



Removal of methyl orange by copper sulfide nanoparticles loaded activated carbon: Kinetic and isotherm investigation



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ABSTRACT

This work describes the synthesis of new nanoparticle based adsorbent for the removal of methyl orange (MO) from colored waste water. Copper sulfide nanoparticles were synthesized and impregnated on the commercial activated carbon (CuS-NP-AC) and successfully characterized using UV–Vis spectrophotometer, FESEM, XRD and BET. BET surface area of prepared adsorbent was measured as 1286 m²/g. Effects of various parameters such as pH, initial MO concentration (mg/L), mass of prepared adsorbent dosage (g L⁻¹) and contact time (min) were studied. The optimization conditions were set as: 4.0 min, 14 mg/L and 25 mg/L for contact time, mass of prepared adsorbent dosage and initial MO concentration, respectively, at pH 5.0. The adsorption equilibrium data were analyzed using different models and Langmuir model represented the equilibrium data well with adsorption capacity of 122 mg/g. Kinetic data revealed that the adsorption of MO dye on the adsorbent surface followed pseudo-second order model.

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1. Introduction

The extensive application of dyes compound in various industries such as textile, paint, leather, paper and plastics discharges toxic colored effluent and contaminates surface and ground water. Dyes are resistant to heat, chemical reagent and have ability to generate cancer and mutagens. Presence of dyes in water creates serious impact for different aquatic organism [1–4]. Therefore, their removal from wastewater before the wastewater entered into human bodies is great demand. Among these pollutants, methyl orange (MO) dye belongs to anionic category and toxic dye and it has been widely applied in the above industries. MO leads to health hazards such as difficulties in breathing, vomiting, diarrhea and nausea. It is a weak acid–base indicator with potential applicability as toxic and carcinogenic agent. Therefore, its removal and elimination from various aqueous waste is required. Generally, coagulation, precipitation, adsorption, electrochemical techniques, ozonation and biosorption are suitable candidates for such purpose [5–11]. These techniques have some limitations that include requirement of large amount of supporting material and generation of large amount of secondary waste. Adsorption is one of the most studied dye removal technique that leads to producing high-quality purified water. The adsorption based procedure was widely used due to its high efficiency, capacity and ability for large scale dye removal application [12–15]. The nontoxic, low cost and easily available adsorbents are

the best candidate for wastewater treatment. On the other hand, extent of adsorption significantly depends on the presence of active sites on the surface of adsorbents. Recently M. Ghaedi et al. widely use metallic nanostructures loaded on activated carbon as adsorbents for the removal of pollutants in aqueous environment [6,8,10,15–20]. Carbon-based materials and their modification by novel nanoscale materials lead to benefits such as high surface area, porous structure, large adsorption capacities, fast adsorption kinetics and simultaneously providing easy, low cost and efficient method for the removal of pollutants from the wastewater [21–23].

In this work, CuS nanoparticles were synthesized by green method and loaded on the surface of commercial activated carbon (CuS-NP-AC). The prepared CuS-NP-AC was characterized by UV–Vis spectrophotometer, field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and BET analysis. The dye removal performance of prepared adsorbent was investigated at various operating conditions. Kinetics and equilibrium study during adsorption was also performed. A calculation procedure is proposed to know the amount of adsorbent requirement for known amount of water to be treated.

2. Experimental

2.1. Materials and instruments

MO dye was used for the preparation of colored waste water. pH of the solution was adjusted by HCl and NaOH solution. All the chemicals including NaOH, HCl, KCl and Copper (II) acetate (analytical reagent grade)

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were supplied by Merck Company (Darmstadt, Germany). A pH/ion meter (Metrohm, Switzerland, Swiss, model 692) was used for the measurement of pH. MO concentration was determined using an UV–Vis spectrophotometer (Jasco, model V-530, Japanese) at a wavelength of 465 nm. The morphology of the CuS-NP-AC was observed by field emission scanning electron microscopy (FESEM; Hitachi S-4160, China) under an acceleration voltage of 15 and 20 kV. X-ray diffraction (XRD) pattern was recorded by X-ray diffractometer (Philips X'Pert, 40 kV and 30 mA, American) for 2θ values over $10\text{--}80^\circ$. A BET surface analyzer (Quanta chrome NOVA 2000e, Company Quantachrome Instruments, American) was used for examining the nitrogen adsorption–desorption isotherm at 77 K. Before each experiment, the samples were degassed using helium at 553 K for 3 h. According to BET analysis result some useful information about surface properties of adsorbent including surface area, total pore volume and size and porosity was obtained.

2.2. Batch experiments methods

Stock solution of MO of concentration 1000 mg/L was prepared by dissolving an accurately weighed quantity (0.1 g) of solid dye in 100 mL of deionized water (pH is 6.9). Experimental solutions of desired concentrations were obtained by successive dilution of the stock solution. For studying the effect of solution pH on dye adsorption, experiments in different pH (varying from 1 to 8) were conducted for initial dye concentration of 15 mg/L. To observe the effect of adsorbent dose on dye adsorption, different amounts of adsorbent (varying from 2 to 16 mg/L) are used. A common adsorbent dose of 14 mg/L, stirring speed of 400 rpm and pH 5.0 was used for all the above experiments.

In order to study the adsorption isotherm, 1.0 mg of adsorbent was kept in contact with 100 mL dye solution of different concentrations at pH 5.0 for 4.0 min with constant shaking at ambient temperature. After 4 min the solution attains equilibrium and the amount of dye adsorbed (mg/g) on the surface of the adsorbent was determined by the difference of the two concentrations. Duplicate experiments were

carried out for all the operating variables studied and only the average values were taken in to consideration. The average deviation of duplicate results in the units of concentration is found to vary within $\pm 2.0\%$. Blank experiments were carried out with dye solution and without adsorbent to ensure that no dye was adsorbed onto the walls of the beakers. The amount of dye adsorbed per unit weight of adsorbent at time t , q_t (mg/g) and percentage dye removal efficiency (R %) was calculated as follows:

$$q_t = \frac{(C_0 - C_t)V}{M} \quad (1)$$

$$R = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

where, C_0 is the initial dye concentration (mg/L), C_t is the concentration of dye at any time t , V is the volume of solution (L) and M is the mass of adsorbent (g) [6,15,18].

2.3. Preparation of copper sulfide nanoparticle

CuS nanoparticles were synthesized based on the reaction of the mixture of Copper(II) acetate ($\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) with thioacetamide (CH_3CSNH_2) in aqueous media. In a typical synthesis, 10 mL of a 0.1 mol L^{-1} ($\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) and 20 mL 0.2 mol L^{-1} tri-sodium citrate as capping and reducing agent were taken in a 100 mL beaker. In the next step, 5.0 mL of 0.4 mol L^{-1} thioacetamide (TAA) as source for S^{2-} ions was added to it slowly. Finally, double distilled water was added to the solution to make the volume close to 100 mL and the solution was stirred for 10.0 min for well mixing of the constituents. The resulting mixture was taking place at room temperature and the citrate stabilized CuS nanoparticles started to grow slowly. In the next step, 100 mL of the freshly prepared CuS nanoparticles suspension was mixed with 3.0 g of the activated carbon in a 250 mL Erlenmeyer

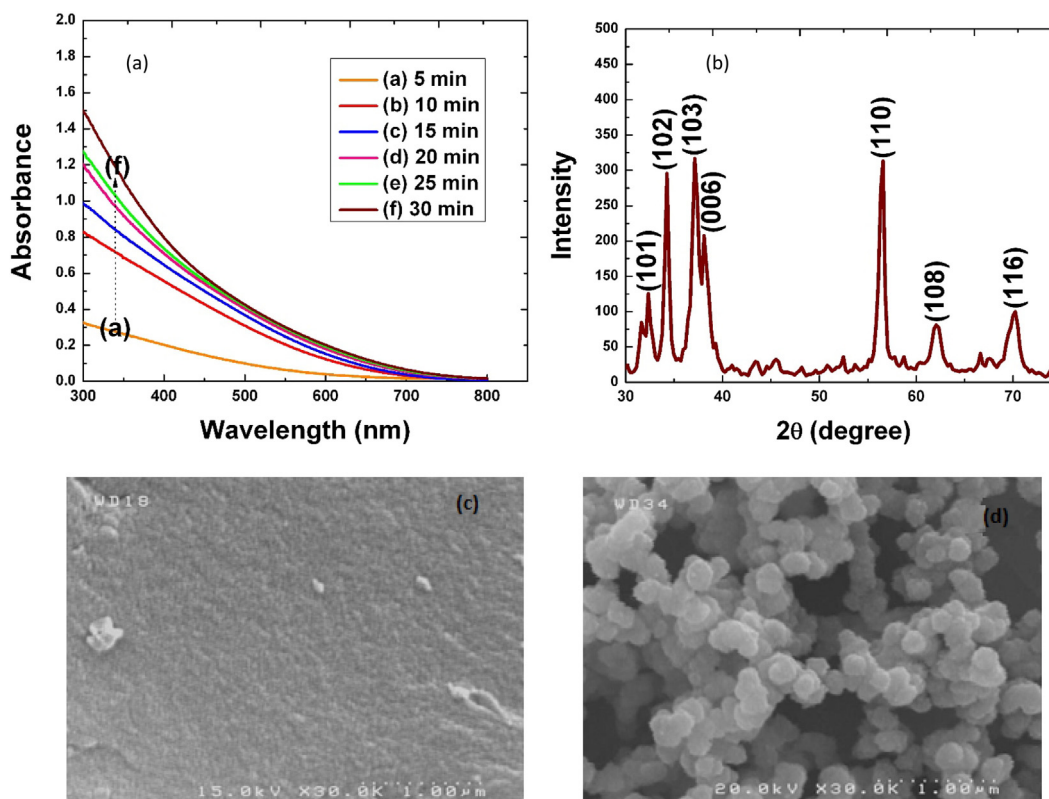


Fig. 1. Absorption spectra of the CuS nanoparticles taken at 5 min (a), X-ray diffraction (XRD) pattern of the CuS nanoparticles (b), FESEM images of activated carbon (c) and FESEM images of CuS nanoparticles deposited on activated carbon (d).

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