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Mechanisms and chemistry of dye adsorption on manganese oxides-modified diatomite

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ABSTRACT

The investigations into structural changes which occur during adsorbent modification and the adsorption mechanisms are essential for an effective design of adsorption systems. Manganese oxides were impregnated onto diatomite to form the type known as δ-birnessite. Initial investigations established the effectiveness of manganese oxides-modified diatomite (MOMD) to remove basic and reactive dyes from aqueous solution. The adsorption capacity of MOMD for methylene blue (MB), hydrolysed reactive black (RB) and hydrolysed reactive yellow (RY) was 320, 419, and 204 mg/g, respectively. Various analytical techniques were used to characterise the structure and the mechanisms of the dye adsorption process onto MOMD such as Fourier transform infrared (FTIR), X-ray diffraction (XRD) and atomic absorption spectrometry (A.A.). A small shift to higher values of the d-spacing of dye/MOMD was observed indicating that a small amount of the dye molecules were intercalated in the MOMD structure and other molecules were adsorbed on the external surface of MOMD. Two mechanisms of dye adsorption onto MOMD were proposed; intercalation of the dye in the octahedral layers and adsorption of the dye on the MOMD external surface. Moreover, the results demonstrated that the MOMD structure was changed upon insertion of MB and RY with an obvious decrease in the intensity of the second main peak of the MOMD X-ray pattern.

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1. Introduction

Textile wastewater is rated the most polluting among industrial sectors with respect to both discharge volume and effluent composition. The removal of colour arising from the presence of the water-soluble reactive dyes is a major problem. Current methods, relying on activated sludge systems, are inadequate, both at on site installations and also even after dilution with domestic wastewater at sewage works. Despite extensive research into colour removal from textile wastewater, problems still remain with high capital cost plant requirements, high operating costs, poor extent of colour reduction (particularly for sulphonated azo dyes). In addition, there is a significant sensitivity to variable wastewater inputs in the application of the chemical/physical treatment methods for the treatment of reactive dyes in a wastewater.

Two properties of reactive dyes limit the rate of colour removal: the highly soluble hydrolysed azo structure and the low biodegradability. Due to the chemical nature, molecular size and structure of the dyes, activated carbon appears to offer some prospects as an adsorbent material in effluent treatment. A number of investigations were conducted to evaluate adsorption of dyes onto a range of adsorbents such as activated carbon, peat, lignite, and fly ash (Mohan et al., 2002; McKay and Allen, 1980). The use of activated carbon in the areas of the Middle East is restricted due to high capital and regeneration costs required for the adsorbent. The search for locally abundant, low-cost alternatives is essential for the survival of a large number of local industries. The use and reuse of the water is vital since water is considered such an important commodity. One material under investigation is diatomite in its raw and modified forms. Al-Ghouti (1999) showed that the diatomite impregnated by manganese oxides (MOMD) was an effective adsorbent for removing heavy metals from aqueous solution. Manganese oxides are regarded as a good scavenger of heavy metal ions to the extent that it has been considered as a sink for heavy

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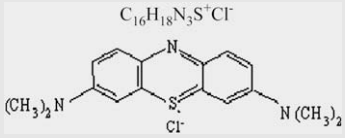
Table 1
Main characteristics of the dyes used in this study.

Dye	MB	RB	RY
Type	Basic dye C.I.52015	Cibacron reactive black C-NN	Cibacron reactive golden yellow MI-2RN
Phase	Solid	Liquid, 33%wt	Liquid, 33%wt
λ_{max}, nm	663	597	430
ε, $dm^3/g/cm$	170.1	22.6	23.0
Chemical structure		Unknown	Unknown

metals (Al-Degs et al., 2001). Manganese oxides are an important material in terrestrial, marine geochemistry and in sediments. The birnessite-type ($Na_4Mn_{14}O_{27}\cdot 9H_2O$) or ($\delta$-$MnO_2$) is one of the most active and common forms of mineralised manganese in soil, sediments, and water. It is a strong adsorbent of mineral ions and acts as a scavenger in marine and freshwater environments (James and Stahl, 1991; Renuka and Ramamurthy, 2000; Wang and Kanoh, 2001).

To our knowledge, there are no reported investigations on the adsorption of dye from aqueous solution using diatomite or manganese oxides-modified diatomite (MOMD) and no information on mechanisms of dye interaction onto MOMD (birnessite-type) reported in the literature. Thus, it is the aim of this study to highlight the mechanisms of dye adsorption in an effort to successfully design adsorption systems. In this study MOMD was investigated before and after dye adsorption. A variety of analytical techniques such as Fourier transform infrared (FTIR), X-ray diffraction (XRD), atomic absorption spectrometer (AA), and scanning electron microscopy (SEM) were employed in an effort to elucidate adsorption mechanisms.

2. Materials and methods

Diatomite samples were obtained from borehole BT-36, depth 34–36 m in Al-Azraq region in East Jordan. Methylene blue (MB), a basic dye, Cibacron reactive black (RB), and Cibacron reactive yellow (RY) dyes were used; a summary of the main characteristics of these dyes is given in Table 1. The standard stock solutions of the dyes were prepared by appropriate dilution with deionised water to a final concentration of 1000 mg/dm^3. To represent real textile effluent conditions, the reactive dyes were hydrolysed using the method described by Laszlo (1996).

The diatomite was modified by treatment with manganese chloride and sodium hydroxide. The details of the preparation are given elsewhere (Khraisheh et al., 2004). To study the structure characteristics of MOMD before and after dye insertion in the octahedral layers, equilibrium adsorption isotherms were carried out for MB, RB, and RY. The spent MOMD samples were dried in an oven at 70 °C overnight and allowed to cool in a desiccator. FTIR and XRD analyses of fresh and spent MOMD were then undertaken. Various adsorption isotherms onto spent MOMD with RB named, RB/MOMD were performed with fresh MB and RY. The experimental conditions of preparing RB/MOMD were: initial concentration of RB: 1000 mg/dm^3, pH: 2.00, particle size: <106 μm, shaking time: 48 h, shaking speed: 125 rpm, and temperature: 22 °C.

XRD analysis was carried out using a Siemens Diffractometer D 5000, Cu Kα1 radiation ($\lambda = 1.5406$ Å, 40 kV, 40 mA) and the sample was scanned from 3 to 55° (2θ) in step sizes of 0.04°. Infrared spectra were obtained by KBr pellet method using an FTIR-Perkin Elmer Spectrophotometer RX I. In order to examine the mechanisms of adsorption, atomic absorption spectrometer (A.A. Perkin Elmer Analyst 100) was used to analyse the concentration of sodium ions in equilibrium dye solution before and after dye adsorption.

3. Results and discussion

Initial investigations by the authors (Khraisheh et al., 2004) showed that the adsorption behaviour of RB is different from those for MB and RY and the summarised results are shown in Fig. 1. In an effort to elucidate the adsorption mechanisms, a variety of analytical techniques such as FTIR, XRD, and A.A. were employed and the results are discussed in the following subsections.

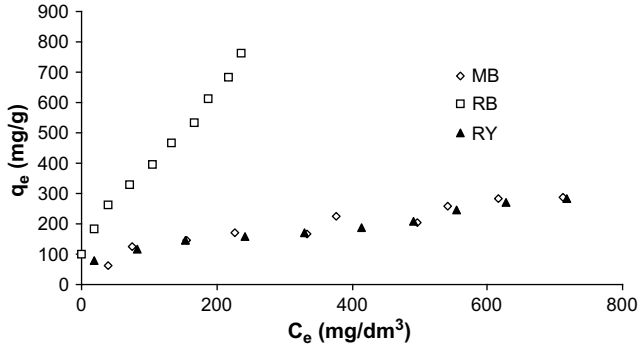


Fig. 1. Adsorption isotherms of MB, RB, and RY onto MOMD. Experimental conditions: mass of the adsorbent = 0.05 g, volume of solution = 50.0 cm^3, pH of MB, RB, and RY = 11, 2, 2 respectively, particle size = 106–250 μm, equilibrium time = 48 h, shaking speed = 125 rpm and temperature = 20 ± 2 °C.

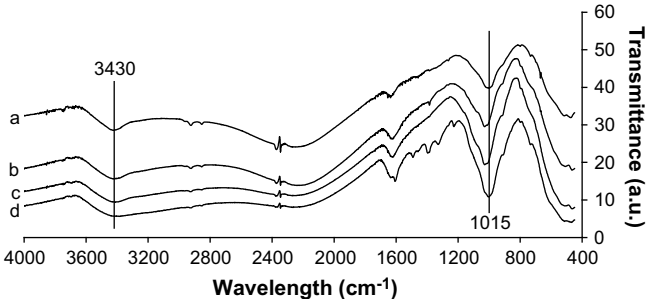


Fig. 2. Infrared spectra of: a) fresh MOMD and spent MOMD after adsorption of b) MB, c) RY and d) RB adsorption.

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