



Application of magnetic ionomer for development of very fast and highly efficient uptake of triazo dye Direct Blue 71 from different water samples



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ABSTRACT

This research focuses on removing Direct Blue 71 (DB 71) from aqueous solution in an efficient and very fast route by ionic liquid mediated γ -Fe₂O₃ magnetic ionomer. 2-hydroxyethylammonium sulphonate immobilized on γ -Fe₂O₃ nanoparticles (γ -Fe₂O₃-2-HEAS) was used for this purpose. The influence of shaking time, medium pH, the concentration of sorbent and NaNO₃ on removal was evaluated to greatly influence removal extent. The optimal removal conditions were determined by response surface methodology based on the four-variable central composite design to obtain maximum removal efficiency and determine the significance and interaction effect of the variables on the removal of target triazo dye. The results have shown that an amount of 98.2% as % removal under the optimum conditions. The adsorption kinetics and isotherms were well fitted to a pseudo-second order model and Freundlich model, respectively. Based on these models, the maximum dye adsorption capacity (Q_m) of 47.60 mg g⁻¹ was obtained. Finally, the proposed nano-adsorbent was applied satisfactorily for removal of target triazo dye from different water samples.

1. Introduction

Dyes are basically natural or synthetic organic compounds that applied to color the products of various industries such as paper, plastic, rubber, textile, cosmetics, pharmaceuticals and food. However, the results of this usage in the industry are generating a large amount of colored wastewater. This is one of the main sources of vigorous pollution problems worldwide (Cervantes and Dos Santos, 2011; Maljaei et al., 2009). Since most of the dyes are complex organic molecules and are stable to light and not biologically degradable, therefore, wastewater effluents contain dyes especially synthetic dyes which may cause a potential hazard to the environment. Some of these dyes are highly toxic and have mutagenic or carcinogenic activity (Caliman and Gavrilescu, 2009; Gavrilescu, 2009). In addition, the loss of dyes to the environment may show eco-toxic hazards and introduce the potential danger of bioaccumulation that may finally affect human by transport through the food chain (Dos Santos et al., 2006).

The azo dyes are one of the most widely used of the synthetic dyes, which are usually specified by the presence of one or more azo groups. The presence of large degree of aromatic compounds such as anthracene, benzene, toluene, xylene, and etc. in the dye molecule and the stability of dyes, cause that the dyes are difficult to destroy or

decompose by common treatment in conventional wastewater treatment methods (Baughman and Weber, 1994; O'Neill et al., 2000). Hence, development of suitable treatment methods to efficiently and effectively treat the effluents discharged to the environment especially water resources to agree with environmental legislation restricting and prevent deterioration of ecosystems is very important.

Different physico-chemical processes such as precipitation, coagulation, flocculation, membrane filtration, electrochemical treatment, ion exchange and solvent extraction have been applied for the treatment of colored wastewater. Some of these methods suffering from many restrictions such as incomplete ion removal need to high cost, high-energy requirements, and production of toxic sludge or other waste products. Among the above-mentioned methods, adsorption is preferred to other techniques because of low cost, simple design and easy operation, viability, and high quality of the treated effluents particularly for sorption processes (Arami et al., 2006).

A wide range of materials has been employed as potential candidates for the adsorptive removal of azo dyes. Activated carbon, zeolites, and graphene based materials have exhibited high affinities and rapid kinetics to remove of azo dye (Cheng et al., 2017a, 2017b; Liu et al., 2012; Saygili and Güzel, 2016; Saygili et al., 2015). Despite such positive features exhibited via the composites mentioned above, high cost

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for synthesizing high-quality products with uniformity and regeneration of the spent sorbents is one of the major reasons that cause to prevent using them as the attractive alternative removing azo dyes for large-scale application. To overcome this issue, functionalized ionic liquids have received considerable attention in dye removal due to their high thermal stabilities, ionic conductivities, and possession of functionalities that are capable of interacting with the target compounds (Gao et al., 2013a, 2013b; Pei et al., 2007). Ionic liquids (ILs) are considered as highly efficient eco-friendly solvents for liquid–liquid extraction (Ferreira et al., 2014; Gharehbaghi and Shemirani, 2012) and adsorption (supported ionic liquids) (Lu et al., 2017; Zarezadeh-Mehrizi et al., 2013; Zhang et al., 2016; Zhao et al., 2015) of organic dyes and heavy metals due to the presence of strong electrostatic attractions from the polar functionalities, ease of operation, tunable and high solubility, etc. (Goossens et al., 2016; Mahadevi and Sastry, 2013).

Nanoparticles have distinguished properties such as a high number of reactive atoms, high mechanical and thermal strength, highly ordered structure and a large number of vacant reactive surface sites in addition to metallic or semi-metallic behavior applied for removal of various toxic materials (Ghaedi et al., 2012, 2013). Magnetic nanoparticles (MNPs) are popular in terms of their high reactivity, abundance, low cost, ease of separation, reusability, and environmental friendliness (Das et al., 2017). Magnetic separation of MNPs is easy, economical, and saves time and energy. Moreover, MNPs such as γ -Fe₂O₃ have excellent thermal stability against degradation, large high surface area, surface modification ability, ease of synthesis and low toxicity and cost (Ding et al., 2006; Wilczewska and Misztalewska, 2014). The aforementioned advantages of γ -Fe₂O₃ MNPs have made it as a promising support for ionic liquids (ILs) (Zhang and Xia, 2009).

In the present work, 2-hydroxyethylammonium sulphonate immobilized on γ -Fe₂O₃ (γ -Fe₂O₃-2-HEAS) with a very fast and highly efficient response was applied as a new supported IL for removal of DB 71 as model azo dyes to determine the effectiveness of this nanomaterial on the removal of dyes from aqueous solution. Influence of important variables (shaking time, pH, the concentration of sorbent and NaNO₃) were investigated and optimized by central composite design (CCD) combined with response surface methodology (RSM) using the desirability function (DF) as maximizing criterion of the response. The results obtained from the presented models were compared with the experimental values. The kinetics and thermodynamics of the adsorption and regeneration of the adsorbent have also been investigated.

2. Experimental

2.1. Reagents and solutions

All chemicals and reagents used for experiments and analyses were of analytical grades and purchased from Merck (Darmstadt, Germany) or Aldrich (Chemical Co., Milwaukee, WI, USA). Direct Blue 71 (DB71) used in this study was purchased from Aldrich. This azo dye was used without further purification. A stock solution of the dye (1000 mg L⁻¹) was prepared by adding the appropriate amount of dye powder in deionized water. Deionized water was used throughout the experiments. γ -Fe₂O₃-2-HEAS was synthesized according to the procedure that we have reported in our recent paper (Khani et al., 2016a).

2.2. Instrumentation and software

A Perkin Elmer (Lambda 25, www.perkinelmer.com) spectrophotometer with 10 mm quartz cells was used for UV–Vis spectra acquisition. A Universal 320 R refrigerated centrifuge equipped with an angle rotor (6-place, 9000 rpm, Cat. No. 1620A) was from Hettich (Kirchlengern, Germany). A Metrohm digital pH-meter (model 692, Herisau, Switzerland) equipped with a glass combination electrode was applied for adjusting pH of the under study solutions.

The Design-Expert, a statistical package software version 9.0

(StatEase Inc., Minneapolis, MN, USA) was used for experimental design analysis and subsequent regression analysis of the experimental data.

2.3. Adsorption experiment

A 10 mL vessel containing known initial concentration 10 – 60 $\mu\text{g mL}^{-1}$ of DB71 for the adsorption of DB71 by γ -Fe₂O₃-2-HEAS was used. The pH of this solution was adjusted to 3.50 using HCl or NH₃ solution then 10.93 mg of the nanoadsorbent was added and the solution was shaken for 80 s to facilitate adsorption of the target dye on the nanoparticles. Finally, by the application of an external field, the magnetic nanoadsorbent was collected at the bottom of the tube and the DB71 content of the solution was determined by UV–Vis spectrophotometer. All adsorption experiments were repeated two times, and the average values of measurements were reported. The amount of DB71 adsorbed onto the γ -Fe₂O₃-2-HEAS nanoadsorbent was determined according to the following Eq. (1):

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

where C_0 was the adsorbate initial concentration (mg L^{-1}), C_e was the adsorbate equilibrium concentration (mg L^{-1}), V was the liquid volume (L), and W was the dosage of the solid adsorbent (g), respectively.

3. Results and discussion

3.1. Physicochemical properties of the adsorbent

FT-IR spectra of γ -Fe₂O₃ and γ -Fe₂O₃-2-HEAS were shown in Fig. S1 (See supporting information). Broad bands at around 450–600 cm^{-1} in the FT-IR spectra of γ -Fe₂O₃ and γ -Fe₂O₃-2-HEAS attributed to the Fe–O vibration (Zhang et al., 2011). Typical bands at 2960, 2933 and 2866 cm^{-1} in the FT-IR spectrum of γ -Fe₂O₃-2-HEAS were related to the C–H stretching vibrations of alkyl chains. S=O stretching vibrations in the sulphonate functional groups occurred at 1111 cm^{-1} . N–H bending vibrations in the ammonium groups were observed at 1600, 1502 cm^{-1} . These results indicated that IL was successfully immobilized on γ -Fe₂O₃ MNPs.

In the XRD pattern of the synthesized γ -Fe₂O₃ MNPs (Fig. S2), diffraction patterns had a good agreement with the cubic structure of maghemite (JCPDS file No 04–0755), a unit cell dimension of 8.35 Å and the space group of P4132 (213). The average crystallite size was calculated to be 13.1 nm using Scherrer equation in which $K = 0.9$ and $k = 0.154$ nm.

Surface area and mean pore diameter of γ -Fe₂O₃ were 91 $\text{m}^2 \text{g}^{-1}$ and 14.3 nm, respectively, according to the Brunauer Emmet-Teller (BET) method. For γ -Fe₂O₃-2-HEAS, surface area reduced to 67 $\text{m}^2 \text{g}^{-1}$ and mean pore diameter increased to 18.2 nm. These observations confirmed the immobilization of IL on the surface of γ -Fe₂O₃.

The particle size distribution of γ -Fe₂O₃-2-HEAS was evaluated using transmission electron microscopy (TEM) (Fig. S3) and showed that the mean diameter of MNPs is 14 nm. In addition, the magnetization curves of γ -Fe₂O₃ and γ -Fe₂O₃-2-HEAS were measured at room temperature using a vibrating sample magnetometer (VSM) (Fig. S4). There was no reduced remanence and coercivity that indicates both unmodified γ -Fe₂O₃ and γ -Fe₂O₃-2-HEAS were superparamagnetic. The saturation magnetizations of γ -Fe₂O₃ and γ -Fe₂O₃-2-HEAS were 68.9 and 63.4 emu g^{-1} , respectively. A slight decrease of the saturation magnetization of γ -Fe₂O₃-2-HEAS was due to the immobilization of IL on the surface of γ -Fe₂O₃ MNPs.

The thermogravimetric analysis (TGA) of adsorbent (γ -Fe₂O₃-2-HEAS) was done to determine the thermal stability (Fig. S5). TGA curve of γ -Fe₂O₃-2-HEAS showed the weight loss around 180 °C, which was related to the adsorbed water molecules on the support. The organic parts were decomposed completely at 487 °C. According to the TGA

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