equations, C_e is the equilibrium concentration of pollutant in solution (mg L^{-1}), q_e is the amount of dye molecules adsorbed (mg g^{-1}) per unit of adsorbent at equilibrium (mg g^{-1}). Based on a forementioned considerations, it was found that the adsorption process obey very well from Langmuir model ($R^2 > 0.98$ with 99% confidence level) describing the physical maximum adsorption capacity of product.

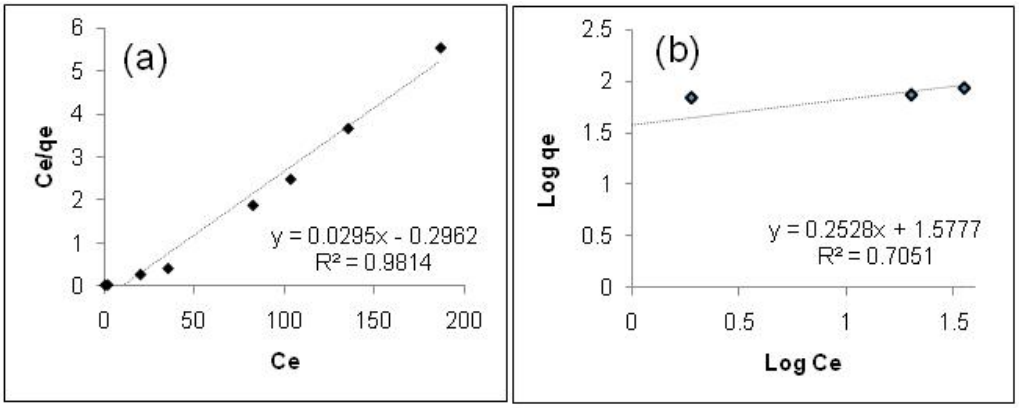

Fig 5. Langmuir (a) and Freundlich (b) isotherm

4. Conclusion

We synthesized the magnetic zinc ferrite nanoparticles by a facile, short time and environmental friendly microwave process. The results revealed the microwave irradiation as a heating source easily treats synthesis reaction of nanomaterials and can draw a useful perspective for the synthesis of nanomaterials. Another aim of this study was to evaluate the adsorption process for CR dye pollutant on the magnetic $ZnFe_2O_4$ nanoparticles. It was concluded that the obtained product can be introduced as a qualified adsorbent with a desirable efficiency to decolorize CR dye from water. Therefore, this specimen can be nominated for further studies.

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