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Materials Research Bulletin

Preparation and characterization of highly hydrophobic magnetic polyaniline nanocomposite for fast and efficient separation of diesel oil from seawater

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ARTICLE INFO

Article history: Received 17 March 2016 Received in revised form 20 July 2016 Accepted 21 July 2016 Available online 25 July 2016

Keywords:

A. Magnetic materials B. Chemical synthesis C. Electron microscopy C. X-ray diffraction

D. Thermodynamic properties

ABSTRACT

In this work, hydrophobic magnetic polyaniline nanocomposite (PAni/Fe₃O₄/C) was prepared by insitu emulsion polymerization of aniline in the presence of magnetic carbon. Magnetic nanocomposites were characterized by FT-IR spectra, X- ray diffraction (XRD), scanning electron microscope (SEM) and transmission electron microscope (TEM). Brunauer-Emmett-Teller (BET) method was utilized to measure specific surface area and pore size distribution of magnetic nanocomposites. The water contact angle values for both magnetic nanocomposites indicated high hydrophobic properties for PAni/Fe₃O₄/C. PAni/Fe₃O₄/C was evaluated for removal of diesel oil from seawater by magnetic separation. Separation capacity (g/g) was governed by separation time, magnetic nanocomposite dose as well as initial oil concentration. Kinetic studies showed that oil separation process followed pseudo-second-order model. Thermodynamics studies of oil sorption process were investigated at temperature range (283 K–323 K) and indicated endothermic, spontaneous and random behavior. The reusability of magnetic polyaniline sorbent was carried out for ten cycles.

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1. Introduction

Oils can cause environmental pollution during production, transportation, storage, refining and usage [1]. Oil spills resulted in great damage to the coastal environment mainly in sensitive marine ecosystems and also caused negative economical impacts on tourism and fisheries [2]. The conventional methods used to separate oil from water include oil containment booms, mechanical extraction [3], chemical dispersants [4], membranes [5], bioremediation [6], absorbent materials [7], in situ burning [8], etc and large number of natural and synthetic absorbent materials have been widely employed such as activated carbon [9], wool fibers [10], zeolites [9,11], straw [7,11] and fly ash [12].

Polyaniline is the most characterized and important among conducting polymers family due to its superior properties and wide applications in its micro- and nanostructures. Polyaniline (PAni) has attracted great interest for its doping/dedoping mechanism, environmental stability and tuneable conductivity [13]. Superhydrophobic materials have high water repellency and their water contact angle measurements are higher than 150° [14],

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http://dx.doi.org/10.1016/j.materresbull.2016.07.022 0025-5408/© 2016 Elsevier Ltd. All rights reserved. these materials have wide applications in prevention of snow adhesion, self cleaning traffic indicators, metal refining and stain-resistant textiles [15].

Synthesis of conducting polymers with a superhydrophobic properties has become an interesting object in materials science. Concerning preparation of superhydrophobic conducting polymers, literature has mentioned few researches. Among these studies, superhydrophobic conducting polymers were applied successfully in corrosion prevention coatings [16,17].

Carbon nanoadsorbents are characterized by their high adsorption capacity, easy regeneration and fast adsorption kinetics [18]. So, they can be applied in removing various pollutants, including heavy metals, organic dyes and antibiotics [19].

Synthesis of magnetic carbon nanocomposites is of great importance and achieved efficient separation and recycling for carbon adsorbents [20]. The effective separation of heavy metals and organic pollutants by magnetic graphene and magnetic carbon nanotubes has been reported [21,22]. Among all kinds of magnetic nanoparticles, magnetite (Fe₃O₄) nanoparticles are of special interest due to their unique magnetic properties and easier synthesizing process [23].

On the other hand, the preparation of magnetic polyaniline has been widely reported and the most common method for synthesizing magnetic polyaniline nanocomposites is chemical oxidation polymerization. Electromagnetic polyaniline nanofibers containing Fe_3O_4 nanoparticles were synthesized using a template-free method [24]. PAni/Fe₃O₄ nanocomposite with a coreshell structure was obtained by in situ polymerization of aniline monomer in the presence of Fe_3O_4 nanoparticles and a surfactant [25]. Also, another route to synthesize well-dispersed PAni/Fe₃O₄ nanoparticles was reported [26]. The application of magnetic polymers for oil separation is few listed [27–30].

The search for better sorption materials which have high hydrophobicity, high uptake capacity, and high rate of uptake is necessary, so, this work aimed to prepare $Fe_3O_4/C/hydrophobic$ PAni hybride nanocomposite. To the best of our knowledge, PAni was not applied before in the field of oil separation. The hybride nanocomposite is characterized by FT-IR spectroscopy, SEM, TEM and XRD techniques. The oil/water separation experiment, kinetics, thermodynamics and reusability are studied.

2. Materials and experimental methods

2.1. Materials

Glucose, ferrous chloride tetrahydrate, ferric chloride hexahydrate, aniline (Ani), sodium dodecyl benzene sulphonate (SDBS) and ammonium persulphate (APS) were purchased from Aldrich. Anhydrous sodium sulphate, hydrochloric acid, sodium chloride and carbon tetrachloride were obtained as Sigma products. Oil used in this study is diesel oil and it was purchased from the market. Other chemicals are of analytical grade and were used without further purification. Double distilled water was used for all preparations.

2.2. Preparation of Fe₃O₄/C nanocomposite

Fe₃O₄/C nanocomposite was prepared by hydrothermal method [31]. In a typical experiment: definite weights of FeCl₂·4H₂O and FeCl₃·6H₂O were mixed with constant molar ratio of 1:1.5. The pH of the solution was raised to 12 with NaOH aqueous solution. A known weight of glucose was added and the mixture was stirred for 30 min. The mixture was transferred to a Teflon lined stainless steel autoclave and heated to 160 °C for 6 h. The reaction was cooled to room temperature and the Fe₃O₄/C nanocomposite was collected by magnetic separation. The obtained nanocomposite was washed several times with distilled water and dried overnight under vacuum at 80 °C.

2.3. Synthesis of PAni/Fe₃O₄/C nanocomposite

The magnetic polyaniline was prepared as follows:- 300 mg of Fe_3O_4/C nanocomposite was dispersed in 50 mL deionized water and sonicated for 20 min. 0.01 mol of aniline in 50 mL 1 M HCl and 0.5 g SDBS were added with magnetic stirring for 10 min at room temperature. 0.01 mol APS in 20 mL 1 M HCl was added to the above mixture. The resulting solution was stirred for 30 min to ensure complete mixing and then the reaction was allowed to proceed without agitation for 12 h at room temperature. Finally, the product was filtered, washed with distilled water and methanol for several times until the filtrate became colourless, and then dried at 70 °C in vacuum oven for 24 h.

2.4. Characterization

2.4.1. Fourier transform infrared spectra (FT-IR)

The chemical structures of magnetic carbon and magnetic polyaniline nanocomposites were confirmed by Jasco 3400 FT-IR spectrophotometer, in the range of 4000–400 cm⁻¹. Traces of

powdered samples were mixed with a definite weight of KBr and pressed into pellets.

2.4.2. Powder x-ray diffraction (XRD)

The structures of the prepared magnetic nanocomposites were ascertained by recording their patterns using EMPYREAN diffractometer in the range of $2\Theta = 10-80^\circ$. The instrument operates at 45 kV with Cu K α ($\lambda = 1.5405 A^\circ$) radiation.

2.4.3. Transmission electron microscope (TEM)

TEM images of the magnetic nanocomposites suspended solutions were taken by high- resolution transmission electron microscopy (TEM, JEM-1230, JEOL Ltd., Japan) with an accelerating voltage of 100 kV.

2.4.4. Scanning electron microscope (SEM)

In SEM, an electron beam was passed through the specimens which scattered them back as electrons and secondary electrons. Back scattered secondary electrons were used to form the image on the computer monitor. The acceleration of the electron beam was 10 kV.

2.4.5. Water contact angle (CA) measurement

The water contact angle measurement was performed by contact angle instrument of type KYDWR, Japan. This test was carried out by pressing the nanocomposites into discs of 2 mm in thickness and 1 cm in diameter. A distilled water droplet was used as the indicator.

2.4.6. Specific surface area and pore size distribution measurements

Brunauer-Emmett-Teller (BET) [32] method was used for estimation of specific surface area and pore size distribution for magnetic carbon and magnetic polyaniline nanocomposites. Nitrogen gas was applied as model gas and adsorption/desorption of N₂ gas is considered as the main process on which surface area and pore size distribution values can be calculated. This operation was carried out at temperature 77 K and before measurement, the investigated materials were degased by heating at 353 K to expel gases and contaminants from the sample. The measurements were made on Quantachrome Nova 2000, U. S. A analyser.

2.5. Oil sorption experiment

In this study, diesel oil was employed as a model oil and its chemical composition was represented in Table 1. As represented in Fig. 1, the oil removal test was carried out as follows: Firstly, 3 g of diesel oil was poured into 150 mL beaker containing 100 mL of artificial seawater (35 g NaCl/IL distilled water). 0.1 g of magnetic polyaniline nanocomposite was added to oil/water surface. The mixture was left for 20 min at room temperature. The nano-composite/oil was removed by magnetic separation using magnet bar. Finally, the remained amount of oil in water was determined by FT-IR spectrophotometer of type A-4600 according to IS 3025 (part 39) [33] and IS 10500 methods [34]. Typically, Spectra were acquired over the range 3200–2700 cm⁻¹ at 4 cm⁻¹ resolution with

Chemical composition of diesel oil.

Component	Amount (µg/g)
Normal alkanes	190,763
Branched alkanes	368,404
Saturated cycloalkanes	52,798
Naphthalenes	1793
Alkylated naphthalenes	25,302
Alkyl benzenes	44,796

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