



Modification of nanosized natural montmorillonite for ultrasound-enhanced adsorption of Acid Red 17



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ABSTRACT

This work aims to modify montmorillonite (MMT) via dodecyltrimethylammonium bromide (DTMA) and investigate its ability in ultrasound (US) assisted decolorization of a polluted solution. BET surface area of MMT was increased from 19.76 to 42.57 m²/g and basal spacing of MMT structural layers was increased from 1.13 to 1.69 nm by DTMA modification. The application of DTMA-modified MMT (DTMA-MMT) and US for the decolorization of Acid Red 17 (AR17) showed that US could improve the ability of DTMA-MMT on decolorization of AR17 solution due to simultaneous adsorption and sonocatalysis. The ability of US assisted DTMA-MMT was slightly decreased with pH, the initial dye concentration and the presence of inorganic anions.

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1. Introduction

The wastewaters containing various kinds of synthetic dyestuffs from industries such as textile, paper, plastic, leather, food and mineral processing are the main source of aquatic pollution [1,2]. Pollution of water with these colorful organic compounds reduces the penetration of light in water and subsequent decrease of photosynthesis as well as increases its chemical oxygen demand (COD) [3]. Therefore, discharge of industrial wastewater containing these kinds of pollutants to the receiving water sources without any treatment is not desirable and the pollutant removal is of great importance from an environmental point of view [1,4]. Different chemical and physical methods such as coagulation and flocculation [5], membrane filtration [6], ozonation [7], biological processes [8] and adsorption [9] techniques are usually used to remove the pollutants from contaminated water and wastewater. Adsorption process is considered as one of the promising processes for effective removal of pollutants from aqueous media [10]. Different adsorbents are prepared and used in the removal of chemical and biological pollutant from wastewater until today. Clays as cheap, abundant and environmentally friendly, easily extracted, non-toxic and mechanically and chemically stable solid materials

have potential to be used as adsorbent in the treatment of wastewater [11]. By the way, pristine clay is not sufficiently effective for the adsorption of some pollutants especially anionic contaminants from aqueous solutions. A method extensively used to modify clays is exchange of the inorganic cations in the interlayer galleries of clay with organic cations using organic salts [11,12]. Furthermore, it has been understood that combination of adsorption with other efficient processes such as sonolysis is another method to increase the efficiency and decrease the cost of pollutant removal [13,14]. As known, ultrasonic irradiation leads to acceleration in chemical reactions and mass transfer as a result of acoustic cavitation which involves the formation, growth, and collapsing of bubbles [14–17]. The collapse of bubbles generates localized high temperatures and high pressures which induce a shock wave in the solution to increase mass transfer rate as well as causing the thermal dissociation of water molecules into hydroxyl (·OH) and hydrogen (·H) radicals. Destruction of organic pollutants in aqueous solution is attributed to direct pyrolysis of pollutants and/or degradation by reactive radicals [2,15–17]. It is necessary to mention that the presence of solid particles such as adsorbent that increases the efficiency of acoustic cavitation and consecutively the degradation rate of organic contaminants, resulting in the decrease in the required time, energy and cost of wastewater treatment process [2,14].

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The aim of this study is to investigate the chemical modification of pristine montmorillonite via dodecyltrimethylammonium bromide and subsequently its application for the US-enhanced adsorption of a model pollutant, Acid Red 17. Due to the high cation exchange capacity, swelling properties and large surface area, the MMT as smectite clay mineral having 2:1 expandable-layered structure was used [11,18]. The effect of some important parameters on the dye removal process, such as initial dye concentration, pH of solution, adsorbent dosage, reaction time and presence of inorganic anions on decolorization efficiency of AR17 solution has been studied.

2. Materials and methods

2.1. Chemicals

Montmorillonite was obtained from Karakaya Mineral Co., Ankara, Turkey. The cation exchange capacity (CEC) of the MMT is 149 meq/100 g. The chemical composition of the MMT sample was determined by Rigaku RIX-3000 X-ray fluorescence spectrometry (Rigaku Corporation, Tokyo, Japan). The MMT sample contains SiO₂ (59.32), Al₂O₃ (17.19), Fe₂O₃ (5.95), MgO (3.63), CaO (2.21), Na₂O (1.67), K₂O (0.97), and others (9.06) wt.%. The dye, Acid Red 17, was purchased from Shimi Boyakhsaz Company, Iran and used without any further purification. All other chemicals and reagents were of analytical grade and purchased from Merck, Germany and they were used without further purification. The specifications of the AR17 along with DTMA are given in Table 1.

2.2. Preparation of DTMA-modified MMT

To modify the MMT, 1 g of the clay was dispersed in 100 mL distilled water and stirred at 250 rpm for 24 h to swell and reach homogeneity. The DTMA by amount equal to 1.0 CEC of pure MMT was slowly added to the clay suspension. After stirring the mixture for 1 h, the resulting DTMA-modified MMT sample was filtered and washed with distilled water to remove excess salts, and then dried at 90 °C in an oven. Finally, the prepared sample was ground and the obtained modified clay sample was stored in a closed vessel to use in the next experiments [18,19].

2.3. Characterization methods

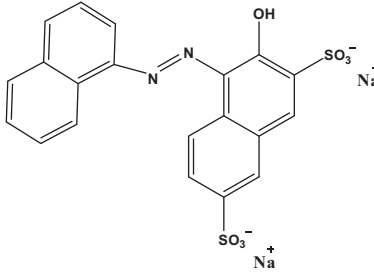
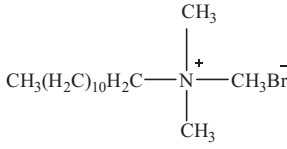
The physicochemical properties of the samples were determined by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), and scanning electron microscopy (SEM) techniques. XRD patterns of pure MMT and DTAB-modified MMT

samples were gained in Bruker D8 Advance X-ray diffractometer with a Cu K α (λ : 1.5404 Å) radiation, operating at 40 kV and 30 mA over a 2 θ range of 2–80°. FT-IR spectra of the samples were run on a Perkin Elmer Model 1600 FT-IR spectrophotometer using KBr pellets between 400 and 4000 cm⁻¹. The morphology of the samples were examined by a Quanta 400 F Field Emission SEM (FEI Company, USA) at 30 kV. The pH of zero point charge (pH_{zpc}) of the DTMA–MMT sample was determined according to the method designated by Bessekhoud et al. with some modification [20]. Accordingly, a series of 100 mL 0.01 M NaCl was prepared and transferred in conical flask. The pH values of the solutions were adjusted from 2.5–10.5 by adding either HCl or NaOH solution. 0.1 g of DTMA–MMT sample was added to each solution and the obtained suspension was stirred for 48 h. The final pH of each solution was measured. The difference between the initial and final pH values (Δ pH) were plotted against related initial pH. The pH_{zpc} was found equal to the initial pH value in which Δ pH was equal to zero.

2.4. Experimental procedure

Batch adsorption and US-enhanced adsorption experiments were carried out in thermostatic shaker (Julabo, SW22, Germany) and ultrasonic bath (640 W, 40 kHz, Bandelin Sonorex, Germany), respectively. The experiments were performed using 100 mL, glass-stoppered, round-bottom flasks immersed in a thermostatic shaker and ultrasonic baths. 0.1 g of clay was mixed with 100 mL of the aqueous solutions of different initial AR17 concentrations except for the experiments in which the effect of adsorbent dosage was examined. The flasks and their contents were shaken at 293 K, for various times (2.5–60 min) and at natural pH. The initial pH adjustments of the solutions were done using concentrated HCl and NaOH solution and measured by a pH meter (WTW Inc., Weilheim, Germany). The initial concentration of the AR17 solution, pH of solution, adsorbent dose and time were selected as experimental parameters and their effects on color removal efficiency were evaluated. Furthermore, the effect of inorganic ions on dye removal efficiency was investigated by adding sodium chloride and sodium sulfate. The residual concentration of AR17 in the reaction mixture was obtained by measuring absorbance at maximum wavelength of 514 nm via a Varian UV–visible spectrophotometer (Cary 100, Australia) and computing the concentration from AR17 concentration/absorbance calibration curve prepared prior to the experiments. In order to calculate the percent decolorization efficiency in different intervals, the equation of $[(C_0 - C_t)/C_0] \times 100$ was used, where C₀ is the initial concentration of AR17 solution and C_t is its concentration (mg/L) at time t.

Table 1
Characteristics of Acid Red 17 and DTMA.

Characterization	Acid Red 17	DTMA
Chemical structure		
Molecular formula	C ₂₀ H ₁₂ N ₂ Na ₂ O ₇ S ₂	CH ₃ (CH ₂) ₁₁ N(CH ₃) ₃ Br
Mw (g/mol)	502.435	308.34
Color index number	16,180	-
λ_{\max} (nm)	514	-

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