



## Dye removal using some surface modified silicate minerals



Selim K.A. \*, Youssef M.A., Abd El-Rahiem F.H., Hassan M.S.

Central Metallurgical Research and Development Institute, Cairo, Egypt

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### ABSTRACT

The objective of this work is to study the efficiency of some surface modified phyllosilicate minerals (bentonite and glauconite) in the removal of dyes from textile waste water. It is found that complete dye removal was achieved by using 10–25 g modified glauconite from solutions having a dye concentration of 10–50 mg/L. Adsorption data were modeled using Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherms. Adsorption capacities and optimum adsorption isotherms were predicted by linear regression method. The analysis of experimental isotherms showed that Langmuir isotherm reasonably fit the experimental data in the studied concentration range for the adsorption of dye onto glauconite mineral surface where Freundlich isotherm fit the experimental data for the adsorption of dye onto bentonite mineral surface.

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### 1. Introduction

The world-wide high level of production and use of dyes generates colored wastewaters, which give cause of environmental concern due to their impact on water bodies, and growing public concern due to their toxicity and carcinogenicity. Textile companies, dye manufacturing industries, paper and pulp mills, tanneries, electroplating factories, distilleries, food companies and a host of other industries discharge colored wastewater. As a matter of fact, the discharge of such effluents in the environment is worrying for both toxicological and esthetical reasons. The methods of color removal from industrial effluents include biological treatment, coagulation, floatation, adsorption, oxidation and hyper filtration. Among the treatment options, adsorption appears to have considerable potential for the removal of color from industrial effluents [1]. The process of adsorption has an edge over the other methods due to its sludge free clean operation and completely removed dyes, even from the diluted solution. The major advantage of an adsorption system for water pollution control are less investment in terms of both initial cost and land, simple design and easy operation, no effect by toxic substance, and superior for the removal of organic waste constituents as compared to the conventional biological treatment processes.

The physico-chemical properties of clay minerals are the basis for their technological applications. Such properties are closely linked to the mineral fabric, surface area, porosity, crystal

morphology, structure and composition. These properties can vary with thermal and mechanical treatment [2–6]. The above mentioned treatments could lead to loss of water, modification of the surface characteristics, porosity and partial destruction of structure [7]. Also, these treatments could lead to enhancement of some technologically important properties as adsorption capacity, catalyst support, and absorbency [8].

In this paper, different modification techniques have been tested to get the information on treating such effluents in this study. The composition and structure of the minerals were studied by X-ray Diffraction (XRD) and Fourier Transform IR (FTIR) spectroscopy. In addition, Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) were used for surface morphology observations and for evaluation of the modification efficiency.

### 2. Materials and methods

#### 2.1. Materials

The bentonite mineral was collected from Northern Coast of Egypt. About 85% of the collected sample is a high iron montmorillonite with some substitutions of  $\text{Al}^{3+}$  for  $\text{Si}^{4+}$ . The principle exchangeable cation is calcium. The main associated gangues are kaolin and quartz. Meanwhile, glauconite mineral was collected from new valley locality, Egypt and it consists of spherical, loaf-like or ellipsoidal, grass green aggregates (pellets) of glauconite in addition to quartz. Methyl violet dye used in this study was supplied by Air Products and Chemicals, Inc.

\* Corresponding author. Tel.: +20 22598716.

E-mail address: [rehamrashad33@gmail.com](mailto:rehamrashad33@gmail.com) (K.A. Selim).

## 2.2. Methods

### 2.2.1. Surface modification of the two minerals

Ca-montmorillonite sample was calcined at different temperature (600–900 °C) for 2 h. The calcined samples were treated with 25% (by weight) sulfuric acid for one hour at 98 °C [9]. The activated samples were washed several times until the removal of aluminum sulfate. After that, the samples were dried overnight at 60 °C.

Glaucanite sample was screened to the proper effective size (–850 +250 μm), followed by water soaking, (1:3) solid/liquid ratio, and shaken gently from time to time. The turbidity of water was measured from time to time to confirm the removal of clay from the glaucanite surface using a Turbidity Meter, HI 93703, Micro-processor, HANNA Instruments. The treatment was applied through a series of washing cycles.

### 2.2.2. Separation experiments

The separation experiment was carried out in glass column, 25 mm diameter and 50 cm height which filled with 10–15 g (5–7.5 cm height) of treated minerals. The column was saturated with water then 200 mL of dye solution was added at the top of the column at a flow rate of 10 mL/min. The initial dye concentration ranged from 10 to 100 mg/L was studied. The concentration of dye in the filtrate was determined through measurement of total organic carbon content in the supernatant using a 'Phoenix 8000' Total Carbon Analyzer, TOC. The average of three readings was taken as a measure for dye concentration. All the experiments were done at room temperature (25 ± 2 °C).

### 2.2.3. Characterization

A Philips PW 1730 powder X-ray diffractometer with Fe-filtered Co (K-alpha) run at 30 kV and 20 mA was used to examine Ca-montmorillonite and glaucanite minerals. Infrared vibrational spectra were recorded on a Nicolet Magna 750 Fourier-transform spectrometer. For each sample, 28 scans were accumulated over the 4000–400 cm<sup>-1</sup> spectral range employing the transmittance mode and a resolution of 4 cm<sup>-1</sup>. The pressed KBr disc employed for this purpose was prepared using 0.4 mg of sample and 200 mg of KBr. Selected samples were observed on fractured surface under a JSM-6400 scanning electron microscope (SEM) to examine the morphology of montmorillonite and glaucanite before and after treatment. A Laser particle size analyzer "FRITSCHE" model "Analytite 22" was employed for size analysis of samples before and after treatment. Quantachrome NOVA 1200 was used for measurement of specific surface area and porosity.

### 2.2.4. Adsorption equilibrium study

Batch adsorption experiments were carried out at room temperature (25 ± 2 °C). Exactly 100 mL of reactive dye solution of known initial concentration, (Ci) (10–100 mg/L) was shaken at the constant agitation speed (200 r/min) with a required dose of adsorbents (10–25 g) for a specific period of contact time (5–180 min) in a mechanical shaker, after noting down the initial pH of the solution to the optimum pH. The pH of the solutions was adjusted by adding either 1 M HCl or 1 M NaOH solution. After equilibrium, the final concentration (Ce) was measured. The percentage removal of dye was calculated using the following relationship:

$$\% \text{Removal of dye} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (1)$$

where Ci and Ce are the initial and final (equilibrium) concentrations of dye (mg/L), respectively [10].

The adsorption capacity, q<sub>e</sub> (mg/g), was calculated according to the following equation:

$$q_e = \frac{(C_o - C_e) v}{w} \quad (2)$$

where C<sub>o</sub> and C<sub>e</sub> are the initial and equilibrium solution concentrations (10<sup>-6</sup>), respectively; V the volume of the solution (L); W the weight of the sample (bentonite and glaucanite) in grams [11].

### 2.2.5. Adsorption isotherm models

The equilibrium adsorption of methyl violet dye onto bentonite and glaucanite minerals was analyzed using Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherms.

**2.2.5.1. Langmuir isotherm.** Langmuir model is the simplest theoretical model for monolayer adsorption onto a surface with finite number of identical sites [12–14]. The general Langmuir equation is as follows:

$$q_e = \frac{k_L C_e}{1 + a_L C_e} \quad (3)$$

When linearized, Eq.(2) becomes:

$$\frac{C_e}{q_e} = \frac{1}{k_L} + \frac{a_L C_e}{k_L} \quad (4)$$

where k<sub>L</sub> and a<sub>L</sub> are the equilibrium constants. Plotting C<sub>e</sub>/q<sub>e</sub> against C<sub>e</sub> yields a straight line with slope, a<sub>L</sub>/k<sub>L</sub>, and intercept 1/k<sub>L</sub>. The ratio a<sub>L</sub>/k<sub>L</sub> indicates the theoretical monolayer saturation capacity, Q<sub>o</sub>.

**2.2.5.2. Freundlich isotherm.** Freundlich expression is an empirical equation applicable to non-ideal sorption on heterogeneous surface as well as multilayer sorption. The model is given as

$$q_e = K_F C_e^{1/n} \quad (5)$$

Eq. (5) is linearized into logarithmic form for data fitting and parameter evaluation as follows:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (6)$$

By plotting log q<sub>e</sub> versus log C<sub>e</sub>, constant K<sub>F</sub> and exponent 1/n can be calculated.

**2.2.5.3. Temkin isotherm.** The Temkin isotherm has been used in the following form [15]:

$$q_e = \frac{RT}{b} \ln(AC_e) \quad (7)$$

Using Eq. (7) to analyze the sorption data, a plot of q<sub>e</sub> versus ln C<sub>e</sub> enables determination of constants A and b.

**2.2.5.4. Dubinin–Radushkevich isotherm.** This isotherm model was chosen to estimate the characteristic porosity of the biomass and the apparent energy of adsorption. The model is represented by the equation below:

$$q_e = q_D e^{(-B_D [RT \ln(1 + \frac{1}{C_e})]^2)} \quad (8)$$

where B<sub>D</sub> is related to the free energy of sorption per mole of the sorbate as it migrates to the surface of the biomass from infinite distance in the solution and q<sub>D</sub> is the Dubinin–Radushkevich isotherm constant related to the degree of sorbate sorption by the sorbent surface [16]. The linear form of Eq. (9) is given as:

$$\ln q_e = \ln q_D - 2B_D RT \ln \left( 1 + \frac{1}{C_e} \right) \quad (9)$$

## 3. Results and discussion

### 3.1. Minerals composition

Fig. 1 indicated that the Egyptian bentonitic clay was dominated by Ca-montmorillonite associated with traces of kaolinite

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