



Adsorption/desorption behavior of acid orange 10 on magnetic silica modified with amine groups

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ABSTRACT

Two silica samples were prepared through precipitation of silica in the presence and absence of magnetite particles (Fe_3O_4). The products were immobilized with 3-aminopropyltriethoxysilane and characterized by means of FT-IR and X-ray. Surface area (BET), pore volume and pore diameter were also measured. The adsorption behavior of both silicas towards C.I. acid orange 10 in aqueous solutions was studied at different experimental conditions including contact time, pH and initial concentrations. The monoamine modified magnetic silica (MAMMS) displayed higher and faster adsorption relative to magnetite free one (MAMPS). The maximum adsorption capacity of the dye on MAMPS and MAMMS are 48.98 and 61.33 mg g^{-1} , respectively. Adsorption of the dye on both MAMMS and MAMPS fitted to Langmuir adsorption model and followed the pseudo-second order kinetics. The values of Gibbs free energy of adsorption (ΔG°) were found to be -26.52 and -28.49 kJ/mol at 298 K for MAMPS and MAMMS, respectively. These negative values indicated the spontaneity of the adsorption of the dye on both silica samples. Regeneration of the dye-loaded silica was carried out using aqueous solution of pH 10. Desorption ratio of 98% was obtained over three adsorption/desorption cycles.

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1. Introduction

Industrial development is pervasively connected to the disposal of a large number of various toxic pollutants including dyes. Dyes are present in the wastewater streams of many industrial sectors such as, dyeing, textile, tannery and the paint industry [1]. Dye bath effluents are not only aesthetic pollutants by their color but also interfere with light penetration that disturbs biological processes [2]. The total dye consumption of the textile industry worldwide is in excess of 10^7 kg per year. Consequently, one million kilograms per year of dyes are discharged into waste streams by the textile industry [3,4]. Many of conventional treatment technologies for dye removal have investigated extensively such as chemical coagulation or flocculation combined with flotation and filtration, membrane filtration, oxidation, and photo-degradation processes [5,6]. The adsorption process provides an attractive method for the treatment of textile effluent especially if the adsorbent is inexpensive and readily available [7,8]. The adsorption of acid dyes has been studied using different adsorbents; bentonite [5], hectorite [7], peat [9], activated bleaching earth [10], chitosan [11], montmorillonite [12], titania-silica composite [13], etc.

Among inorganic compounds, silica deserves particular attention, considering its stability, very low degree of swelling, controlled porosity and the chemical reactivity of their surfaces, resulting from the presence of silanol groups [14]. Coating magnetic particles with silica is becoming a promising and important approach in development of magnetic particles for both fundamental study and technology application [15]. Many of investigators used magnetic silica carriers for DNA purification and immobilization [16,17], protein adsorption [18], enzyme immobilization [19], etc. Little work has been found in the literature related to the use of magnetic silica in removal of dyes from their aqueous solutions.

Azo dyes are an interesting class of compounds that are widely used in textile industries [20]. In this study, precipitated and magnetic silica samples were prepared and immobilized with 3-aminopropyltriethoxysilane. The adsorption/desorption behavior of acid orange 10 (as a model of azo dyes) on the obtained silica samples was clarified. Kinetic and thermodynamic parameters of adsorption were calculated.

2. Materials and methods

2.1. Materials

3-Aminopropyltriethoxysilane (APTS) was purchased from Fluka, Italy; silica gel and C.I. acid orange 10 (AO-10) (80%) were obtained from Sigma-Aldrich Chemical Co., Germany. All other

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chemicals were of analytical grade and were used as received. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ were used as sources for Fe(II) and Fe(III), respectively.

2.2. Preparation of magnetite

Magnetite was prepared following the modified Massart method [21]. A 250 cm^3 of 0.2 mol dm^{-3} Fe(III) solution was added with stirring to a freshly prepared 250 cm^3 of 1.2 mol dm^{-3} Fe(II) solution. A 200 cm^3 of (30%) NH_4OH was suddenly poured to the previously prepared Fe(III)/Fe(II) solution while vigorous stirring was going on. A black precipitate was allowed to precipitate for 30 min with stirring. The precipitate was washed with deoxygenated water (water was boiled to repeal any gases then bubbled with nitrogen gas) under magnetic decantation until the acidity of suspension became below pH 7.5. The precipitate was dried at ambient temperature to give a black powder.

2.3. Preparation of magnetic silica

Five grams of silica was dissolved in 120 cm^3 of 4 mol dm^{-3} NaOH solution with heating to 353 K and stirring until completely dissolution. A half gram of grinded Fe_3O_4 was dispersed in 100 cm^3 of NaOH. The pH of the two solutions was adjusted to 12–13 by 2 mol dm^{-3} HCl. Sodium silicate solution was poured into the magnetite suspension with stirring for 30 min at 353 K. Hydrochloric acid of 2 mol dm^{-3} was added dropwise to adjust the pH value to 8 ± 0.2 . The obtained precipitate was washed several times with bi-distilled water and then dispersed in 100 cm^3 of methanol. The silica-coated magnetite particles were removed from the medium using a magnet. The magnetite free precipitated silica was obtained following the same procedure in the absence of magnetite.

2.4. Immobilization of precipitated silicas with monoamine

Magnetite-coated silica and magnetite-free silica obtained in the above step were loaded by monoamine as follows: one cubic centimetre of 3-aminopropyltriethoxysilane (APTS) was dissolved in 100 cm^3 of bi-distilled water acidified with acetic acid (pH 4). Two grams of precipitated silicas (activated by drying in an oven at 423 K for 18 h) was added in the silane solution and stirred for 2 h at room temperature. The products were filtered off and kept in an oven at 393 K for 4 h. The dried products were washed repeatedly with distilled water, ethanol, and acetone to remove the unreacted material and then dried in the oven at 393 K for another 2 h and then referred as MAMMS (for magnetic-silica) and MAMPS (for magnetite-free silica) [22].

2.5. Characterization of the modified silicas

Nitrogen specific surface area (BET) of the obtained modified silicas was measured at 77.35 K by Quantachrome Instrument, NOVA 2000 series, USA using nitrogen as the sorbate. All measurements were taken after heating the samples up to 403 K and evacuation at a pressure of 10^{-4} Torr for 4 h. FT-IR measurements were performed in KBr discs using Nexeus-Nicolite-640-MSA FT-IR, Thermo Electronics Co., USA. X-ray diffraction (XRD) was carried out using Philips X-ray diffractometer model PW 1775 with $\text{Cu K}\alpha$ radiation and Ni filter.

The chemical stability of the magnetite containing silica was checked by soaking silica-coated magnetite particles in strong acidic medium (pH 3) for 3 h. No appreciable dissolution of magnetite was observed. This indicates that the investigated silica displays a good chemical stability in strong acidic medium.

2.6. Preparation of AO-10 solutions and concentration measurements

Stock solution (240 mg dm^{-3}) of the investigated dye was prepared in bi-distilled water. The desired concentrations were obtained through dilution. The concentration of the dye was measured by spectrophotometric method [7]. Calibration curve of the investigated dye was prepared by measuring its absorbance against concentration at $\lambda_{\text{max}} 475 \text{ nm}$ using UV/vis spectrophotometer Model SP-850, Metertech Inc., Taipei, Taiwan, with 1.0 cm path-length cell. Adsorption of the dye on the silica samples obtained was determined using the mass balance equation [4]:

$$q = \frac{(C_i - C_f) \times V}{W} \quad (1)$$

where q represents the amount of dye adsorbed (mg g^{-1}), C_i and C_f are the initial and final concentrations of dye (mg dm^{-3}), V is the volume of solution (dm^3) and W is the weight of silica sample (g).

3. Batch adsorption

3.1. Effect of pH

The effect of acidity of the medium on the adsorption of AO-10 on modified silicas was investigated at 298 K. Portions of 0.1 g of dry silica samples were placed in a series of flasks containing 20 cm^3 of stock solution of 240 mg dm^{-3} of dye. The desired pH was controlled by 2 mol dm^{-3} HCl and 2 mol dm^{-3} NaOH. The solution was completed to 100 cm^3 with bi-distilled water to give initial concentration of 48 mg dm^{-3} of dye. The contents of the flasks were equilibrated on a Gallenham Shaker, England, at 400 rpm for 4 h. The residual concentration of the dye in each flask was determined spectrophotometrically at $\lambda_{\text{max}} 475 \text{ nm}$.

3.2. Effect of contact time

The effect of contact time on the adsorption was studied by placing 0.1 g samples of dry silica in a series of flasks each contains 20 cm^3 of stock solution of dye of concentration 240 mg dm^{-3} at pH 3 and 298 K. The solution was completed to 100 cm^3 with bi-distilled water to give initial concentration of 48 mg dm^{-3} of dye. The flasks were removed from the shaker at different time intervals. Five cubic centimeters of the solution was taken and centrifuged to determine the residual concentration of the dye as above.

3.3. Isotherms

Complete adsorption isotherms were obtained by placing samples of 0.1 g of dry silica in a series of flasks containing 100 cm^3 of dye solution at the desired initial concentrations (40 – 160 mg dm^{-3}) and at pH 3. The flasks were conditioned for 4 h at 400 rpm while keeping the temperature constant at 298, 313, 323 or 333 K. Later on, the residual concentration of the dye was estimated.

3.4. Desorption

Silica sample was loaded by the dye as follows: a 0.1 g of dry sample was added to a flask containing 20 cm^3 of stock solution of dye of concentration of 240 mg dm^{-3} at pH 3. The solution was completed to 100 cm^3 with bi-distilled water to give initial concentration of dye of 48 mg dm^{-3} . The flask was conditioned for 4 h at 400 rpm while keeping the temperature at 298 K. Later on, the contents of the flask were filtered off and the dye-loaded silica was placed in another flask containing 100 cm^3 of aqueous solution at pH 7–10 using 2 mol dm^{-3} NaOH. The flask was agitated at 400 rpm for 2 h at 298 K. The concentration of the dye released was estimated as

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