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A. Safa Özcan[a]; Şerife Tetik[a]; Adnan Özcan[a]
[a] Department of Chemistry, Faculty of Science, Anadolu University, Eskişehir, Turkey

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Adsorption of Acid Dyes from Aqueous Solutions onto Sepiolite

A. Safa Özcan,* Şerife Tetik, and Adnan Özcan

Department of Chemistry, Faculty of Science, Anadolu University,
Eskişehir, Turkey

ABSTRACT

This research deals with an investigation of the adsorption of two acid dyes, namely Acid Red 57 (AR57) and Acid Blue 294 (AB294) onto sepiolite. Batch kinetics and isotherm studies were carried out. The results indicate that the adsorption of acid dyes obeys Freundlich isotherm and the second-order kinetics model. In addition, the effectiveness of sepiolite on adsorption of AR57 and AB294 from aqueous solution was studied as a function of time, pH, and temperature. Thermodynamic parameters for the adsorption of dyes were calculated and are discussed. The maximum removals of acid dyes was observed around 90% and 75% at pH = 2 for AR57 and AB294, respectively.

Key Words: Adsorption; Acid dyes; Clays; Isotherm; Sepiolite; Kinetics.

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*Correspondence: A. Safa Özcan, Department of Chemistry, Faculty of Science, Anadolu University, Yunusemre Campus, 26470 Eskişehir, Turkey; Fax: 90-222-3353616; E-mail: asozcan@anadolu.edu.tr.

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INTRODUCTION

Many industries, i.e., textile, leather, food, cosmetics, and paper, use dyes or pigments to dye their products. Colored dye wastes frequently contain heavy metals and other toxic organic pollutants, and the presence of dye colors may indicate the existence of toxicants. The removal of color from textile effluents is one of the challenging problems in the field of environmental chemistry, because of the difficulty of treating such waters by conventional chemical and biological treatment methods.[1] Removal of dyes using several of adsorbents by using an adsorption technique was found to be an efficient and economically cheap process.[2–12] Adsorption is a physicochemical wastewater treatment process in which dissolved molecules are attached to the surface of an adsorbent by physical/chemical forces. Activated carbon is the most widely used adsorbent for this process[13] because it has a high capacity for the adsorption of organics, but its use is limited compared with other cheaper natural adsorbents.[9,12,14]

Despite the fact that natural clays have a lower adsorption capacity than activated carbon, they are inexpensive and are also abundant materials. The use of clay to decolorize and refine oils, fats, and waxes has also been known.[4,9,15] A limited number of studies were carried out using sepiolite as an adsorbent[16–18] but none of them investigated adsorption of acid dyes on sepiolite. The removal of acid dyes by fixed beds of activated carbon was achieved by Walker and Weatherley.[19,20] The design of adsorption systems is the basic requirement as known adsorption isotherms obtained from equilibrium data.[20]

The clay mineral sepiolite with $Si_{12}O_{30}Mg_8(OH)_4(H_2O)_4\ 8H_2O$ as a unit cell formula is a silicate mineral with fibrous texture and a high surface area containing channels of molecular dimensions. Sepiolite is currently widely used in a variety of industrial applications as a raw material. This is mainly due to the existence of a high physicochemical activity on its surface. The fundamental structural sepiolite unit consists of two layers of tetrahedra and one layer of octahedron, which are not sheets but ribbons. They are linked, forming an open cavity, similar to that found in zeolites. In the structure, the linked Si–O chains face each other between the adjacent continuous oxygen sheets. The sheets formed by apices of the tetrahedra are completed by hydroxyls and magnesium in octahedral coordination, which tie the sheets together. Both arrangements leave channels in the structure between and parallel to the ribbons. At the edges of the ribbons, exposed hydroxyls are neutralized by protons, giving rise to bound water designated as OH_2. Water molecules, designated as zeolitic water, may enter the channels. The exchange capacity of sepiolite is less than that of smectites but their significant capacity to absorb organic molecules has lead to their use as an adsorbent.[21,22]

The objective of this research was to evaluate adsorption kinetics of two acid dyes, namely AR57 (C.I. No. 17053) and AB294 (unknown), using sepiolite as an adsorbent. In addition, adsorption equilibrium experiments were undertaken and the influence of pH and temperature were also investigated.

EXPERIMENTAL

Materials

Commercial grade dyes AR57 (Nylosan Red EBL) and AB294 (Nylosan Blue EBL) were supplied by Sandoz (Switzerland) and used without further purification. The adsorbent used in this study was a Turkish sepiolite. It was provided by Sivrihisar, Eskişehir. The moisture content was 9.10% and the sepiolite was used as such without further processing just after being sieved through a 63-μm sieve and drying at 120°C for 2 hours. The surface area and the cation exchange capacity (CEC) of sepiolite determined by the methylene blue adsorption method[23] are 216.3 $m^2\,g^{-1}$ and 276 mmol kg^{-1}, respectively.

Material Characterization

The chemical analysis of sepiolite was determined by using an energy dispersive x-ray spectrometer (EDX-LINK ISIS 3OO) attached to a scanning electron microscope (SEM-Cam Scan S4). The crystalline phases present in the sepiolite were determined by using an x-ray diffractometer (XRD-Rigaku Rint 2000) with CuK_α radiation.

Batch Adsorption

Batch pH studies were conducted with 50 mL of a 1000 ppm dye solution with 0.5 g of adsorbent for 1 h at a range of pH values from 1 to 11. The pH of the dye solution was adjusted by adding NaOH or HCl solution. Once the optimum pH was determined, kinetic studies were conducted at this pH for an increasing period of time, until no more dye was removed and equilibrium was achieved. After that time, the samples were filtered and their equilibrium concentrations were ascertained by spectrophotometric determination using a Shimadzu UV-2101PC spectrophotometer. The wavelength was selected so as to obtain maximum absorbance for each dyestuff and the λ_{max} values are 512.5 nm and 605.5 nm for AR57 and AB294, respectively.

Isotherm studies were also carried out with 50 mL of dye of different concentrations for 8 h to allow attainment of equilibrium at constant temperatures for three temperatures 20°C, 40°C, and 60°C.

RESULTS AND DISCUSSION

Chemical Composition of Sepiolite

The chemical composition of sepiolite obtained by using EDX analysis, given in Table 1, shows the presence of silica and magnesium oxide as major constituents along with traces of sodium, iron, and titanium oxides in the form of impurities. XRD results combined with EDX analysis show that most of the magnesium is in the form of sepiolite and calcium, with some magnesium in the form of dolomite. XRD also showed the presence of free quartz in sepiolite. It is, thus, expected that the adsorbate species will be removed mainly by SiO_2 and/or MgO.

pH Studies

Figure 1 indicates the changes in adsorption capacities of sepiolite for each dye with changes in pH. Upon dissolution anionic dyes release colored dye anions into the solution. The adsorption of these charged dye groups onto the adsorbent surface is primarily influenced by the surface change on the adsorbent, which in turn, is influenced by pH of the solution. At acidic pHs, the dissociation of the metal hydroxide complexes causes the surface to

Table 1. Chemical composition of sepiolite.

Constituents	Percentage by weight
SiO_2	51.17
MgO	25.50
CaO	7.52
Al_2O_3	1.04
K_2O	0.80
Na_2O	0.54
Fe_2O_3	0.40
TiO_2	0.05
Loss on ignition	12.98

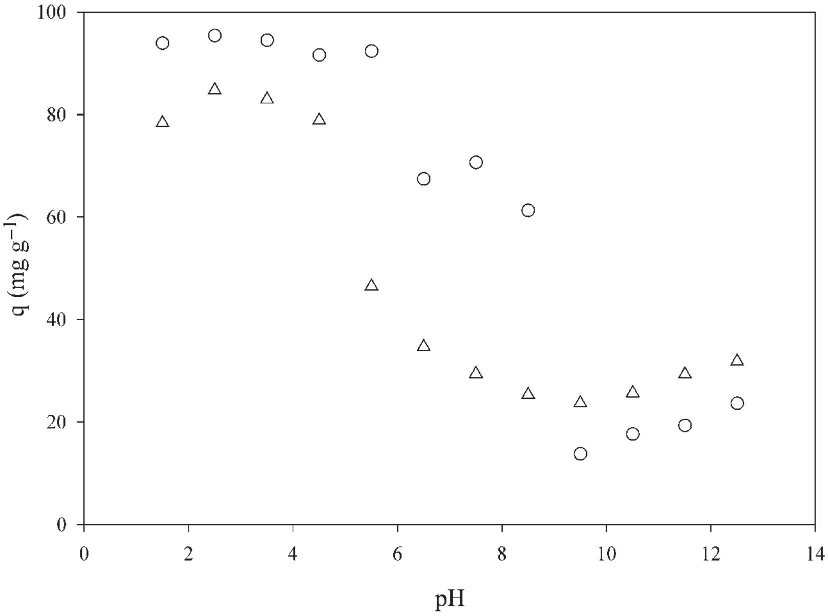

Figure 1. Effect of pH of the solution on adsorption of AR57 and AB294 onto sepiolite: ○, AR57; △, AB294.

become positively charged, which is very ideal for the removal of anionic dyes. The repulsion forces toward anionic dye groups is so reduced with a decreased in pH values and as a result, the maximum removals of AR57 and AB294 were observed at pH 2. The improvement in adsorption at a lower pH may be explained by the assumption that as the surface contains varying amounts of hydroxyl groups on the end groups of the surface, with an increase in the concentration of the hydrogen ion in the dye solution, the surface OH^- ions would get neutralized by protonation, and the cationic centers remaining on the surface or the diffusion of dye molecules in the vicinity of the adsorbent occurs. On the other hand, a diminished adsorption at higher pH may be due to the abundance of OH^- ions on the surface and, consequently, ionic repulsion between the negatively charged surface and the anionic dye molecules.

Kinetic Studies

Figures 2 and 3 indicate that the concentration of dye on the adsorbent surface increases with time and at some point in time, reaches a constant value

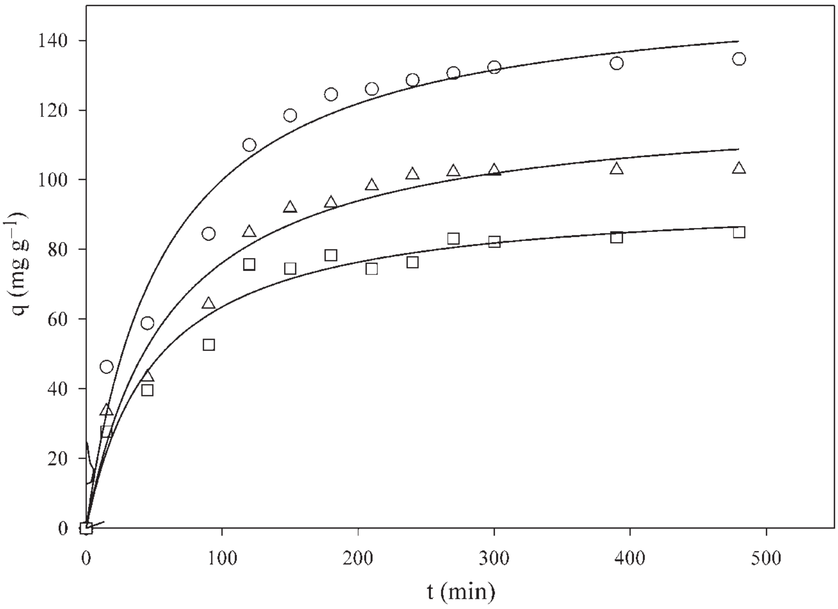

Figure 2. Kinetic studies on AR57 by sepiolite at various temperatures: ○, 20°C; △, 40°C; □, 60°C.

beyond which no more dye is removed from solution. At this point, the amount of dye being adsorbed onto the adsorbent is in a state of dynamic equilibrium with the amount of dye desorbing from the adsorbent. The amount of dye adsorbed at the equilibrium time reflects the maximum dye adsorption capacity of the particular adsorbent under these particular operating conditions. In this study, an equilibrium time of 8 h was chosen and found to be satisfactory for each dye. Sepiolite exhibited that dye removal percentages at 20°C and optimum pH are 90% and 75% for AR57 and AB294, respectively.

Rate Constant Studies

To find out whether the adsorption process followed first-order or second-order kinetics and to find the rate constants of the first-order sorption described by Eq. (1) is known as Lagergren rate equation cited by Trivedi et al.:[24]

$$\log(q_e - q) = \log q_e - \frac{k_1}{2.303}t \qquad (1)$$

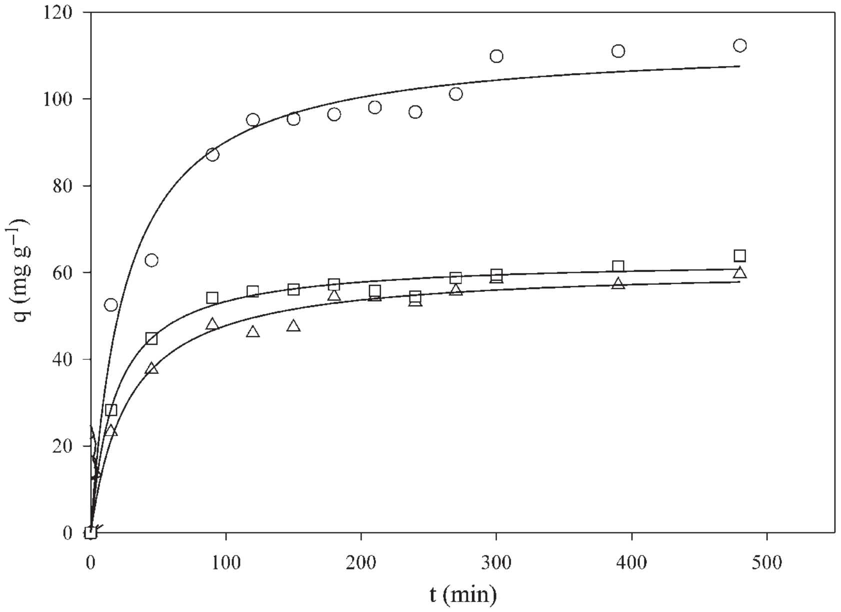

Figure 3. Kinetic studies on AB294 by sepiolite at various temperatures: ○, 20°C; △, 40°C; □, 60°C.

where q_e and q are the amounts of dye adsorbed at equilibrium and time t. The straight line plots of $\log(q_e - q)$ vs. t (see Figs. 4 and 5) show the fit of the Lagergren model. The intercept of the straight line plots should be equal to $\log q_e$. The experimental values for AR57 and AB294 did not show very much agreement with the plots of Lagergren. This can be considered that the first-order model does not appear to be able to characterize the adsorbent–adsorbate system in this case. The values of k_1 and correlation coefficients, r_1^2 values of dyes for different systems were determined from the respective plots and are given in Table 2.

The rate constant for second-order sorption may be obtained from the following equation:

$$\frac{dq_t}{dt} = k_2(q'_e - q_t)^2 \tag{2}$$

The kinetic rate equation can be rewritten as follows:

$$\frac{dq_t}{(q'_e - q_t)^2} = k_2\,dt \tag{3}$$

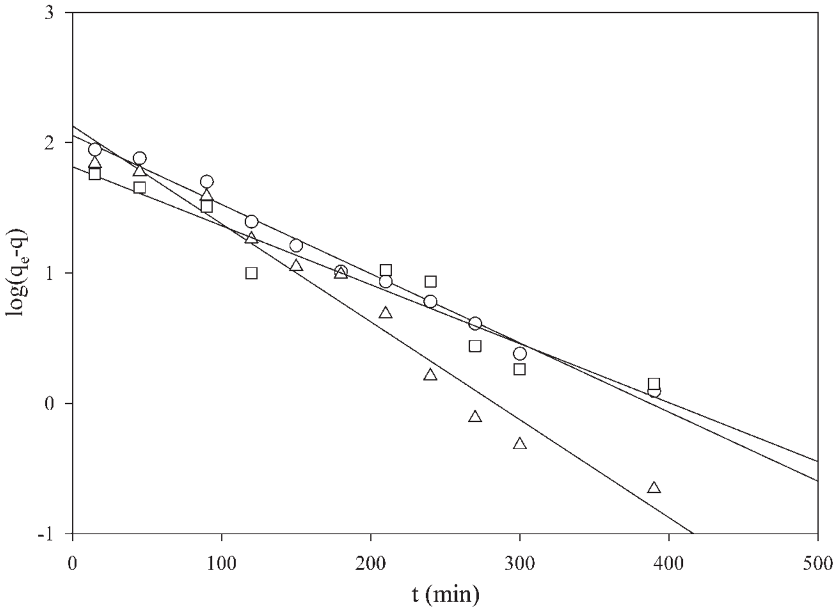

Figure 4. First-order plot for the adsorption of AR57 onto sepiolite from aqueous solution: ○, 20°C; △, 40°C; □, 60°C.

Integrating Eq. (3) for the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, gives:

$$\frac{1}{(q'_e - q_t)} - \frac{1}{q'_e} = k_2 t \tag{4}$$

which is an integrated rate law for a second-order reaction, where q'_e is the amount of dyes sorbed at equilibrium and k_2 is the equilibrium rate constant of the second-order sorption. Equation (4) can be rearranged to obtain:

$$\frac{t}{q_t} = \frac{1}{k_2 {q'_e}^2} + \frac{1}{q'_e} t \tag{5}$$

The equilibrium sorption capacity, q'_e, and the second-order rate constant, k_2, can be determined from the slope and intercept of a plot of t/q_t against t.

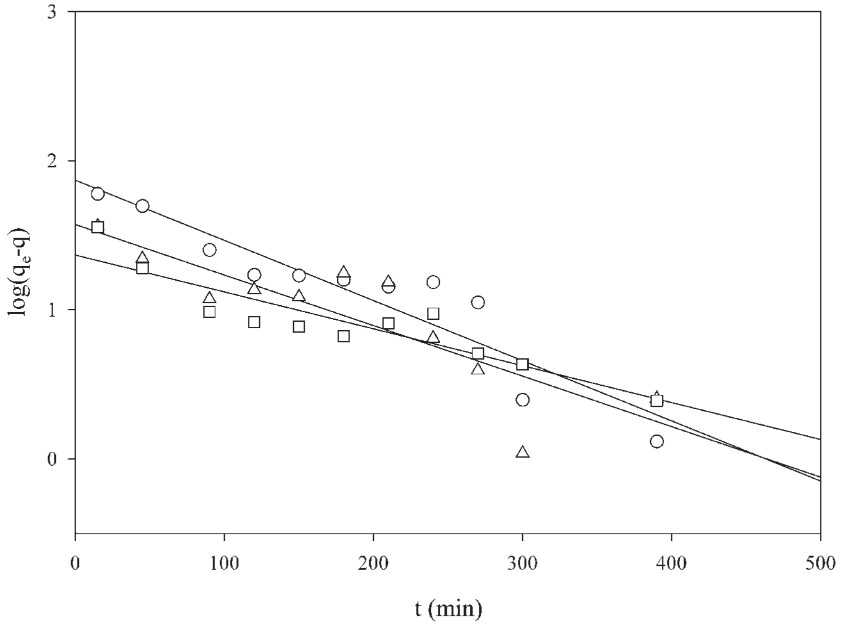

Figure 5. First-order plot for the adsorption of AB294 onto sepiolite from aqueous solution: ○, 20°C; △, 40°C; □, 60°C.

The values of k_2, q'_e, and the correlation coefficient, r_2^2, of dyes were calculated from the plots (see Figs. 6 and 7) and are given in Table 2.

The rate parameter of intraparticle diffusion can be defined as:

$$q = k_i\sqrt{t} \tag{6}$$

where, k_i is intraparticle diffusion rate constant. The k_i values under different temperatures were calculated from the slopes of the respective plots and are given in Table 2.[25,26] Figures 8 and 9 show the plot of q against the square root of t based on an intraparticle diffusion mechanism for AR57 and AB294 onto sepiolite. The fact that such experimentally determined plots give poor correlation with the linear plots expected from diffusion model theory, suggests that the process is not appropriate for the diffusion model.

The correlation coefficients, r_1^2, the first-order rate parameters, k_1, and sorption capacity, q_e, are compared with r_2^2, the second-order rate parameters, k_2, and sorption capacity, q'_e. The values of r_1^2, r_2^2, and r_i^2, for AR57 are found to

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Table 2. Kinetic parameters for the adsorption of AR57 and AB294 at various temperatures.

Dyes	T (°C)	q_e (Experimental data) (mg g^{-1})	q_e (Calculated data) (mg g^{-1})	k_1 ($\times 10^{-3}$ min^{-1})	r_1^2	q_e' (Calculated data) (mg g^{-1})	k_2 ($\times 10^{-4}$ g mg^{-1} min^{-1})	r_2^2	k_1 (mg g^{-1} min$^{-1/2}$)	r_i^2
AR57	20	134.6	113.6	12.2	0.987	151.4	1.36	0.992	5.33	0.831
	40	103.2	134.3	17.3	0.969	117.4	1.69	0.989	4.25	0.821
	60	84.9	65.2	10.4	0.921	94.3	2.26	0.992	3.24	0.805
AB294	20	112.3	73.8	9.29	0.871	120.3	2.10	0.993	3.28	0.874
	40	59.6	37.3	7.81	0.736	62.9	4.65	0.996	1.82	0.840
	60	63.9	23.3	5.70	0.828	65.4	6.05	0.995	1.56	0.753

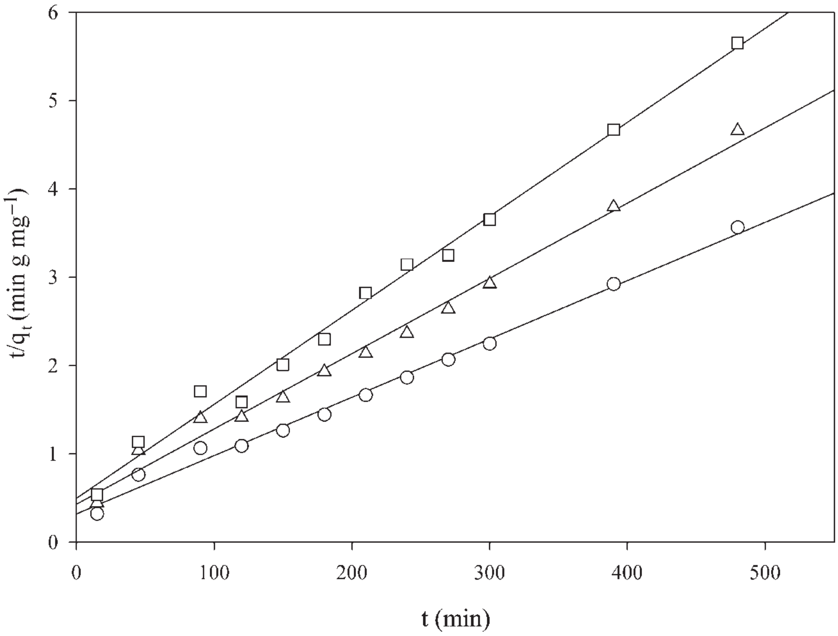

Figure 6. Second-order kinetic plot for the adsorption of AR57 onto sepiolite at various temperatures: ○, 20°C; △, 40°C; □, 60°C.

be higher than the values of AB294. The results for both of the sorption of AR57 and AB294 show that agreement between the calculated and experimental values for equilibrium sorption capacity is good for a second-order rate model. In the case of the sorption of AR57 and AB294 onto sepiolite, the correlations are the lowest for the intraparticle diffusion rate parameter, suggesting that the diffusion controlled mechanism does not predominate. This also supports that the sorption of AR57 and AB294 onto sepiolite follows a second-order model.

The values of the sorption capacity at equilibrium, q'_e, are decreased from 151.4 to 94.3 mg g^{-1} for AR57 and from 120.3 to 65.4 mg g^{-1} for AB294 when the temperature is increased from 20 to 60°C. This suggests that a low temperature favors acid dye removal by adsorption on sepiolite. Although the values of q'_e for the sorption of AR57 and AB294 decrease with increase in temperature, the values of the rate constant, k_2, were found to increase from 1.36×10^{-4} to 2.26×10^{-4} g mg^{-1} min^{-1} for the sorption of AR57 and from 2.10×10^{-4} to 6.05×10^{-4} g mg^{-1} min^{-1} for the sorption of AB294 for an increase in the solution temperature from 20 to 60°C.

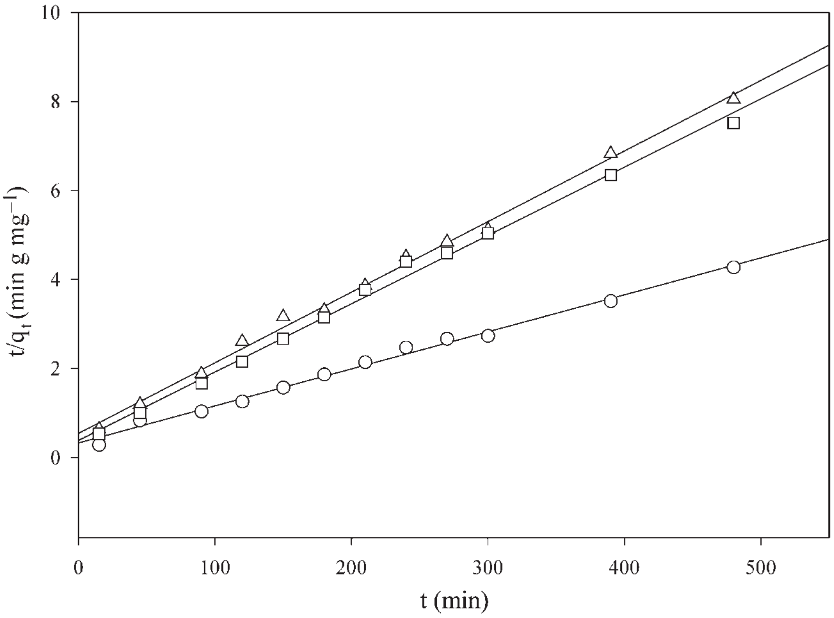

Figure 7. Second-order kinetic plot for the adsorption of AB294 onto sepiolite at various temperatures: ○, 20°C; △, 40°C; □, 60°C.

Adsorption Isotherm

The equilibrium data for adsorption of acid dyes on sepiolite fit the Freundlich adsorption isotherm according to the equation

$$q_e = K_F C_e{}^{1/n} \tag{7}$$

where q_e is the amount adsorbed in mg g^{-1}, K_F and n are Freundlich constants, and C_e is the equilibrium concentration of the dye solution. The plots of q_e vs. C_e (Figs. 10 and 11) indicate that the adsorption isotherms of AR57 and AB294 onto sepiolite and for the present system exhibit multiple sites available for adsorption on a rough, inhomogeneous surface. A plot of $\ln q_e$ vs. $\ln C_e$ enables the empirical constants K_F and $1/n$ to be determined from the intercept and the slope of the linear regression. The values of the constants are given in Table 3.

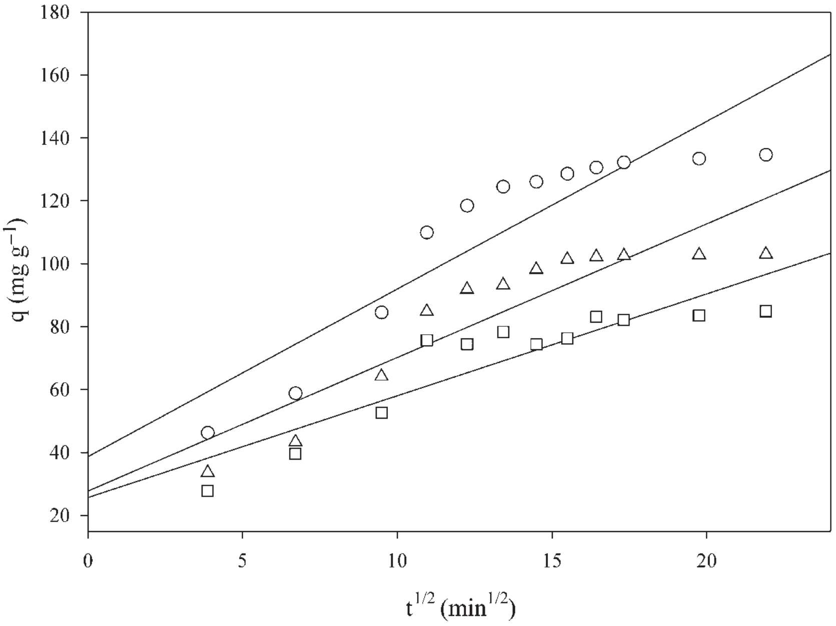

Figure 8. Intraparticle diffusion plot for the adsorption of AR57 on sepiolite: ○, 20°C; △, 40°C; □, 60°C.

Thermodynamic Parameters

The mechanism of adsorption from dye solution consists of four steps: (1) migration of dye molecules from the bulk solution to the boundary layer to the surface of the adsorbent; (2) diffusion through the boundary layer to the surface of the sepiolite; (3) adsorption at site; and (4) intraparticle diffusion into the interior of the sepiolite.[5] Although a review of published research indicated that the majority of the adsorption studies can be represented by a first-order rate equation,[27] in this work, the second-order equation was applied for the behavior over the whole range adsorption process. The temperature dependence of the second-order reaction can be fit to the Arrhenius equation;

$$\ln k_2 = \ln A - \frac{E_a}{RT} \tag{8}$$

where E_a is the Arrhenius activation energy and A is the Arrhenius factor. The activation energy was determined from the slope of linear plot of $\ln k_2$ vs. $1/T$

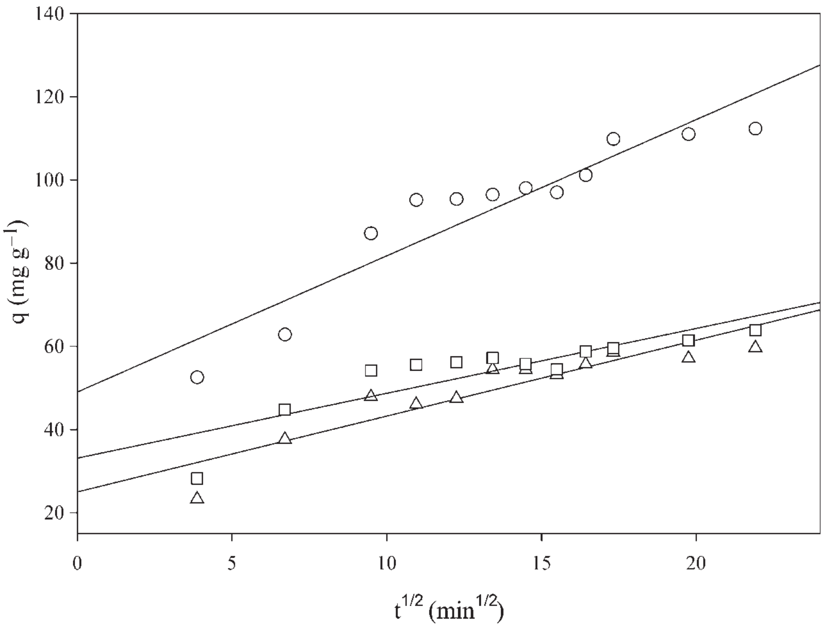

Figure 9. Intraparticle diffusion plot for the adsorption of AB294 on sepiolite: ○, 20°C; △, 40°C; □, 60°C.

(Fig. 12). The corresponding activation energies were calculated from the slope of linear plot for the adsorption of AR57 and AB294. The results obtained are 10.28 kJ mol^{-1} and 21.72 kJ mol^{-1}, respectively. The adsorption mechanism (i.e., chemical or physical) is often an important indicator to describe the type of interactions between the adsorbate and adsorbent. If adsorption decreases with increasing temperature, it may be indicative of physical adsorption and the reverse is generally true for chemisorption.

The changes in the free energy (ΔG^*), enthalpy (ΔH^*), and entropy (ΔS^*) of activation may be calculated by following equations and are given in Table 4:

$$k_2 = \frac{k_B T}{h} K^* \tag{9}$$

$$\Delta G^* = -RT \ln K^* \tag{10}$$

$$\Delta H = E_a - RT \tag{11}$$

$$\Delta S^* = \frac{\Delta H^* - \Delta G^*}{T} \tag{12}$$

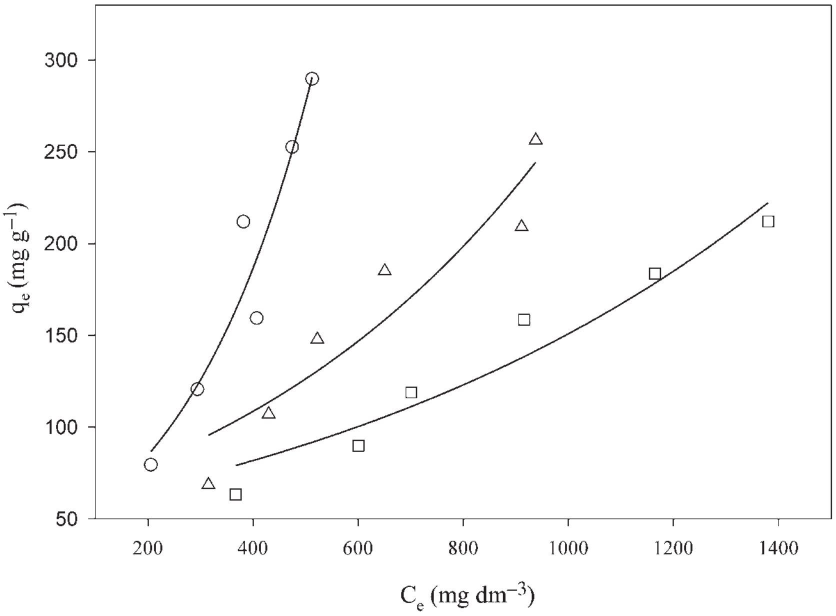

Figure 10. Adsorption isotherms of AR57 onto sepiolite: ○, 20°C; △, 40°C; □, 60°C.

where k_B and h are Boltzmann's and Planck's constants, respectively; R is the gas constant, and K^* is the equilibrium constant at temperature T.[28] In principle, K^* in Eqs. (9) and (10) should be in terms of molar unit ($\mathrm{dm^3\,mol^{-1}}$). The values of ΔG^* and ΔS^* can be calculated only if the molar mass of the dye is known. Since the dyes used here are commercial products, their molecular weights are mostly unavailable. Therefore ΔG^* and ΔS^* cannot be calculated for AB294. It is expected that ΔG^* is negative, indicating that the process is spontaneous. For example, ΔG^* is $76.9\,\mathrm{kJ\,mol^{-1}}$ for AR57 at 20°C since it has a molar mass of $526\,\mathrm{g\,mol^{-1}}$.

The positive enthalpy can be understood since the net adsorption process corresponds to the results of two simple processes. Firstly, the exchange of the molecules of solvent (water) previously adsorbed and then the adsorption of the molecules of dyes. Each molecule of dye has to displace more than one molecule of the solvent. The net results correspond to an endothermic process.[29] The enthalpy change due to chemisorption (41.8 to $125.4\,\mathrm{kJ\,mol^{-1}}$) is larger than that due to physisorption ($<41.8\,\mathrm{kJ\,mol^{-1}}$).[4,29] It can be suggested that the adsorption of AR57 and AB294 onto sepiolite observed was mainly physical.

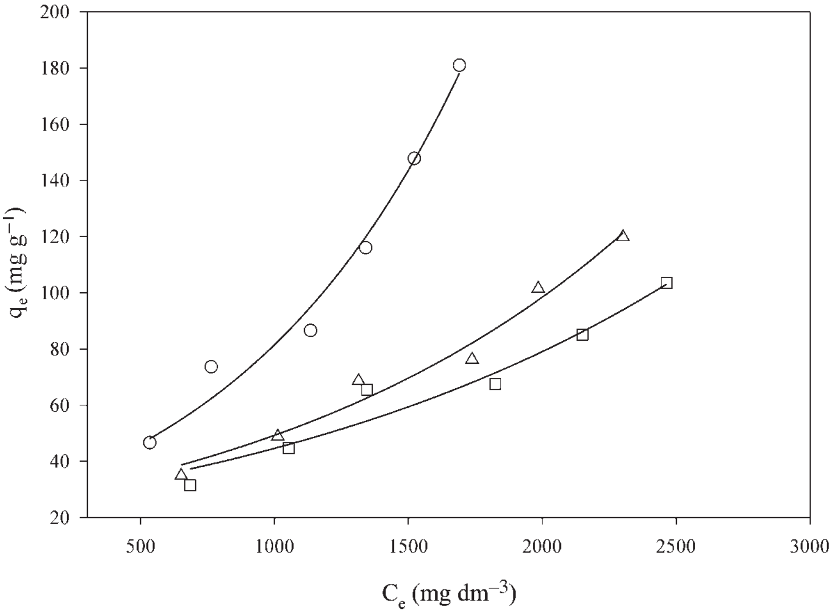

Figure 11. Adsorption isotherms of AB294 onto sepiolite: ○, 20°C; △, 40°C; □, 60°C.

A negative entropy change found in this study may be understood in terms of restriction of the movement of the molecules two dimensions on the surface, as against three dimensions in the bulk. The fact that the activation entropy is negative this is a result of interactions between AR57 and sepiolite, as expected.

Table 3. Freundlich isotherm constants for the adsorption of AR57 and AB294.

Dyes	T (°C)	K_F ($dm^3\,g^{-1}$)	n	r^2
AR57	20	0.047	0.720	0.933
	40	0.138	0.912	0.953
	60	0.237	1.06	0.984
AB294	20	0.051	0.922	0.951
	40	0.066	1.04	0.975
	60	0.099	1.13	0.964

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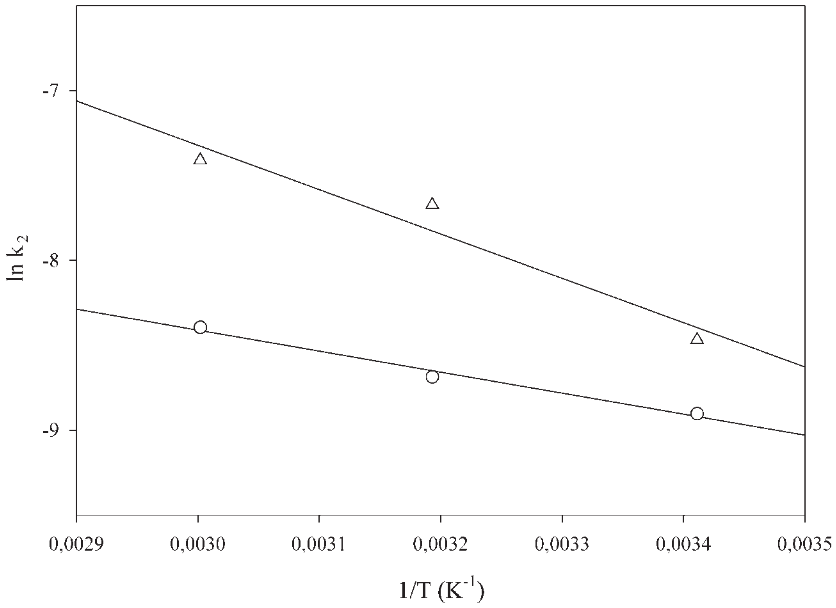

Figure 12. Arrhenius plots for the adsorption of AR57 and AB294 on sepiolite: ○, AR57; △, AB294.

CONCLUSION

Sepiolite was shown to be an effective and economical material for the adsorption of acid dyes from aqueous solutions. The surface charge on the adsorbent and the solution pH play a significant role in influencing the capacity of an adsorbent toward dye ions. Having an excess positive charge on

Table 4. Thermodynamic parameters calculated with the second-order rate constant.

Dyes	Temperature (°C)	ΔG^* (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J K^{-1} mol^{-1})
AR57	20	76.9	7.84	−235.7
	40	81.8	7.67	−236.6
	60	86.4	7.51	−236.7
AB294	20	—	19.3	—
	40	—	19.1	—
	60	—	18.9	—

the surface, sepiolite has a greater capacity to adsorb acid dyes. The experimental adsorption capacities for AR57 and AB294 at 20°C and pH 2 were found to be 134.6 mg g^{-1} and 112.3 mg g^{-1}, respectively. Silica and magnesium oxide are the major components. Despite the fact that most of the magnesium is in the sepiolite form, calcium and some of the magnesium are in the form of dolomite. The failure to analyze the rate data satisfactorily for AB294 suggest that the adsorption process is quite complex. For the adsorption of AR57 and AB294 onto sepiolite, the first-order chemical reaction does not provide the best fit model; it can be described best by the second-order chemical reaction kinetics, which provide the best correlation of the data in most cases. For the systems, the Freundlich equation gives a reasonably good fit to the isotherm data, rather than the Langmuir isotherm model, especially for AR57. The process being endothermic and indicating the nature physisorption, the enthalpies of adsorption at 20°C were obtained 7.84 and 19.3 kJ mol^{-1} for AR57 and AB294, respectively. An increase in the uptakes of AB294 at 60°C may be a result of the enhanced the number of active sites available for adsorption.

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