



Functionalization of magnetic chitosan with graphene oxide for removal of cationic and anionic dyes from aqueous solution



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ABSTRACT

In the present study, we decorated chitosan (©) with Fe₃O₄ nanoparticles followed by cross-linking with GO to prepare Fe₃O₄ supported chitosan-graphene oxide composite (Fe₃O₄©-GO). Different properties of synthesized material were investigated by SEM, XRD, FTIR, TGA and EDX. Batch adsorption experiments were performed to remove toxic cationic and anionic dyes from industrial wastewater. To maximize removal efficiency of composite material, effect of pH (4–12), time (0–80 min), Fe₃O₄©-GO dosage (2–10 mg), initial dye concentration (2–30 µg mL⁻¹) and temperature (303, 313, and 323 K) were studied. The uptake of dyes presented relatively fast adsorption kinetics with pseudo-second-order equation as the best fitting model. To understand the interaction of dye with adsorbent, Langmuir and Freundlich isotherm were applied. Thermodynamic studies were conducted to calculate the changes in free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0). In view of practical application, the influence of ionic strength, recycling as well as investigations based on percent recoveries from spiked real water samples were also taken into account.

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1. Introduction

Environmental issues have become a major focus of both political as well as scientific attention in 21st century, also termed as the “Century of Environment”, (Khamparia, Jaspal, & Malviya, 2015; Perreault, de Faria, & Elimelech, 2015). Disruption of ecosystems due to urbanization, deforestation, unsuitable agricultural practices and pollution are among the key factors undermining the

environment’s capacity to deliver ecosystem services such as clean water (“Water for a sustainable World; The United Nations World Water Development Report”, 2015). In modern era, textile industries hold crucial position in global trade arena (Dasgupta, Sikder, Chakraborty, Curcio, & Drioli, 2015). However, it is reported that approximately 20% of global water pollution is caused by these industries thus presenting a serious threat to the health and environment (Ghaly, Ananthashankar, Alhattab, & Ramakrishnan, 2014; Gul et al., 2016; Scott, 2015).

Over the years, environmental sustainability perspectives have augmented the interest of scientific community in exploiting natural materials (Fan, Luo, Li et al., 2012; Maity & Ray, 2014). In this respect, chitosan (©), a deacetylated derivative of chitin, has been extensively utilized. (Huang et al., 2013; Tanhaei, Ayati, Bamoharram, Lahtinen, & Sillanpää, 2015). Chitin has been ranked as world’s second most important natural polysaccharide mainly

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obtained from shrimp and crabs (Rinaudo, 2006). Until today, © has been widely exploited for environmental remediation due to high functional groups content, i.e. amino group, acetamido group, both primary hydroxyl and secondary hydroxyl groups located at C-2, C-3 and C-6 positions, respectively (Terzopoulou, Kyzas, & Bikiaris, 2015). Among other desirable features are; intrinsic physico-chemical characteristics, chemical stability, high reactivity and exceptional chelating ability (Tanhaei et al., 2015). In addition, abundance, non-toxicity, biodegradability, biocompatible nature and relatively low cost of application makes it an ideal candidate for use in wastewater treatment (Harris & Hajela, 2015; Takeshita & Yoda, 2015). However, there is still a long way to go in designing and exploring novel adsorbents with excellent adsorption capacity and separation rate (Liu, Hu, Fang, Zhang, & Zhang, 2008). Unfortunately, pure © suffers from several drawbacks such as unsatisfactory mechanical properties, excessive shrinkage, deformation when dried, solubility in acidic conditions and compressibility at high operating pressure (Fan, Luo, Li et al., 2012; Guijuan et al., 2012). Also, © being cationic fails to effectively adsorb cationic dyes due to mutual repulsion (Wang, Zhang, & Wang, 2011). Although, it is suitable for removing anionic dyes but textile effluents are usually very complicated containing a blend of cationic and anionic dyes. To perform equally well in dealing with such mixtures, it is important to bring about some modifications in the basic structure of © (Jiang et al., 2011; Yang, Yang, et al., 2013). These drawbacks can be tackled via: (i) cross-linking to stabilize © in acid environment and enhance its mechanical properties, (ii) decorating iron oxide nanoparticles (Fe_3O_4) to ensure magnetic separation and enhanced removal efficiency (iii) improving the inherent properties of © by fabricating its composites via functionalization with other promising materials (Thin et al., 2013; Travlou, Kyzas, Lazaridis, & Delianni, 2013).

Previous studies have revealed that incorporating nano-sized features into © network facilitates magnetic separation thus ensuring easy and quick recovery of used © from treated solution (Roosen, Spooen, & Binnemans, 2014). In addition, material with nano-scale domain increases the surface area and reduces the adsorbent's dosage required (Elwakeel, 2009; Fan, Luo, Li et al., 2012; Fan, Luo, Sun et al., 2012).

Graphene (G), initially synthesized in 1855, represents the most extreme case of high-surface area materials (Sanchez, Jachak, Hurt, & Kane, 2011). Unfortunately, the hydrophilic nature, strongly restacking and agglomeration trend observed in G has severely restricted its widespread usage (Pumera, 2011). Graphene oxide (GO) is a highly oxidized form of graphene showing high density of carboxyl, hydroxyl, carbonyl, and epoxy groups. (Chowdhury & Balasubramanian, 2014; Compton & Nguyen, 2010). Amongst many salient features, its hydrophilic nature, high surface area and functional group density permit a wide variety of chemical functionalizations over GO sheets thus offer incredible prospects in wastewater treatment (Algothmi, Bandaru, Yu, Shapter, & Ellis, 2013; Perreault et al., 2015).

The functionalization of © can be achieved via cross-linking its amino groups to the epoxy groups present in GO (Shao et al., 2013) and the resulting composite offers tremendous potential as biosorbent (Fan, Luo, Sun et al., 2012). In particular, these composites exhibit excellent removal efficiency for cationic and anionic dyes simultaneously because of the new effective p–p stacking interactions taking place between dye molecules and GO sheets (Yang, Bai et al., 2013). It is important to mention that adsorption phenomenon in composite materials is not a cumulative situation. In case of graphene-based materials, there is a drastic increase in porosity, surface area and possibility for the diffusion of pollutants inside GO pores. Therefore, incorporating GO into © skeleton can bring about significant modification in adsorption capabilities



Fig. 1. Digital photographs of (a) MV, (b) AY, (c) ©, (d) GO.

(Terzopoulou et al., 2015) as well as other attributes of the resulting composite (Table 1).

Embedded in aforementioned perspective, we have synthesized Fe_3O_4 ©-GO by impregnating magnetic nanoparticles into © matrix and later functionalization with GO as shown in Scheme 1. The novelty of current study involves following aspects. (i) Since, textile effluents are complex mixtures, so we picked up both cationic and anionic dyes, namely Methyl violet (MV) and Alizarin yellow R (AY), respectively. (ii) The effect of ionic strength on removal efficiency of synthesized composite was studied, keeping in view that textile effluents are rich in salts. To ensure practical applicability, recyclability tests were conducted to ensure that Fe_3O_4 ©-GO remains fairly stable after successive cycle of reuse. In particular, we considered the clean-up of spiked real water samples as to the best of our knowledge very little work has been reported on the treatment of dye laden environmental samples using Fe_3O_4 ©-GO.

2. Experimental

2.1. Materials

All the reagents and chemical used were of analytical grade. GO (Fig. 1d) was prepared from graphite powder (50 mesh, molecular weight 12.01 g mol^{-1}), purchased from AVONCHEM, UK. Powdered © (78% DD, Fig. 1c) extracted from crab shells was supplied by Primex Ingredients (Norway). Glutaraldehyde, hydrogen peroxide, potassium permanganate, sodium nitrate, ammonium ferrous sulfate and ammonium ferric sulfate were obtained from Sigma–Aldrich. Acetonitrile, acetone, HCl and H_2SO_4 were purchased from Merck, Darmstadt, Germany. The commercially available textile dyes used in this work are Methyl violet (MV) and Alizarin yellow R (AY). The physicochemical properties of selected dyes have been summarized in Table 2. Deionized (DI) water was used throughout the study.

2.2. Synthesis of Fe_3O_4 ©-GO

Materials synthesized are: (i) GO, (ii) cross-linked © followed by Fe_3O_4 impregnation (Fe_3O_4 ©) and finally (iii) Fe_3O_4 ©-GO from precursors, i.e. Fe_3O_4 © and GO (Scheme 1).

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