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Research article

Design and testing of a pilot scale magnetic separator for the treatment of textile dyeing wastewater

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ABSTRACT

Iron nanoparticles can be incorporated on the structure of natural clays to obtain magnetic clays, an adsorbent that be easily removed from a wastewater by magnetic means. Magnetic clays have high adsorption capacities of different contaminants such as heavy metals, fungicides, aromatic compounds and colorants and show rapid adsorption kinetics, but crucial data for achieving its full or pilot scale application is still lacking. In this work, magnetic bentonites with different amounts of magnetite (iron fractions on the clay of 0.55, 0.6 and 0.6) were used to remove color from a real textile wastewater. On a first stage the optimal conditions for the adsorption of the dye, including pH, temperature and clay dosage were determined. Also design parameters for the separation process such as residence time, distance from magnet to magnetic clay and magnet strength were obtained. Finally a pilot scale magnetic drum separator was constructed and tested. A removal of 60% of the dye from a wastewater that contained more than 250 ppm of azo dye was achieved with only 10 min of residence time inside the separator.

1. Introduction

The release of chemical dyes and other hazardous chemicals by the textile industry poses serious and immediate threats to both ecosystems and human health (Greenpeace International, 2011). The textile industry produces large volumes of wastewater from different steps in the dyeing and finishing processes. Textile wastewater is often rich in color, contains residues of reactive dyes and chemicals and has high chemical and biological oxygen demands (COD and BOD) as well as several recalcitrant compounds (Verma et al., 2012; Lin and Chen, 1997).

Due to the complexity of the textile industry wastewater, the treatment process often includes several steps mainly focused on the reduction of BOD and COD and the discoloration of the waste. Many processes have been adapted to treat the wastewater produced by the textile dyeing process, including biological treatment (Sarayu and Sandhya, 2012), adsorption (Yagub et al., 2014), coagulation and flocculation (Verma et al., 2012) advanced oxidation techniques (Abo-Farha, 2010; Akpan and Hameed, 2010; Kumar and Rao, 2017), membrane filtration (Ciardell et al., 2001) and a combination of these processes.

Adsorption is widely used on the removal of chemical contaminants from waters due to its flexibility and simplicity. Current work is focused on the search of cost effective adsorbents, with those derived from natural materials being particularly attractive (Yagub et al., 2014; Ali et al., 2012; Babel and Kurniawan, 2003). In recent years, there has been an increasing interest in the use of raw clay materials (kaolinite, diatomite, bentonite and Fuller's earth) as adsorbents of different inorganic and organic contaminants, including heavy metals (Babel and Kurniawan, 2003), amino acids (Fushan et al., 1997), phenols and chlorophenols (Lee et al., 1997; Lawrence et al., 1998) and chemical dyes (Abo-Farha, 2010).

Clays have a layered structure with exchangeable ions on its structure and act as natural scavenger of charged molecules through ion exchange and adsorption. They also possess large

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surface areas ranging up to 800 m^2g^{-1} (Ali et al., 2012), and high removal efficiencies have been demonstrated for different contaminants (Yagub et al., 2014). They can be applied on fixed or fluidized bed columns, systems that show bed saturation, frequent replacement of the adsorbent (or the need for adsorbent regeneration) and high pressure drops as main drawbacks. Due to its micrometric size, clay particles require large times to settle and are not suitable either for separation by sedimentation (Akther et al., 2008; Roussy et al., 2005). Thus, structural and chemical modifications of natural clays are being studied aiming at lowering the settling time of this adsorbent and its flocculation capability (Roussy et al., 2005; Divakaran and Sivasankara, 2001; Chatterjee et al., 2009) One of the most promising modifications of natural clays is the addition of magnetic iron oxides nanoparticles (Oliveira et al., 2003). With this modification, the adsorption features of clays and the magnetic properties of iron oxides are combined to produce magnetic clays, an adsorbent that can be easily removed from the medium by magnetic separation (Ambashta and Sillanpaa, 2010).

As recently reviewed (Chen et al., 2016), magnetic clays have been tested at lab scale on the adsorption of several contaminants including heavy metals (Oliveira et al., Giakisikli and Anthemidis, 2013), herbicides (Liu et al., 2014), pesticides (Ouali et al., 2015) aromatic compounds, humic acids (Peng et al., 2006) and crude oil (Hsu et al., 2010), among others. The removal of colorants such as methylene blue (Chang et al., 2016; Cottet et al., 2014; Tireli et al., 2014) and solutions of organic dyes such as methyl orange (Yu and Yang, 2010), acid red (Hashemian, 2015) and acid blue (Özcan et al., 2005) has been also analyzed. Experimental conditions that maximize the adsorption of the contaminants (clay dosage, contact time, pH, and temperature) have been determined, kinetic parameters have been calculated for different combinations of contaminants and adsorbents and the regeneration of modified clays for its reuse after the adsorption was investigated (Lou et al., 2017; Jiang et al., 2018). Despite all this progress, research is still on a laboratory scale and crucial data for achieving full or pilot scale application of magnetizable clays on wastewater treatment is lacking.

In this work, useful parameters for the design of a magnetic separation process of the magnetic clay from the wastewater were obtained. The optimal pH and clay dosage for the removal of color were determined. The effect of the magnetite content of the clay on the separation time were experimentally and analytically analyzed. The magnetic properties of the clays were determined and the time required to separate the clay particles by magnetic means was compared with that of gravity separation, for different particles sizes and magnet geometries. Also magnetic force profiles were estimated and the maximum allowable distance between the array of magnets and the clay particles for achieving efficient separation was analytically estimated. Finally, with the obtained results a pilot-scale magnetic drum separator was designed and preliminary tested for the treatment of a real textile wastewater.

2. Experimental

2.1. Wastewater characteristics

Real textile wastewater was collected from local dyeing industry, stored at 5 °C and used upon one week after collection. Wastewater contained Direct Black 22 (chemical structure shown on Fig. S1 of the electronic supporting material, ESM), sodium chloride, and sodium carbonate and had a pH of 8.5. The amount of dye on the wastewater was determined by measuring its absorbance at 480 nm and using a calibration curve performed with aqueous Direct black solutions of known concentration.

2.2. Bentonite characterization and modification with iron oxides

Bentonite used in this work had a cation exchange capacity of 1.05 meq/g and was supplied by Minarmco S.A (Argentina). Iron oxide nanoparticles were obtained by mixing solutions of ferric chloride in water (20% m/V) and ferric sulfate in hydrochloric acid (0.6% m/V) and adding to the mixture 13% v/v of a solution containing 25% ammonium hydroxide. Final solution was slowly stirred for 10 min. Bentonite was swelled in distilled water at room temperature. Different quantities of nanoparticles solution were added (3 ml, 6 ml and 12 ml of magnetite solution per gram of clay for bentonites I, II and III respectively). The mixture was stirred for 2 h and then centrifuged. The precipitated solid was dried at 90 °C for 24 h, milled and sieved. Images of sieved clay particles were1 acquired with for measuring particle size (Fig. S2 on ESM). X-ray diffraction patterns of the raw clay and the modified clay were obtained to verify the presence of magnetite on the structure of the modified clay (Fig. S2). X-ray fluorescence was used to determine the fractions of chemical elements (higher than sodium) on each of the modified clays. Iron fraction on raw bentonite was 0.427, whereas for Bentonites I, II and III was 0.559, 0.604 and 0.652, due to the different magnetite contents of each clay. The complete composition of the bentonites is shown on ESM. The density of the clay was determined by compressing the powder with a hydraulic press obtaining 1 cm^3 samples that were weighed on an analytical balance. Density of the clays did not vary with the amount of magnetite added on the modification. Obtained density value was 2150 $kg\,m^{-3} \pm 160\,kg\,m^{-3}$. Magnetization (M) vs field (H) curves were performed to magnetic clays in a VSM magnetometer LakeShore 7004 at room temperature (Fig. S4 on ESM). Samples weight was around 100 mg. Specific saturation magnetization of 5.8, 8.5 and 17 Am^2/Kg were found for bentonites BI, BII and BIII respectively.

2.3. Wastewater discoloration assays

Batch assays were performed at different pH and clay dosages. The solution pH affects the adsorption of contaminants on the clay, since it alters the ionization of the dye molecule and the surface properties of the adsorbent (Yagub et al., 2014; Özcan et al., 2005; Ramakrishna and Viraraghavan, 1997). The pH was varied between 3 (the pH for magnetite dissolution) and 8.5 (raw wastewater pH) by addition of H_2SO_4 and NaOH solutions. Dosage of modified bentonite (BIII) on the samples was varied between 5 and 30 g per liter at different pHs. Wastewater samples with modified pH were transfer to a glass beaker and clay was added under constant agitation. A paddle stirrer was used, as stir bars would have attracted the magnetic adsorbent. After 10 min a sample of the liquid was taken and centrifuged for 5 min at 10,000 rpm. The fraction of dye removed from wastewater was determined by measuring samples absorbance at 480 nm (Durruty et al., 2015). Initial concentration of dye on the wastewater was 460 ppm. Temperature was kept constant at 25 °C during adsorption assays.

2.4. Settling tests

Settling time of the particles was evaluated with and without application of magnetic field on the sample. When the clay with the adsorbed dye settles, the wastewater is progressively discolored. Thus, absorbance of the wastewater can be used as an indicator of the progress of the settling process. The evolution of the absorbance of a sample containing wastewater and modified clay was