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Efficient removal of dye from industrial wastewater using modified-clay as an eco-friendly adsorbent: Characterization and adsorption isotherm

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Abstract

Removal of dye pollutants produced from industrial wastewater has become a major concern of the environmental engineers. In this study, bentonite clay was modified by HDTMA surfactant for an efficient adsorption of dye from industrial wastewater. The characterization of the modified-clay was performed by scanning electron microscope (SEM) equipped with an X-Ray energy dispersion spectrometer (EDAX), and Fourier Transform Infrared Spectroscopy (FTIR). The effect of operational parameters such as pH_{solution} , initial concentration and adsorbent dosage on the removal efficiency of dye from aqueous solution were examined. Adsorption isotherm models were evaluated to determine the adsorption mechanism. The characterization results showed that bentonite clay was successfully modified by CTAB. The maximum dye adsorption efficiency of 90% by modified clay (8 fold more than clay with our modification) was recorded at the optimal conditions of $pH = 5$, initial concentration = 50 mg/l and adsorbent dosage = 0.8 g/L. Based on the isotherm adsorption modeling, the Langmuir model ($q_m = 77$ mg/g, $K_L = 0.28$ mg/g, $R^2 = 0.984$) showed better match with the experimental data in comparison with Freundlich model ($q_m = 72$, $K_F = 21$, $n = 2.4$, $R^2 = 0.954$) which indicates that the adsorption mechanism was physically and monolayer.

Keywords: Industrial wastewater; Dye removal; Modified clay; Adsorption isotherm



1. Introduction

Water scarcity has led to the rise of reusing wastewater after treatment as an important resource in many parts of the world [1]. In recent years, industries that consume a significant amount of water, such as textiles, refineries, pulp, and paper, have faced severe restrictions [2]. Industries must create a novel technique that lowers the price of wastewater treatment to solve this issue [3]. Dyes manufactured artificially fall into five categories: Reactive, Azoic, Basic, Acidic and Direct. An Azo dye contains one or more Azo bridges (N=N) that connect the aromatic structure with one or more Azo groups [4]. An Azo dye shows inability to dye effectively remove which is mainly due to the presence of sulfur removed from wastewater using biological, chemical, and physical methods. This means that paint-contaminated water must be treated and purified before releasing to the environment.

Dye degradation using biological agents such as bacteria [5], fungi [6], and algae has been investigated previously [7]. Various chemical processes are used to remove dyes, including photocatalysis [8], and advanced oxidation process (AOP) [9]. Chemical methods are problematic due to their high cost, high energy consumption, and production of secondary pollutants [10]. Dye removal can be accomplished using various technologies, such as adsorption [11], coagulation and flocculation [12]. Among all the available treatment methods, adsorption has gained more attention due to its high efficiency, simplicity of management, and low cost [13]. However, significant factors influencing the application of industrial adsorption technologies consider the cost-treatment for wastewater pollution removal from aqueous solutions. So far, various adsorbents, including activated carbon, zeolite, polymers, and chitosan, has been evaluated to absorb industrial dyes from wastewater [14].

Raw bentonite which is freely available everywhere has been applied for dye adsorption but it has low non-organic compound adsorption [15]. Clay must be modified with surfactants (e. g. cationic surfactant such as CTAB) to improve its absorption capacity [16]. As a result of ion exchange, the surfactant cation is replaced with an inorganic cation, and the clay's hydrophilic properties are transformed into hydrophobic ones [16].

The main objective of the current study was to efficiently remove dye from wastewater by modifying clay with surfactant to gain an effective and eco-friendly adsorbent. First, the clay and modified-clay were characterized using SEM, EDX, and FTIR characteristics. Then, the effect of basic parameters such as pH, initial concentration, and adsorbent dosage on the performance of modified clay was investigated.

2. Material and methods

1-2- Material

The Na-bentonite used in this study is from Sigma-Aldrich. The cation exchange capacity (CEC) is 53 meq/100 g. The cationic surfactant selected for this study is $qm = 77$ mg/g hexadecyltrimethylammonium bromide (HDTMA) with a purity of 99%, which was obtained from Merck, Germany. To simulate dye wastewater, 100 mg of azo dye acid orange 7 (AO7) ($C_{16}H_{11}N_2NaO_4S$, 350.32 g/M, λ_{max} : 485 nm) was mixed with 1L of distilled water which was used as the main stock solution for all batch experiments (Figure 1). The structures of the AO7 and the CTAB surfactant are shown in Figure 1. Sodium chloride (NaCl) and hydrochloric acid (HCl) were used to adjust the initial pH of the solution. All the chemical materials were of analytical grade and purchased from Merck, Germany.

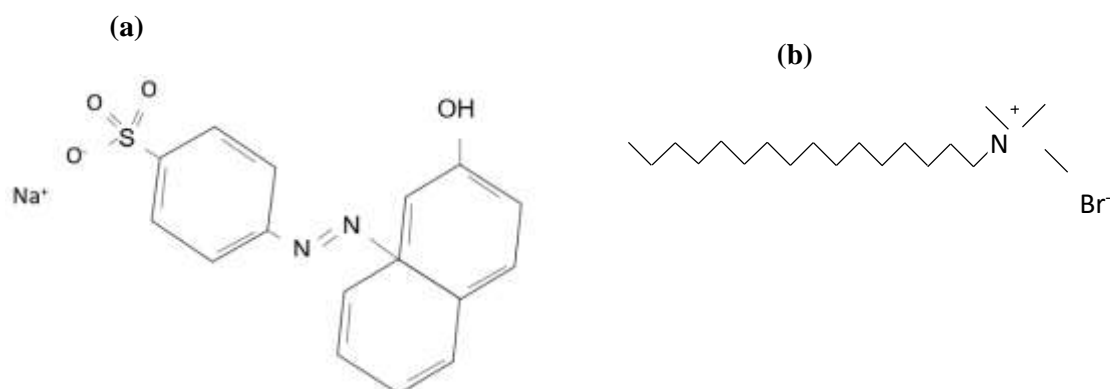


Figure 1: Structure of (a) AO7 and (b) HDTMA used in this study

2-2- Preparation of modified-clay

Na-bentonite was dispersed in 1 L of deionized water and then modified-clay prepared by adding CTAB 1.5 times of the cation exchange capacity of bentonite and stirred for 24h. After this treatment, the solid phase was separated by filtration and then washed with deionized water. Finally, it was crushed, ground, and sieved through a 200-mesh sieve and the samples were collected from under the sieve and dried in an oven at 70°C for 24 h prior to the use (Figure 2).

2-3- Batch experiment

Batch experiments were conducted to evaluate the performance of modified-clay in dye wastewater treatment. Effect of pH (5-9), initial concentration (30-70 mg/l) and adsorbent dosage (0.6-0.8 g/l) on the adsorption were then examined. In addition, the atomic absorption spectrophotometer (2800 UV/VIS UNICO, China) was used to determine the concentration of dye solution before and after the adsorption. The dye removal efficiency (R) was calculated using Eq. (1).

$$R(\%) = \frac{C_0 - C_f}{C_0} \quad (1)$$

where C_0 and C_f are the initial and final concentration of dye (mg/L), respectively.



Figure 2: Preparation of the modified clay



2-4- Adsorption isotherm

The isotherm data was fitted using the Langmuir model (Eq. 2), Freundlich model (Eq. 3). Generally, the Langmuir isotherm assumes monolayer adsorption, uniform adsorption energies onto the surface, and no interaction between adsorbed molecules. The Freundlich isotherm implies multilayer adsorption and is able to apply for heterogeneous adsorbent surfaces [17].

$$Q_e = \frac{K_L Q_m C_e}{1 + K_L C_e} \quad (2)$$

$$Q_e = K_F C_e^{1/n} \quad (3)$$

where Q_e , C_e , q_m and K_L are the mass of the adsorbent at equilibrium (mg.g⁻¹), the equilibrium dye concentration (mg/L) the maximum adsorption (mg/g) and the adsorption equilibrium constant (L.mg⁻¹). K_F is the Freundlich equilibrium constant (Which indicate the strength of the adsorptive bond) and n is the heterogeneity factor (which is lower when the surface is more heterogeneous).

2-5- Characterization

The morphology and chemical composition of the surface were tested using scanning electron microscope (SEM) model LEO1525 equipped with an EDAX detector. Fourier transform infrared (FTIR) spectra of the materials were obtained using Thermo Nicolet Nexus 670 spectrometer between 400 and 4000 cm⁻¹. The final concentration of dye was measured with UV/VIS detector (2800 UV/VIS UNICO, China). Absorbance measurements of the solution were performed at a wavelength of 485 nm.



3. Result

3-1- Characterization of the adsorbent

3-1-1- SEM-EDX analysis

FSEM-EDX analyses were conducted to compare the surface characteristics and elemental composition of Na-bentonite and the Na-bentonite modified with CTAB (at 100% CEC), and the findings are illustrated in Figure 3. Initially, Na-bentonite displayed an irregular and porous structure with an uneven distribution of particle sizes, as depicted in Figure 3a. However, post CTAB modification, the surface morphology of Na-bentonite underwent a transformation. As shown in Figure 3b, it exhibited a more crystalline and rugged surface with coarse pores, which is advantageous for enhancing the clay's adsorption capacity [18]. This alteration could be attributed to the reduction in the amorphous phase associated with Na-bentonite. Similar outcomes were reported by Kiransan et al. (2014) and Dinh et al. (2022) in their studies on the modification of montmorillonite and Na-bentonite with CTAB, respectively. The SEM analysis confirms the successful modification of Na-bentonite by CTAB [16], [18].

EDX analysis, in the case of Na-Bentonite, the weight percentages of various elements were as follows: O (59.39 wt%), Al (6.49 wt%), Si (27.36 wt%), Mg (1.4 wt%), Na (2.05 wt%), Cl (0.5 wt%), Ca (0.63 wt%), K (0.69 wt%), and Fe (1.51 wt%). However, for the organoclay, the weight percentages of these elements were different, with O (47.28 wt%), C (25 wt%), N (2.51 wt%), Al (4.45 wt%), Si (17.57 wt%), Mg (0.9 wt%), Na (0.25 wt%), Ca (0.28 wt%), K (0.4 wt%), and Fe (1.30 wt%). Notably, the presence of C (25 wt%) and N (2.51 wt%) in the organoclay, which were absent in Na-Bentonite, indicates the successful addition of CTAB to Na-Bentonite and suggests their importance for adsorption processes. The results indicated that the major elements were more uniformly distributed on the surface of the organoclay compared to Na-Bentonite. These SEM-EDX analyses support the conclusion that the modification of Na-Bentonite with CTAB significantly enhances the clay's adsorption capacity.

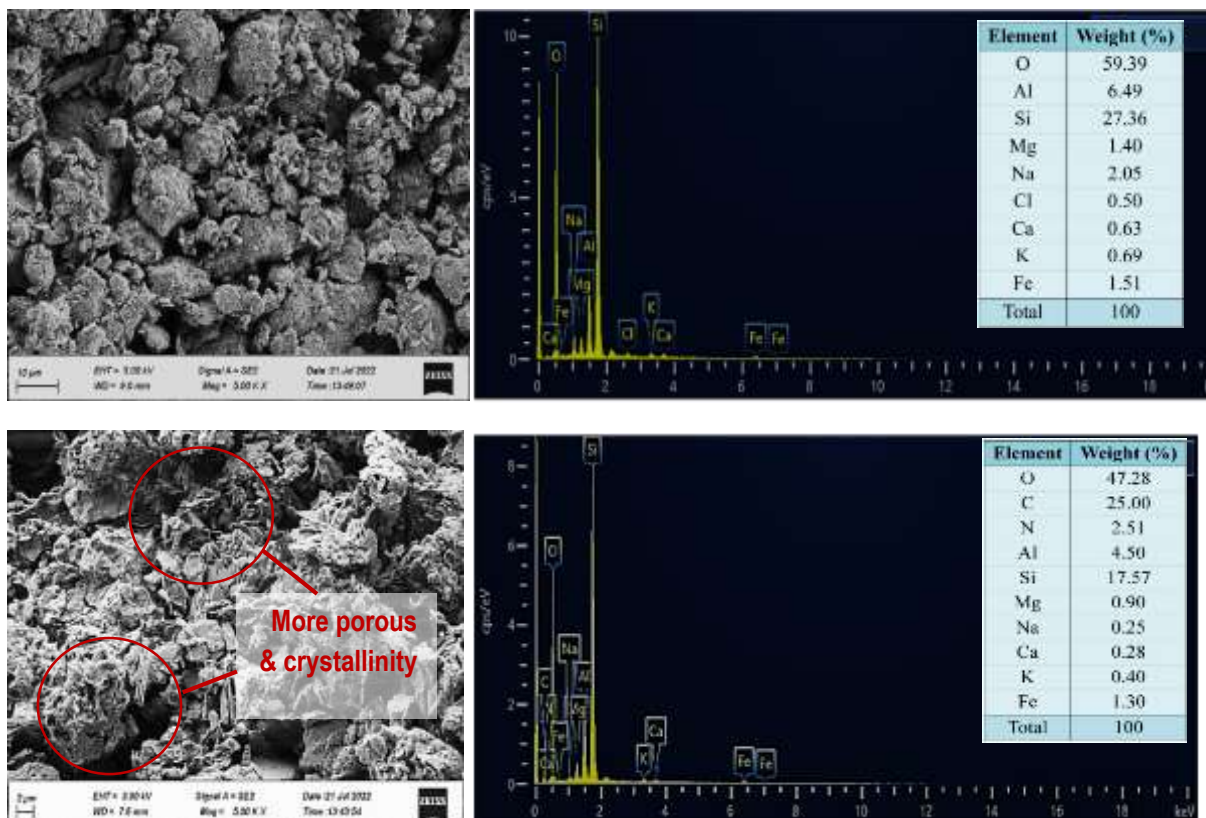


Figure 3: SEM images and EDX analysis of (a) the Na-Bentonite and (b) modified clay

3-1-2- FTIR analysis

FTIR analysis was performed to identify the surface functional groups present in both Na-bentonite and the organoclay, as depicted in Figure 4. A strong peak around a wavenumber of 3620 cm^{-1} was observed in both cases, attributed to the OH stretching vibrations of the structural hydroxyl group [19]. Additionally, the peak at 1640 cm^{-1} corresponded to the deformation of the OH bending vibration of water in both samples [20]. Notably, this peak was less pronounced in the case of the organoclay, indicating a shift from a hydrophilic to a hydrophobic surface on Na-bentonite [21]. Furthermore, a robust peak at around 1000 cm^{-1} , associated with Si-O stretching vibrations [20], was observed in both cases. Two new peaks emerged at approximately 2800 and 2900 cm^{-1} exclusively for the organoclay, which can be attributed to CH_2 and CH_3 groups, respectively [22]. Moreover, a distinct sharp peak at 1470 cm^{-1} was evident for the organoclay, indicating NH_4^+ bending vibrations and signifying the intercalation of surfactant molecules between the silica layers [18]. In summary, the FTIR analysis provided evidence that the modification of the clay was successfully achieved, leading to changes in surface functional groups and hydrophobicity.

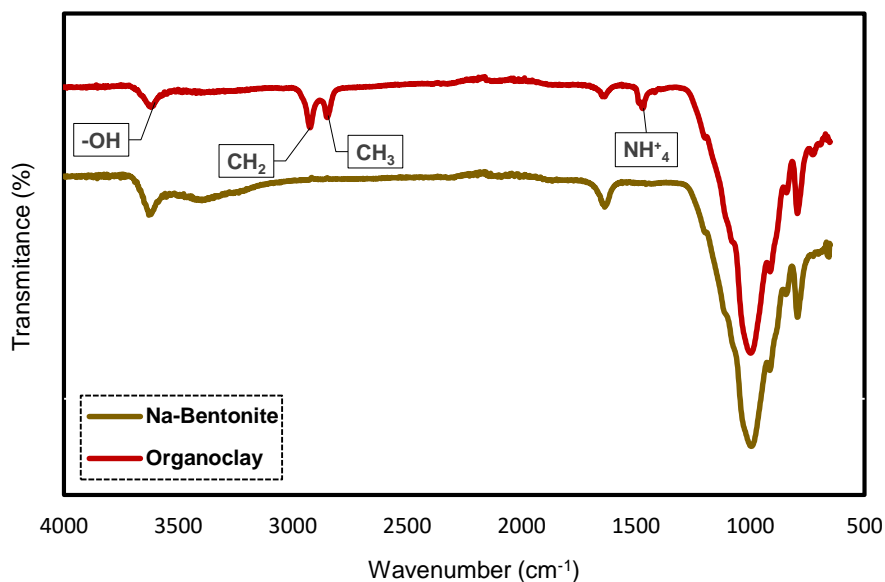


Figure 4: FTIR of (a) the Na-Bentonite and (b) modified-clay

3-2- Adsorption of dye

3-2-1 Effect of pH

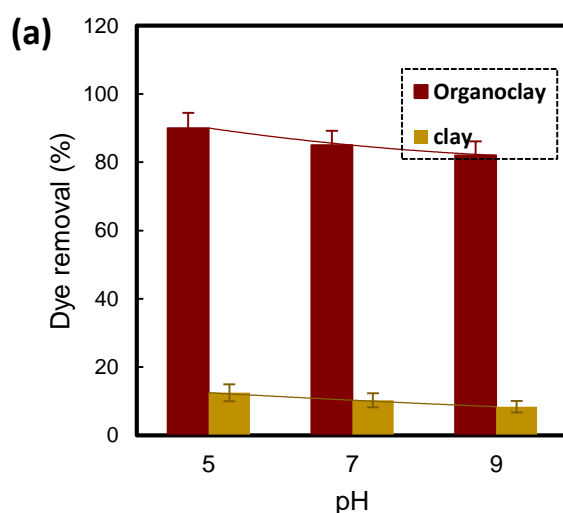
pH is a factor having an important effect on the dye removal because of the formation of dye in different types, as well as the surface charge of the adsorbent. Figure 5a shows that the adsorptive efficiency is decreased with increasing pH from 5 to 9. The maximum removal was obtained at pH = 5.0, which is smaller than the pH_{pzc} value. Therefore, the dye uptake of modified bentonite clay does not depend on the electrostatic attraction. Instead, It can be occurred via various mechanism. Numerous types of interactions are shown between adsorbent and adsorbate during adsorption process such as, adsorbent-dye, dye-dye, chemical bonds, hydrogen bonds, Vander Waal forces, ion exchange and Hydrophobic attraction [23]. At alkaline conditions the decrease in uptake capacity can be attributed to the competition between hydroxyl ions OH^- .

3-2-2- Effect of initial concentration

According to Figure 5b, when the initial concentration of dye increased, the dye removal also increased, but a higher initial dye concentration than an optimum concentration resulted in a lower removal efficiency. This behavior is attributed to the limitation of the available adsorption sites on the surface of organoclay. It was observed in Figure 5b, that an increment in organoclay dosages leads to an increase of in the optimum dye concentration from 40 to 55 mg/L, which is due to the presence of more unsaturated sites of organoclay for dye adsorption. The presence of an optimum value for initial pollutant concentration for maximum removal was previously reported [24], [25].

3-2-4- Effect of adsorbent dosage

Figure 5c shows the effects of changing the adsorbent dosage on dye adsorption. When the initial concentration is kept constant, increasing the adsorbent dosage increase the total surface area available for a typical adsorption process and can be considered an effective parameter for such a process. As the adsorbent dosage increases, the number of active sites and adsorption percentage increase. In contrast, a decrease in the driving force leads to partial saturation of the active sites of the adsorbent and decreases in the adsorption capacity.



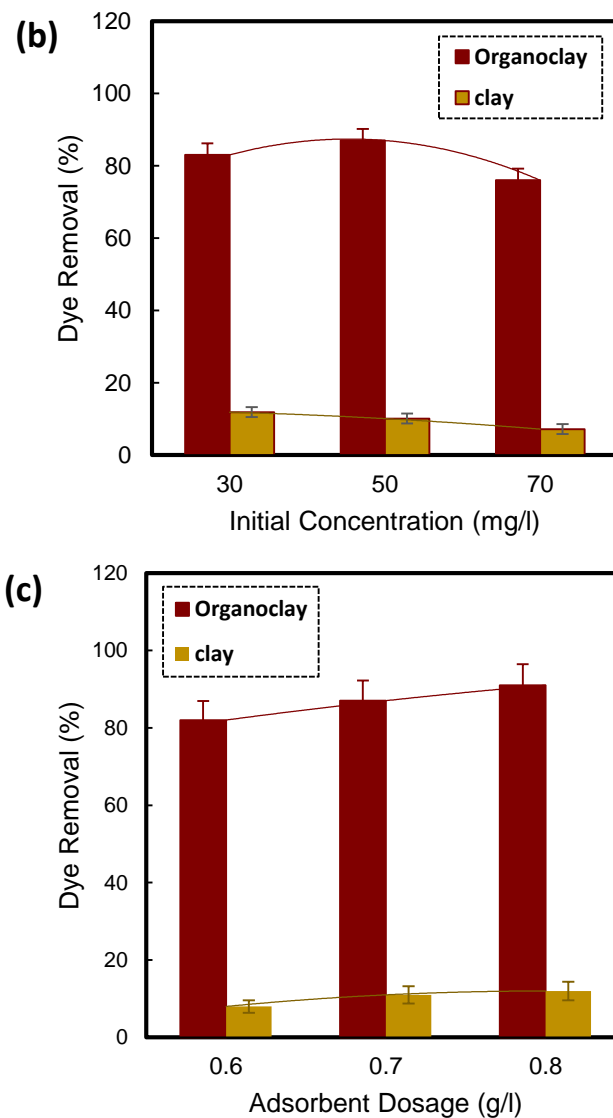


Figure 5: Effect of adsorbent dosage on Na-bentonite and modified-clay

3-4- Isotherm adsorption modeling

Figure 7 displays the amounts of the metal ions adsorbed by modified-clay as a function of dye concentrations in solution at equilibrium. The relationship between the adsorption capacity (solid phase) and concentration (liquid phase) at equilibrium was investigated using the Langmuir and Freundlich. The adsorption constants determined for the Langmuir and Freundlich equations are given in Table 1.

The experimental data were evaluated by the Langmuir and Freundlich isotherms models. It was observed that the adsorption capacity of organoclay increased as increasing the initial concentration of dye. The maximum adsorption capacity of dye to organoclay obtained as 78 mg/g at dye concentrations of 16 mg/L at equilibrium. As can be seen from Table 1, Langmuir model had a better fitting with the experimental data ($R^2 = 0.984$) compared to the Freundlich model ($R^2 = 0.954$), suggestion that dye removal by modified-clay was mainly through monolayer adsorption. Similar results of organically adsorption isotherms for dye wastewater were reported previously [26].

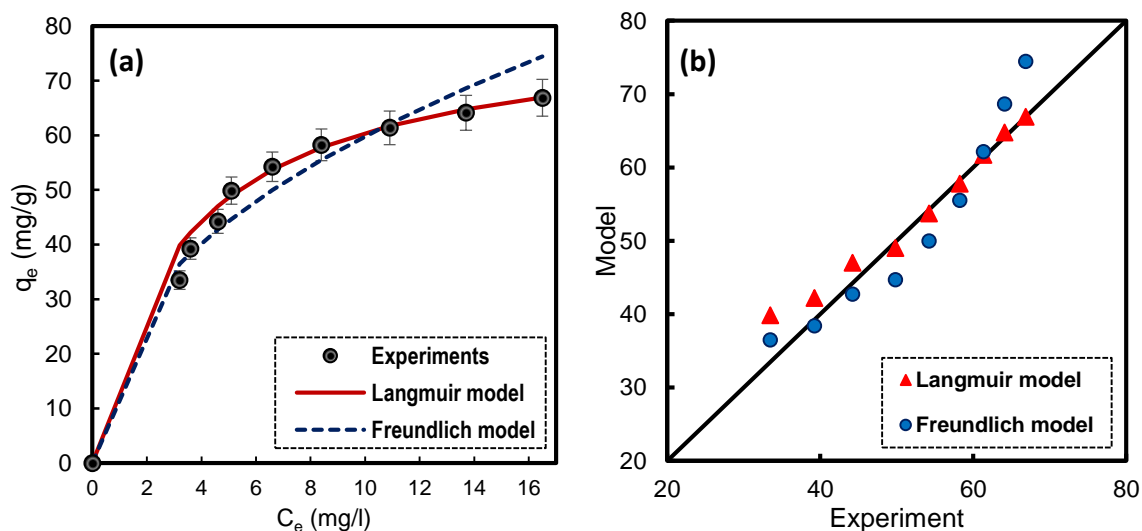


Figure 7: Isotherm adsorption of dye on modified-clay

Table 1: Parameters of isotherm adsorption models.

Model	q_e (mg/g)		Parameters		Error		
	Exp.	Model	K	n	R^2	RSME	
Isotherm	Langmuir	71.37	78.00	0.28	—	0.984	2.176
	Freundlich	69.1	72.51	21	2.3	0.954	16.55



4. Conclusion

In this study, the performance of modified-clay for dye wastewater treatment was investigated in terms of removal efficiency. SEM had clearly shown the modified-clay harsh surface and crystalline structure characteristic of them. FTIR after modification showed these various functional groups on the surface of the adsorbent, after modification the appearance of new peaks in intercalation of surfactant molecules between the silica layers. It was also found that the removal efficiency were sensitive to several operational parameters such as $\text{pH}_{\text{solution}}$, initial concentration and adsorbent dosage. The equilibrium adsorption results revealed that experimental data fitted well with Langmuir ($R^2=0.984$). According to the obtained results, it can be concluded that modified-clay could be used as an alternative adsorbent material for the removal of dye in aqueous solution.

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