

Adsorption of Acid Orange 33 Dye by Bentonite and Surfactant Modified Bentonite

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The batch adsorption of acid orange 33 from water onto Na-bentonite and CTAB-modified bentonite (CTAB-Ben) was studied. The effects of varying factors, such as initial dye concentration, adsorbent dosage, pH, contact time and temperature, which influence the adsorption capacities of Na-bentonite and CTAB-modified bentonite were investigated and discussed. The results revealed that the adsorption capacities of CTAB-modified bentonite toward acid orange 33 were higher than those of Na-bentonite. The maximum adsorption efficiency of CTAB-modified bentonite was 50 mg/g when acid orange 33 concentration, adsorbent dosage, reaction time and equilibrium pH were 50 mg/L, 80 mg, 90 min and 2, respectively. The isotherm study results for CTAB-Ben satisfactorily fit the Langmuir ($R^2 > 0.957$) models. The calculated values of the thermodynamic parameters ΔG , ΔH and ΔS showed that the adsorption was a spontaneous and endothermic reaction

Keywords: Acid orange 33, Bentonite, Surfactant Modified, Dye adsorption.

INTRODUCTION

High concentrations of synthetic dyes observed in the effluents from industries that manufacture and process paper, leather, plastic and textiles [1]. The textile industry comprises a major proportion of industries in developing countries. The quality of produced textile is strongly affects its export. Wastewater from textile plants, which contains synthetic dyes, is discharged into rivers and natural streams; which causes several environmental problems. Acid dyes are the most problematic type of synthetic dyes owing to their intense colors, acidic nature, high solubility in water and reactive characteristics. Acid dyes impart colours to large water bodies even at low concentrations, thus reducing both the scenic value and light penetration into the water bodies needed to support photosynthesis by aquatic flora in the water body. Since, numerous dyes are toxic or carcinogenic, hence, the removal of dyes from wastewater is essential and crucial [2].

Many approaches, such as precipitation, electrodialysis, adsorption, filtration, coagulation, oxidation and membrane separation have been developed for the decolorization of water [3]. However, adsorption has been considered among the most effective techniques for the removal of dyes from water. However, the prohibitive cost associated with adsorption has fueled the search for low-cost sorbents. Recently, low-cost materials such as fly ash [4], natural zeolite [5] and red mud [6] have received considerable attention from the view-point of wastewater treatment. Several modified clays have also reported to be economical sorbents that effectively decontaminate groundwater and surface water [7,8].

Bentonite comprises an octahedral alumina sheet flanked by two tetrahedral layers of silica. The permanent negative charge on bentonite results from the isomorphous replacement of Si⁴⁺ by Al³⁺ in the tetrahedral layers and of Al³⁺ by Mg²⁺ in the octahedral layer. This negative charge is balanced by the presence of replaceable cations (Ca²⁺ and Na⁺) in the lattice structure, which enhance the adsorbtion of cationic pollutants [9]. Several authors have reported the use of bentonite and organobentonite modified using a cationic surfactant for the elimination of acid dyes; high loading quantities of the surfactants are used for this purpose [7,10].

In present study, Na-bentonite (NB) was modified using CTAB, which resulted in the formation of a new adsorbent, namely CTAB-modified bentonite (CTAB-Ben). The modification improved the sorption capacity of Na-bentonite toward trace contaminants in acidic dyes. Na-Bentonite and CTAB-

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TABLE-1 CHEMICAL COMPOSITION OF SODIUM BENTONITE BY EDX								
SiO ₂	Al_2O_3	Fe_2O_3	Na ₂ O	MgO	CaO	TiO ₂	K ₂ O	SO ₃
75	19.7	2.23	1.212	0.85	0.46	0.06	2.7	0.17

modified bentonite were characterized through scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR) and specific surface area (SSA) analysis. The adsorption capacities of Na-bentonite and CTAB-modified bentonite were estimated by evaluating the removal of acid orange 33. The effects of pH, temperature, contact time, equilibrium concentration and adsorbent dosage on the adsorption were also investigated using a batch method.

EXPERIMENTAL

The Na-bentonite used in this investigation was obtained from Sigma-Aldrich with a the surface area was of 230 m²/g. The chemical component composition, determined by energy dispersive X-ray analysis and SEM of Na-bentonite sample is listed in Table-1. Acid orange 33 dye (m.w. 416.38 g/mol) and surfactant cetyltrimethylammonium bromide (CTAB) (m.w. 364.45g/mol) were purchased from Sigma Aldrich.

Synthesis of surfactant modified bentonite (CTAB-Ben): First, 10 g of Na-bentonite was dispersed in 100 mL of distilled water and stirred for 10 h at 250 rpm, then Na-bentonite was allowed to swell and achieve homogeneity. The concentrations of surfactant were fixed at 0.3, 0.7, 1.0, and 1.5 times the cation exchange capacity (CEC) of Na-bentonite. Accordingly, the required amount of CTAB was slowly added to the homogenous Na-bentonite. The mixture was stirred for 1 h and then the resulting CTAB-Ben was filtered and washed with distilled water for several times to remove excess salts. It was then dried at 90 °C in an oven. The product was ground and sieved using standard sieves to obtain a chemically modified adsorbent [11].

Characterization: The specific surface area (SSA) of the adsorbents was estimated using Brunauer-Emmet-Teller (BET) method in which the adsorption of liquid N2 at 196 K is assessed using a surface area analyzer (Quantachrome Instruments, Nova 2200e). The surface morphology of the samples was investigated using a scanning electron microscope (Zeiss Supra 55VP). The FTIR spectra of adsorbent samples before and after adsorption of acid orange 33 were run on a Perkin Elmer Model 1600 FTIR spectrophotometer (USA) by using KBr pellets. Each sample was finely ground with oven-dried spectroscopic grade KBr and pressed into a disc. All samples were dried at 90 °C in an oven. The spectra were then recorded at wavenumbers in the range 4000-400 cm⁻¹. A Mettler-Toledo pH meter (USA) was used to measure the initial pH adjusted using concentrated HCl and NaOH solutions. The CEC of bentonite (58.5 meq/100 g) was determined by ammonium acetate method [12]. Some of the characterization results of Nabentonite and CTAB-modified bentonite are listed in Table-2.

Adsorption studies: The batch sorption method was used on a 200 rpm shaking water bath (Julabo temperature and water bath, Germany). The effects of initial acid orange 33 concentration on the adsorption of capacities Na-bentonite and CTABmodified bentonite were investigated using 0.1 g of adsorbent and 50 mL acid orange 33 solution (initial concentrations: 5-200

TABLE-2 SOME CHARACTERIZATION PROPERTIES OF BENTONITE					
Parameters	Na-bentonite	CTAB-Ben			
Cation exchange capacity (meq/100 g)	58.5	79.0			
Specific surface area (m ² /g)	32.6	27.42			
pH in distilled water	6.8	6.5			

mg/L; pH: 6.5; temperature (T): 25 °C; contact time: 360 min) were used. The reaction mixtures were subjected to shake at 25 °C until an adsorption balance was achieved. The effect of pH on acid orange 33 elimination was examined by adjusting pH (2.0-10.0) of 50 mL samples of solutions of acid orange 33 (50 mg/L acid orange 33) by using 0.01 M NaOH or HCl solutions. The reaction mixtures also contained 0.1 g of adsorbent and the contact time and temperature were 360 min and 25 °C, respectively. The effect of temperature on acid orange 33 solutions (50 mg/L, pH 6.5) containing 0.1 g of adsorbent. The reaction mixture was incubated at different temperatures until an equilibrium was achieved.

The effect of contact time on acid orange 33 elimination was examined in 50 mL samples of acid orange 33 solutions (50 mg/L, pH 6.5) containing 0.1 g of adsorbent at 25 °C for a predetermined contact time. Subsequently, the samples were filtered and the absorbance of residual solution was measured to estimate the concentration of acid orange 33. The quantities of acid orange 33 removed by the sorbents Q_e and percent extracted (% E) can be calculated by the following equation:

$$Q_e = \frac{(C_o - C_e)V}{m}$$
(1)

In eqn. 1, Q_e = amount of dye adsorbed onto Na-bentonite or CTAB-Ben (mg/g), C_o = initial acid orange 33 concentration (mg/L), C_e = final value of acid orange 33 concentration in the adsorbate (mg/L), V = volume of acid orange 33 solution used (mL) and m = weight of bentonite or CTAB-Ben used (mg).

The concentration of acid orange 33 in the residual solution was analyzed spectrophotometrically by using a UV-visible spectrophotometer at 600 nm and the amount of adsorption Q_e was calculated according to eqn. 1.

RESULTS AND DISCUSSION

Optimization amount of CTAB-Ben: Fig. 1 shows the amount of bentonite (and value of CEC ratio) to be used as an intercalated mass with CTAB to increase the removal of acid orange 33. The CEC value of 0.7 produced was associated with the maximally adsorbing amount of 45 mg/g, and it provided a stable result compared with other CEC values. The peak intensity disappeared when the amount of CTAB was increased further, thus implying the formation of dominantly exfoliated nanostructure in CTAB-Ben at a CEC value of 0.8. At CEC values of 1.0 and 1.5, the stack density of organic cations in the interlayer space increased and the arrangement of organic cations changed



Fig. 1. Relationship between amount of bentonite and CTAB with amount of acid orange 33 adsorbed

because different layers have different charges [11]. The specific surface area is a crucial indicator of adsorption capacity. Table-2 shows that the decrease in surface area of the modified bentonite was caused due to the binding of CTAB to the surface and relatively fine intercalated pores of bentonite, which considerably reduced the total surface area [11-13].

The infrared spectra of CTAB-Ben before and after the adsorption of dye show strong absorption bands at 2932 and 3104 cm⁻¹, which can be attributed to the asymmetric and symmetric stretching vibrations, respectively of C-H groups (Fig. 2). The sharp peak observed at 1572 cm^{-1} can be explained by the bending vibrations of NH4⁺, thus supporting the intercalation of surfactant molecules between the silica layers. In case of the Na-bentonite, a band in the region of 1104-950cm⁻¹ (stretching vibration of Si-O groupsis represented by a sharp band at 1059 cm⁻¹ and a shoulder at approximately 1104 cm⁻¹) can be accounted for by perpendicular Si-O stretching. The shape of the band changed and its frequency shifted to a lower wavenumber after modification with CTAB. The absorption band of -OH and bending vibration of H₂O in Na-bentonite shifted significantly from 1650 cm⁻¹ (Na-bentonite) to a low frequency of 1640 cm⁻¹ after the modification (CTAB-Ben), which indicated that water molecules had been removed during the modification process. The removal of water also changed the hydrophobicity of Na-bentonite nanomaterial. These changes contributed to the formation of numerous adsorption





sites on CTAB-Ben and increased its adsorption capacity toward anionic dyes such as acid orange 33 [14].

SEM analysis was performed to evaluate the surface morphology of Na-bentonite and CTAB-Ben. As shown in Fig. 3, both sample types exhibited uneven structures and non-uniform size distributions. Na-bentonite originally comprised fine particles; however, modification with CTAB results in the formation of large particles and a coarse porous surface, which increase in the adsorption capacity of CTAB-Ben for the adsorption of acid orange 33.

Effect of initial dye concentration: In general, the adsorption of dye depended on the initial concentration of the dye. Fig. 4 shows the effect of initial acid orange 33 concentration on the adsorption capacity of Na-bentonite and CTAB-Ben. The Q_e of CTAB-Ben increased sharply from 5 to 75 mg/g when the initial concentration acid orange 33 increased from 10 to 400 mg/L. The increase in adsorption percentage was 5 to 75 % in CTAB-Ben, which is significantly more than that observed in Na-bentonite (1 to 5 mg/g). Thus, an increase in adsorbate concentrations resulted an increase in the gradient of adsorbent, which leads to an increase in the acid orange 33 diffusion rate. The results indicated that CTAB-Ben adsorbs acid orange 33 better than Na-bentonite.

Effect of pH: Na-bentonite and CTAB-Ben effectively removed acid orange 33 by adsorption from an aqueous solution (Fig. 5). The sorption capacity is relatively high at low pH values.



Fig. 3. SEM images: (left) Na-bentonite and (right) CTAB-Ben



Fig. 4. Effect of initial concentration on the adsorption of acid orange 33 on Na-bentonite and CTAB-Ben



Fig. 5. Effect of pH on the adsorption of acid orange 33 on Na-bentonite and CTAB-Ben

When the pH increases from 2 to 11, the amount of acid orange 33 adsorbed at equilibrium (Q_e) steadily decreases from 25 to 2 mg/g for CTAB-Ben and 2 to 0.2 mg/g for Na-bentonite, which is a decrease from 25 to 2 % and from 2 to 0.2 % in adsorption percentage in CTAB-Ben and Na-bentonite, respectively. The decrease in adsorption may be due to the repulsion between acidic dye molecules and the abundance of OH⁻ ions at higher pH values, which can competing with cationic surface of CTAB-Ben.

Effect of dosage of adsorbent: The effect of adsorbent dosage on the removal of acid orange 33 by Na-bentonite and CTAB-Ben is shown in Fig. 6. When the sorbent dose was increased from 10 to 100 mg, the percentage of dye removal by Na-bentonite and CTAB-Ben increased from 0.5 to 4 % and from 11 to 76 %, respectively. The adsorption of acid orange 33 increased rapidly with an increase in the amount of adsorbent from 10 to 80 mg. The adsorption was slightly enhanced from 90 to 100 mg owing to saturation of adsorption sites. This can be attributed mainly to the increase in sorbent surface area and availability of a relatively large number of adsorption sites.

Effect of temperature and time: The sorption capacity of Na-bentonite and CTAB- Ben increased rapidly owing to increase in contact time from 0 to 90 min; more than 80 % of the equilibrium sorption capacity for acid orange 33 is in equilibrium at 90 min. Consequently, 90 min was selected as the contact time for the sorption of acid orange 33 onto the aggregates.



Fig. 6. Effect of adsorbent dosage of CTAB-Ben and Na-bentonite on acid orange 33 removal

The effect of temperature on adsorption was investigated at 25, 45 and 65 °C and the corresponding results are shown in Fig. 7. When the temperature increased from 25 to 65 °C, the maximum amounts of acid orange 33 removed by Na-bentonite and CTAB-Ben increased from 0.5 to 4 mg/g and 5 to 50 mg/g, respectively. The increasing temperature may engender a swelling effect inside the adsorbent structure and facilitate penetration by the additional large dye molecule [15].



Fig. 7. Effect of adsorbent dosage of CTAB-Ben and Na-bentonite on acid orange 33 removal

Adsorption isotherms: The equilibrium adsorption isotherms can be explained using Langmuir isotherm and Freundlich isotherm equations [16], respectively:

$$\frac{C_{e}}{Q_{e}} = \frac{1}{K_{L}q_{m}} + \frac{1}{q_{m}} \times C_{e}$$
(2)

In eqn. 2, K_L is Langmuir equilibrium constant (L mg⁻¹), and q_m (mg g⁻¹) is the monolayer adsorption capacity; n and K_f (mg g⁻¹) are the Freundlich's constants. The Freundlich parameters can be obtained by the following linearized equation:

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e$$
(3)

ISOTHERMAL EQUATION ON THE ADSORPTION OF ACID ORANGE 33 ON CTAB-Ben AND Na-BENTONITE						
Parameters	Langmuir			Freundlich		
	$Q_e (mg/g)$	$K_L(L/g)$	\mathbb{R}^2	$K_{F}(mg^{1\text{-}1/n}L^{1/n}g^{\text{-}1})$	N (g/L)	\mathbb{R}^2
Na-bentonite	0.5	0.011	0.94	0.01	1.5	0.89
CTAB-Ben	22.8	0.0049	0.959	0.28	1.87	0.957

TABLE-3

By plotting a linear plot of log Q_e as a function of log C_e , the values of K_f and n can be determined using the intercept and slope of the graph. The isotherm parameters for the adsorption of acid orange 33 onto Na-bentonite and CTAB-Ben are given in Table-3. Langmuir adsorption model provides the best fit with regard to the experimentally obtained data for Na-bentonite and CTAB-Ben with ($R^2 > 0.93$). However, the Freundlich adsorption can be used also for modeling the equilibrium data ($R^2 > 0.90$). These results show that the surface of CTAB-Ben was enveloped by the monolayer of acid orange 33 molecules.

Adsorption kinetics: The value of calculated q_e exhibited a high level of concurrence with the experimental data and R^2 exceeded 0.986 for all adsorbents; thus, the second-order model is the best-fit model. The relatively rapid decrease in acid orange 33 concentration in a short time scale also supported the appropriateness of the second-order model. These findings imply a strong adsorption interaction between Na-bentonite and CTAB-Ben surfaces. A simple kinetic analysis of adsorption, which represented a pseudo-second-order equation, was used to fit experimental data in the present work as reported previously [17,18]. The linear form [19] can be given as follows:

$$\frac{\mathbf{t}}{\mathbf{q}_{\mathrm{t}}} = \frac{1}{(\mathbf{K}_{2}\mathbf{q}_{\mathrm{e}}^{2})} + \left(\frac{1}{\mathbf{q}_{\mathrm{e}}}\right)\mathbf{t} \tag{4}$$

In eqn. 4, k is the rate constant of pseudo-second-order adsorption (g mg⁻¹ min⁻¹) and q_e and q_t are the amount (mg g⁻¹) of acid orange 33 dye adsorbed at equilibrium and time t, respectively. The rate parameters, k and q_e , can be obtained directly from the intercept and slope of the plot of (t/q_t) against t (not shown), the results are listed in Table-4.

TABLE-4 KINETIC PARAMETERS FOR THE ADSORPTION OF ACID ORANGE 33 ONTO Na-BENTONITE AND CTAB-Ben						
Pseudo second order kinetic model						
	$\begin{array}{c cccc} \hline q_{e} exp. & Q_{e} calc. \\ (ppm) & (mg/g) & k_{2} (g/mg min) & R^{2} \end{array}$					
Na-bentonite	0.5	2.1	0.004	0.997		
CTAB-Ben	75	78	7.1368×10^{-5}	0.987		

Thermodynamic studies: Thermodynamic parameters were calculated using the change in the thermodynamic distribution coefficient (K_c) with change in temperature. The standard free energy change (ΔG°) was calculated using the following expression:

$$(\Delta G^{\circ}) = -RT \ln (K_c)$$
(5)

In eqn. 5, R is gas constant (8.314 J/mol/K), T is the absolute temperature (K) and K_c is the equilibrium constant. Standard enthalpy (Δ H^o) and entropy (Δ S^o) of adsorption are calculated using van't Hoff equation:

$$\log K_{c} = \frac{\Delta S^{\circ}}{2.303R} - \frac{\Delta H^{\circ}}{2.303RT}$$
(6)

The thermodynamic parameters (Table-5) showed that the values of ΔG° are negative for Na-bentonite and CTABmodified bentonite (CTAB-Ben). The negative values of ΔG° for all adsorbents at various temperatures indicated that the process is both feasible as well as spontaneous. In fact, the values of ΔG° decreased with an increase in temperature. This shows that the increase in temperature increased the rate of spontaneous adsorption. For all the sorbents, the positive values of ΔH° indicated the endothermic nature of adsorption process. Moreover, the positive values of ΔS° also indicated an increase in randomness during sorption process.

TABLE-5 THERMODYNAMIC PARAMETERS FOR THE ADSORPTION OF ACID ORANGE 33 ON Na-BENTONITE AND CTAB-Ben						
Material	Temp. (K)	-ΔG (KJ/mol)	ΔH (KJ/mol)	ΔS (KJ/mol)	r^2	
Na- bentonite	298 308 318 328	14.08 14.24 14.89 14.93	13.78	0.179	0.88	
CTAB- Ben	298 308 318 328	28.45 28.87 29.28 29.78	19.85	0.186	0.95	

Conclusion

Comparison results of adsorption percentage efficiency from this study and other similar results concerning adsorption of dyes onto bentonite is included in Table-6. Percentage ranges are because of differences in dye concentrations for each material used and various sampling origin of bentonite. The results of the present study revealed that CTAB-Ben can be a frequently

 TABLE-6

 ADSORPTION PERCENTAGE RANGES OF BENTONITE AND MODIFIED MATERIAL WITH DYES

Material and modification	Removal percentage range	Dye type	Ref.
Bentonite with dodecyltrimethylammonium (DTMA) bromide	50.26-76.23	Reactive blue 19	[7]
Bentonite with 1,6-diamino hexane (DAH)	56.9-66.7	Reactive blue 19	[20]
Bentonite with chitosan-CTAB	60.9-77.9	Weak acid scarlet	[21]
Bentonite with CTAB	51.56-99.94	Congo red	[11]
Bentonite with CTAB	52.74-74.89	Acid orange 7	[22]
Bentonite with CTAB	75-80	Acid orange 33	Present study

reliable adsorbent for application in the treatment of industrial wastewater and water polluted with dyes. The highest amount of acid orange 33 adsorbed among the list of effects for both types were found to increase in order CTAB-Ben (50 mg/g) > Na-bentonite (5 mg/g) which computes to CTAB-Ben (80 %)> Na-bentonite (5 %) adsorption percentage. Results showed that the adsorption capacity increases with the increase of initial acid orange 33 concentration, adsorbent dosage, temperature and pH. The adsorption of acid orange 33 onto CTAB-Ben reached equilibrium within about 90 min. The results could be well described by both Langmuir and Freundlich isotherm models. Thermodynamic reaction revealed that the adsorption process is endothermic and spontaneous.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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