Journal of the Association of Arab Universities for Basic and Applied Sciences (2016) **20**, 26–31

University of Bahrain

Journal of the Association of Arab Universities for Basic and Applied Sciences

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www.sciencedirect.com

ORIGINAL ARTICLE

Adsorption of textile dye onto modified immobilized activated alumina

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Received 24 July 2014; revised 13 September 2014; accepted 27 October 2014
Available online 15 November 2014

KEYWORDS

Adsorption;
Immobilized alumina;
Textile dyes;
Reactive dye;
Isotherms

Abstract The study describes the synthesis of modified immobilized activated alumina (MIAA) and its application for the removal of textile dye from aqueous media. Immobilization was carried out by using the sol–gel method while modifications were made during the synthesis by adding powder activated alumina. Batch adsorption experiments were carried out at 20 ± 1 °C to see the effect of different parameters like contact time, stirring rate, initial concentration of the dye and dose of MIAA. The removal efficiency of Cibacron reactive yellow dye with an initial concentration of 400 mg/L was greater than 90% for 90 min contact time. Langmuir and Freundlich adsorption isotherms were applied which fitted the data with an R^2 value of 0.99. The maximum adsorption capacity of MIAA was 25 mg/g at the initial dye concentration of 400 mg/L. MIAA can be regenerated thermally and chemically with the dye removal efficiency remained above 85% during the first 4 regeneration cycles. Thermal regeneration was achieved in a muffle furnace at 450 °C while chemical regeneration was done by immersing MIAA in 0.1 M NaOH solution for 2 h. MIAA also proved effective for the adsorption of dyes from actual textile wastewater giving a removal efficiency of 75%.

1. Introduction

Textile wastewater is an amalgam of pollutants but mainly it is characterized by high levels of Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), dissolved solids and colors (Naik et al., 2013; Ghaly et al., 2014). During the dyeing operation a significant amount of dyes remain unfixed on the fabric and they are directly discharged into the water bodies in South Asian countries. Most of the textile dyes are resistant to photodegradation, oxidizing agents and biodegradation (Ventura and Marin, 2013). If the textile wastewater is not treated properly they tend to pose serious threats to the environment (Ratna and Padhi, 2012). Colored water is esthetically unbearable and the dyes prevent the sunlight to penetrate through the water which inhibits the photosynthetic activity affecting the aquatic life (Ghaly et al., 2014).

A number of conventional methods are present for textile wastewater treatment generally classified as biological, chemical and physical treatments (Naveed et al., 2006). The current study focuses on the adsorption technique as it is an effective

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Peer review under responsibility of University of Bahrain.

http://dx.doi.org/10.1016/j.jaubas.2014.10.001
1815-3852

and inexpensive treatment option. The adsorption process can be carried out by using various inexpensive and renewable adsorbents making the treatment even more inexpensive (Kumar and Sivanesan, 2007). The most perfect adsorbent used for color removal is an activated carbon (Gupta et al., 2011) but it is expensive and its regeneration is time consuming. Additionally powdered activated carbon requires filtration and centrifugation for its separation after use (Issa and Degs, 2009). Therefore, the researchers have focused their attentions to find out low-cost adsorbents for the removal of dyes from water. In this regard, agricultural waste materials like sugarcane bagasse (Sadaf et al., 2014; Noreen and Bhatti, 2014), peanut hulls (Nawaz et al., 2014), peanut husk Sadaf and Bhatti (2014), bottom ash and de oiled soya (Mittal et al., 2008), and corn stalk (Fathi et al., 2015) have been tried successfully. Similarly, the researchers have also synthesized adsorbents and tested for the removal of dyes (El-Bindary et al., 2014: Fu et al., 2015). Although, the adsorbents used in these studies exhibited excellent adsorption potential the procedures involved time consuming steps.

Powdered activated alumina is another efficient adsorbent but it too requires laborious separation processes after adsorption in water. In a study, powdered activated alumina has effectively been used for the removal of Alizarin Red (textile) dyes from the water sample (Rehman et al., 2011).

The present study was designed to immobilize activated alumina adsorbent in order to facilitate its separation after use. The aim of the research was to prepare an immobilized adsorbent in the form of granules that could easily be separated from water without undergoing filtration and centrifugation processes. The sol–gel method has been adopted (Buelna and Lin, 1999) to prepare immobilized activated alumina granules in order to meet the objectives.

The adsorption efficiency of the modified immobilized activated alumina (MIAA) was tested by undertaking a set of experiments using Cibacron reactive yellow (a textile dye) as an adsorbate. Fig. 1 shows the structure of the dye. Reactive dyes are applied on cotton fabric and they form covalent bond upon fixation. The dyes require a high pH for fixation and still remain partially unfixed (Kolonko, 2005). These dyes have complex structures and are difficult to degrade (Chakraborty et al., 2003) hence cause problems to the environment. MIAA was tested for the treatment of actual textile wastewater collected from a local industry.

2. Materials and methods

2.1. Reagents

Chemicals used in the present study include aluminum tri-sec butoxide (Merck, Germany), paraffin oil (MP Biomedicals, LLC Germany), ammonia solution (Burdick and Jackson, Germany), nitric acid (Panreac, Spain), and activated alumina powder (Merck, Germany). Cibacron reactive yellow dye was obtained from a local textile industry.

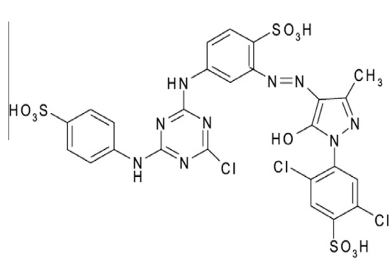

Figure 1 Cibacron reactive yellow dye.

Main equipments used in the study include muffle furnace (NEY M-525, USA), orbital shaker (Stuart SSL1, UK), and UV–visible spectrophotometer (Spectronic Genesys 5, USA).

2.2. Synthesis of immobilized adsorbent

Sol–gel method (Deng et al., 2006) was employed with some modifications in order to synthesize immobilized activated alumina. Boehmite sol was prepared by dissolving drop wise 25 mL of aluminum tri sec butoxide in 100 mL of distilled water and adding 1 g of powdered activated alumina at 75 °C with constant stirring. After dissolution, the solution was heated at 90 °C for one hour and 3.5 mL 1 M HNO_3 was added in the sol. The sol was refluxed in closed vials at 90 °C for 1 h. The sol formed (boehmite sol) was dropped into ammonia solution under paraffin oil layer with the help of a syringe. The droplets remained immersed in the ammonia solution for 45 min in order to form firm granules. After that the granules were washed thoroughly by distilled water and ethyl alcohol, dried and calcined at 450 °C for three hours to obtain new adsorbent named as modified immobilized activated alumina (MIAA). Fresh MIAA granules were synthesized for each batch experiment. The same procedure was applied to synthesize pure immobilized activated alumina that excluded the addition of powdered activated alumina.

Surface morphology of MIAA was examined by a scanning electron microscope (JEOL JSM-6460, Japan).

2.3. Adsorption study

Cibacron reactive dye adsorption experiments were conducted in order to determine the efficiency of MIAA adsorbent and the effect of controlling parameters like dose, contact time and stirring rate. The stock solution of 400 mg/L of dye was prepared by dissolving its 0.4 g in 1000 mL distilled water. All adsorption experiments were carried out in a 250 mL conical flask with 100 mL dye solution at 20 ± 1 °C using an orbital shaker at pH 7. Batch experiments were performed in triplicate for precision. The concentration of the dye in solution after adsorption was determined with the help of a calibration curve. The calibration curve was constructed by running standards of the dye on UV–Visible spectrophotometer at $\lambda_{max} = 396$ nm.

The effect of increase in the adsorbent dose on the dye removal was studied by varying the MIAA dose from 0.2 to 25 g/L in dye solutions with an initial concentration of 400 mg/L. In order to determine the optimum contact time, the flasks containing dye test solutions (with optimum MIAA dose) were agitated on the shaker for 5 to 105 min. Similarly, optimum stirring rate was determined by changing the rpm from 10 to 350.

The concentration of the dye was also varied from 5 to 700 mg/L in order to study its effect on adsorption. The specified amount of dye adsorbed q_e (mg/g) was calculated by Eq. (1).

$$q_e = \frac{C_i - C_s V}{m} \quad (1)$$