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Removal of reactive blue 221 and acid blue 62 anionic dyes from aqueous solutions by sepiolite

Mahir Alkan, Sermet Çelikçapa, Özkan Demirbaş, Mehmet Doğan*

University of Balikesir, Faculty of Science and Literature, Department of Chemistry, 10100 Balikesir, Turkey

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Abstract

The removal of reactive blue 221 and acid blue 62 anionic dyes onto sepiolite from aqueous solutions has been investigated using parameters such as calcination temperature, pH, ionic strength and temperature. After 200 °C calcination temperature, the specific surface area of sepiolite decreased with increasing calcination temperature. The amount adsorbed of reactive blue 221 and acid blue 62 on sepiolite increased with increasing ionic strength and temperature, and decreasing pH. The sepiolite sample calcinated at 200 °C has a maximum adsorption capacity. However, calcination at higher temperature caused a decrease in the amount adsorbed of dye. It was found that the Freundlich isotherm appears to fit the isotherm data better than the Langmuir isotherm. Furthermore, it can be said that sepiolite has a considerable potential as an adsorbent for dyestuffs in a commercial systems.

Keywords: Sepiolite; Anionic dyes; Adsorption; Freundlich isotherm

1. Introduction

The removal of contaminants from industrial wastewaters is currently of great importance in recent years. Precipitation, ion exchange, solvent extraction, biosorption, filtration, electrochemical treatment and adsorption on oxides are the conventional methods for the removal of contaminants from aqueous solutions [1]. These methods are either expensive or cannot cope with high concentrations of contaminants. All these methods have significant disadvantages such as incomplete ion removal, high-energy requirements and production of toxic sludge or other waste products that require further disposal. Therefore, these methods do not suit the needs of developing countries. The adsorption process is used especially in the water treatment field. Activated carbon, in granular or powdered form, is the most widely used adsorbent. It has a good capacity for adsorption of organic molecules. In spite of this, it suffers a number of disadvantages. Activated carbon is quite expensive and the higher the quality the greater the cost. Regeneration produces additional effluent and results in considerable loss (10–15%) of the adsorbent [2]. This has led many workers to search for cheaper substitutes. Studies so far have focused on adsorbents such as alumina, magnetite, pyrolusite, rutile, zirconia, hydrous manganese oxide, silica, goethite, heamatite, amorphous ferric oxide, bentonite, sphalerite, anatase, red mud, mica, illite, kaolinite and clay [1]. The cost of the adsorbent minerals indicates that cheaper and easily obtainable unconventional adsorbents should also be studied for the removal of pollutants from water. The need for economically viable industrial and wastewater processes that protect the environment and public health has led to research into processes using alternative water. Suitable candidate would be sepiolite.

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* Corresponding author. Tel.: +90 266 2491011; fax: +90 266 2491012.

E-mail addresses: malkan@balikesir.edu.tr (M. Alkan), ozkan@balikesir.edu.tr (Ö. Demirbaş), mdogan@balikesir.edu.tr (M. Doğan).

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Nomenclature

Q_m	Monolayer capacity of the adsorbent, mol g^{-1}
K	Adsorption constant
C_e	Equilibrium dye concentration in solution, mol L^{-1}
Q_e	Equilibrium dye concentration on adsorbent, mol g^{-1}
K_F	Freundlich constant
n	Freundlich isotherm exponent
T	Temperature, K
I	Ionic strength, mol L^{-1}
W	Mass of adsorbent, g
V	Volume of aqueous solution to be treated, L
C_0	Initial dye concentration in aqueous solution, mol L^{-1}
R^2	Regression coefficient

Sepiolite, as an adsorbent, may be a good alternative to these systems. Sepiolite is a natural hydrated magnesium silicate clay mineral, $(Si_{12})(Mg_8)O_{30}(OH_6)(OH_2)_4 \cdot 8H_2O$. Structurally, it is formed by blocks and channels extending in the fibre direction. Each structural block is composed of two tetrahedral silica sheets and a central octahedral sheet containing magnesium. Due to the discontinuity of the external silica sheet a significant number of silanol (Si–OH) groups are present at the surface of these minerals [3]. Some investigators have studied sorptive properties of organic compounds, cationic surfactants and metal ions with sepiolite. These include adsorption of thiram from aqueous solution on active carbon and sepiolite [4], the infrared study of the thermal treatment of sepiolite and palygorskite saturated with organic acids [5], the removal of aromatic amines on sepiolite from aqueous solutions [6], the adsorption mechanism of cationic surfactants onto acid- and heat-activated sepiolites [7], the adsorption of cobalt from aqueous solutions onto sepiolite [8], and the spectroscopy of rhodamine 6G adsorbed on sepiolite from aqueous solutions [3]. As can be seen above, only limited number of studies on the use of sepiolite as an adsorbent has been found in the literature.

Therefore, the aim of the present work is to study the adsorption onto sepiolite of reactive blue 221 and acid blue 62 anionic dyes and to discuss the effects of parameters such as calcination temperature, pH, ionic strength and temperature on the adsorption capacity. The results obtained have been applied to a single batch design for the removal of reactive blue 221 and acid blue 62 anionic dyes from aqueous media by using sepiolite.

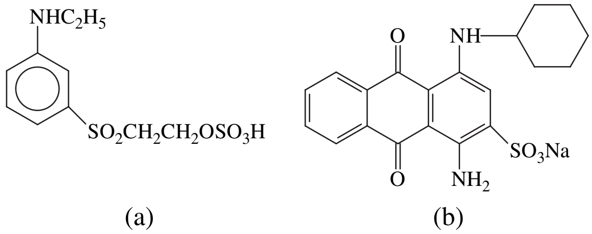

Fig. 1. The structures of dyes: (a) reactive blue 221 and (b) acid blue 62.

2. Materials and methods

2.1. Material

Sepiolite sample was obtained from Aktaş Lületaşı Co. (Eskişehir, Turkey). Reactive blue 221 and acid blue 62 were obtained from Setaş and Eksoy Textile Co. (Bursa, Turkey). The structures of dyes used have been given in Fig. 1.

2.2. Preparation of sample

After cleaning the sample mechanically of the visible impurities, it was ground and sieved to obtain 75-μm size fraction. Then, it was dried at 105 °C, and used in further experiments. Calcinated sepiolite samples have been prepared in the temperature range of 105–700 °C with a Nuve MF-140 furnace. The specific surface areas of the calcinated sepiolite samples at different temperatures were measured by BET N_2 adsorption (Micromeritics Flow Sorb II-2300 equipment). They have been shown in Fig. 2. The cation exchange capacity (CEC) of the calcinated and natural sepiolite samples was determined by the ammonium acetate method and density by the picnometer method [9]. The results are summarised in Table 1. Furthermore, the chemical analysis of sepiolite has shown a composition given in Table 2. All chemicals were obtained from Merck.

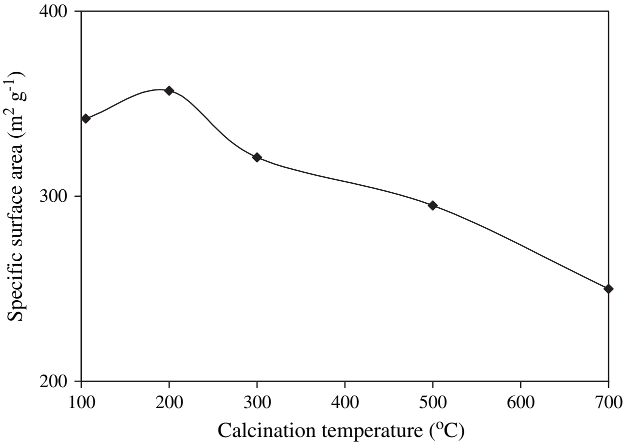

Fig. 2. The changing of specific surface areas against calcination temperatures.

Table 1
Some physicochemical properties of sepiolite

Temperature (°C)	Cation exchange capacity (meg 100 g^{-1})	Density (g mL^{-1})	Specific surface area (m^2 g^{-1})
105	25.0	2.5	342
200	24.5	2.7	357
300	26.0	2.6	321
500	27.0	2.5	295
700	25.0	2.3	250

2.3. Method

Adsorption experiments were carried out by shaking 0.5 g sepiolite samples with 50 mL aqueous solution of reactive blue 221 and acid blue 62 of desired concentrations at various calcination temperatures, pHs (3–11), ionic strengths (0.1–0.5 mol L^{-1}) and temperatures (25–55 °C) for 1 h (the time required for equilibrium to be reached between dyes adsorbed and dyes in solution). The adsorption experiments for calcinated sepiolite samples were only made with reactive blue 221. A thermostated shaker bath was used to keep the temperature constant. The initial concentrations of reactive blue 221 and acid blue 62, C_0, were varied in the range of 5×10^{-5}–80×10^{-5} mol L^{-1}. All adsorption experiments were performed at 25 °C and pH 6.0 of dye solutions except those in which the effects of ionic strength, temperature and pH were investigated. The pH of the solution was adjusted with NaOH or HCl solution by using an Orion 920A pH-meter equipped with a combined pH electrode. The pH-meter was standardized with NBS buffers before every measurement. At the end of the adsorption period, the solution was centrifuged for 15 min at 3000 rpm and then the concentrations of the residual dyes, C_e, were determined with the aid of a Cary |1E| UV–Visible Spectrophotometer (Varian). The measurements were made at the wavelength $\lambda = 602$ nm for reactive blue 221 and $\lambda = 635$ nm for acid blue 62, which correspond to maximum absorbances. Blanks containing no dyes were used for each series of experiments. Each experiment data was an average of two independent adsorption tests. The amounts of dyes adsorbed were calculated from the concentrations in solutions before and after adsorption [10,11].

Table 2
Chemical composition of sepiolite

Component	Weight %
SiO_2	53.47
MgO	23.55
CaO	0.71
Al_2O_3	0.19
Fe_2O_3	0.16
NiO	0.43
LOI	21.49

2.4. Electrokinetic measurements

Zeta potential of sepiolite was measured at 25 ± 1 °C using a Zeta Meter 3.0 equipped with a microprocesser unit. The unit automatically calculates the electrophoretic mobility of the particles and converts it to the zeta potentials in terms of Smoluchowski equation. A 1 g amount of sepiolite was conditioned in 100 mL of 0.0001 M KCl at various final pH values for 24 h. The suspensions were kept still for 5 min to let the larger particles settle. Each data point is an average of approximately 20 measurements. The pH of the suspension was adjusted using dilute HCl and NaOH [12]. All solutions were prepared using bidistilled water.

3. Results and discussion

Adsorption of reactive blue 221 and acid blue 62 onto natural and calcinated sepiolite samples was systematically investigated by parameters such as calcination temperature, pH, ionic strength and temperature. The amount adsorbed of reactive blue 221 and acid blue 62 onto sepiolite at equilibrium was calculated from the mass balance equation as follows:

$$Q_e = (C_0 - C_e)\frac{V}{W} \tag{1}$$

where C_0 and C_e are the initial and equilibrium liquid-phase concentrations of dye solution (mol L^{-1}), respectively, Q_e is equilibrium dye concentration on adsorbent (mol g^{-1}), V is the volume of dye solution (L), and W is the mass of sepiolite sample used (g).

3.1. Effect of activation

The presence of channels of molecular dimensions determines that sepiolite can be considered as an adsorbent with a uniform microporosity constituted by the channels and an appreciable non-microporous surface due to the small size of the fibers. The thermal behaviour of sepiolite has been the subject of several publications in the literature [13,14]. It has been stated that the structural changes of sepiolite with increasing temperature affect the specific surface area and adsorption capacity. Sepiolite has structurally four water molecules coordinated to magnesium. The loss of these water molecules with increasing temperature will affect specific surface area and adsorption capacity of sepiolite. It has been stated that water molecules coordinated to magnesium lose until 500 °C. After losing four water molecules coordinated to magnesium, the structure folds. The folding of the structure is associated with a decrease in the adsorption properties since the channels become narrower and the superficial slots sinter. A further increase in temperature produces the dehydroxylation of the structure and leads to the formation of clinoenstatite. In this way, the changes observed in the adsorption isotherms on both natural sepiolite dried at 105 °C and the sepiolite subjected to a heat treatment can be interpreted on the basis of only the reduction of porosity upon the heat treatment. The results obtained in order to investigate the change taking place in the specific surface area of sepiolite in the calcination temperature range of 105–700 °C have been shown in Fig. 2. As can be seen in Fig. 2, the specific surface area of sepiolite has increased until 200 °C calcination temperature, and then continuously decreased with increasing calcination temperature. Thermal treatment produces a maximum specific surface area of 357 $m^2 g^{-1}$ at 200 °C. This result is generally in line with the reported literature [8,15]. The decrease in the specific surface area of sepiolite may be a result of the removal of most of the micropores and the folding of structure due to heating the sample.

Fig. 3 has shown the adsorption of reactive blue 221 onto calcinated and natural sepiolite samples. As can be seen in this figure, the amount adsorbed of reactive blue 221 on sepiolite has increased until 200 °C calcination temperature, and then decreased the amount adsorbed of reactive blue 221 with increasing activation temperature. After 200 °C calcination temperature, the decrease in the amount adsorbed of reactive blue 221 with increasing activation temperature may be a result of the removal of most of the micropores due to heating the sample [9].

3.2. Effect of pH

pH is an important factor controlling the extent of dye adsorption by hydrous metal oxides. Adsorption depends usually directly on electrokinetic behaviour of oxide as determined by the amount of H^+/OH^- adsorption. Electrokinetic behaviour is determined by the net charge on the particle surface [16]. Fig. 4 shows the zeta potential of sepiolite as a function of pH. The surface is positive at low pH where reaction (2) predominates, and is negative at higher pH when reaction (3) takes over.

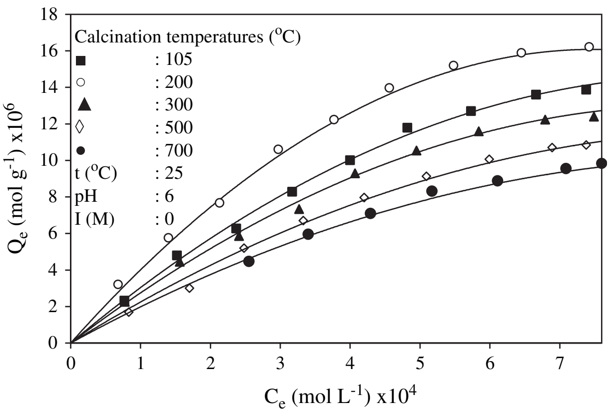

Fig. 3. The adsorption isotherms of reactive blue 221 with natural and calcinated sepiolite samples.

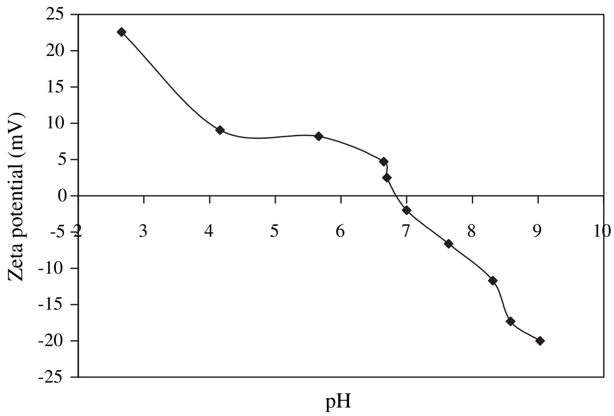

Fig. 4. The electrokinetic behaviour of sepiolite. Conditions: 1.0 g 100 mL^{-1} sepiolite, 0.0001 M KCl, Temperature 25 °C.

$$M{-}OH + H^+ = MOH_2^+ \tag{2}$$

$$M{-}OH + OH^- = MO^- + H_2O \tag{3}$$

Zero point of charge (pH_{zpc}) or isoelectrical point (pH_{iep}) is one of the key points of information of the determination of surface acidity. pH_{zpc} can be distinguished by pH at zero net adsorption of protons and hydroxide ions. Again, pH_{iep} is the point at which zeta potential is zero. pH_{iep} in this case is independent of electrolyte concentration. In that case, the isoelectrical point is the same as the point of zero charge of the surface [17]. As can be seen in Fig. 4, sepiolite has an isoelectrical point around $pH = 6.7$. In order to investigate the effect of pH on the adsorption capacity of sepiolite for reactive blue 221 and acid blue 62, experiments were carried out using various initial solution pH values, changing in the range of 3–11 (Fig. 5a and b, respectively). As seen in these figures, it is found that the amounts adsorbed of reactive blue 221 and acid blue 62 decrease with increasing solution pH. At pH values lower than pH_{iep}, the association of dye anions with more positively charged sepiolite surface because of increasing $S{-}OH_2^+$ groups can more easily take place. In that case, it can be written as follows:

$$MOH_2^+ + Dye^- = MOH_2^+Dye^- \tag{4}$$

With an increase in pH, positive charge at the oxide/solution interface decreases. At pHs above the pH_{iep} of the adsorbent i.e., ~6.7, the adsorbent surface becomes negatively charged and will be associated with

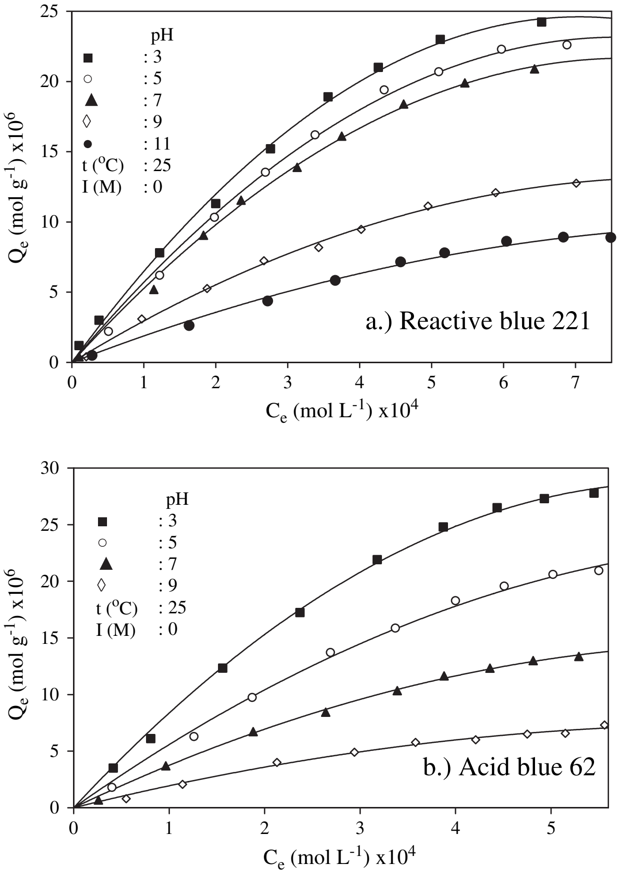

Fig. 5. The adsorption isotherms of reactive blue 221 and acid blue 62 with different pHs on natural sepiolite.

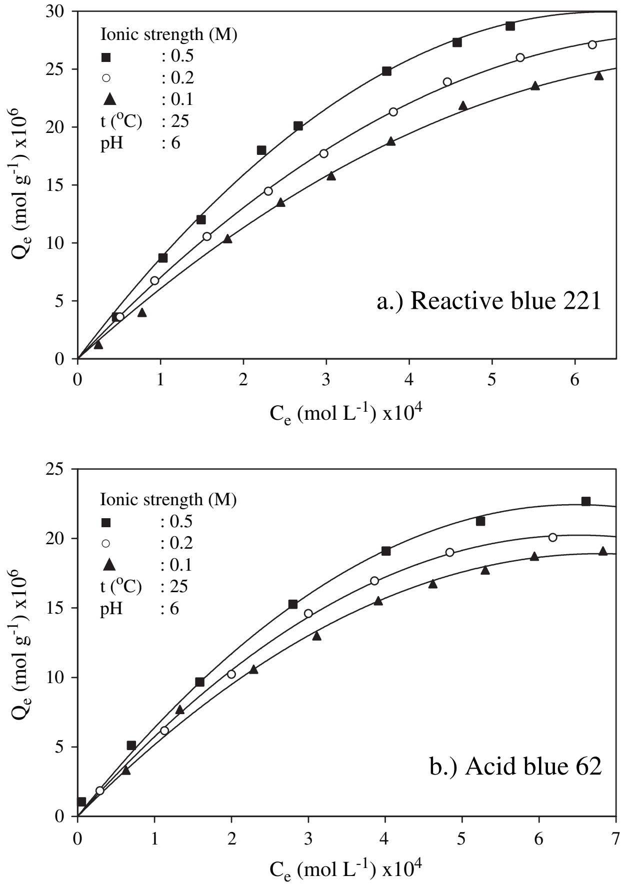

Fig. 6. The adsorption isotherms of reactive blue 221 and acid blue 62 with different ionic strengths on natural sepiolite.

positively charged ions of the solution in the following manner:

$$MO^- + K^+ = MO^-K^+ \qquad (5)$$

Thus, there are no exchangeable anions on the outer surface of the adsorbent at higher pHs and consequently the adsorption decreases. Similar trends were observed in the adsorptions of Congo red on red mud [18] and wollastonite [19] and waste Fe (III)/Cr (III) hydroxide [20].

3.3. Effect of ionic strength

Extensive investigations carried out on adsorption of anionic dyes revealed that the extent of dye uptake was strongly influenced by the concentration and nature of the electrolyte ionic species added to the dyebath [21]. As seen in Fig. 6a and b, the increase in the ionic strength of solution has caused the increase in adsorption of reactive blue 221 and acid blue 62 onto sepiolite surface at pH 6.0. Increase in the extent of dye uptake with increasing electrolyte concentration clearly noticed in these figures is an expected and previously reported behaviour. It has been stated that increase in the ionic strength of the solution increases adsorption at pH values below the isoelectrical point and decreases adsorption at pH values above the isoelectrical point [22]. A cause of increase of adsorption with increasing ionic strength is that increase in the ionic strength increases the positive charge of the surface below the isoelectrical point, resulting in greater attraction of anions, and increases the negative charge of the surface above the isoelectrical point, resulting in greater repulsion of anions. Kara et al. [8] showed that the positive charge of sepiolite surface increased with increasing ionic strength in the pH range of 3.0–6.6. As a result of increase of the positive charge of sepiolite surface with increasing ionic strength, it can be said that the adsorption capacity of sepiolite increases with increasing ionic strength.

3.4. Effect of temperature

The effect of temperature on adsorption was studied by carrying out a series of isotherms at 25, 35, 45 and 55 °C for sepiolite as shown in Fig. 7a and b. Comparing the adsorption values at different temperatures, one sees that the adsorption capacity at higher

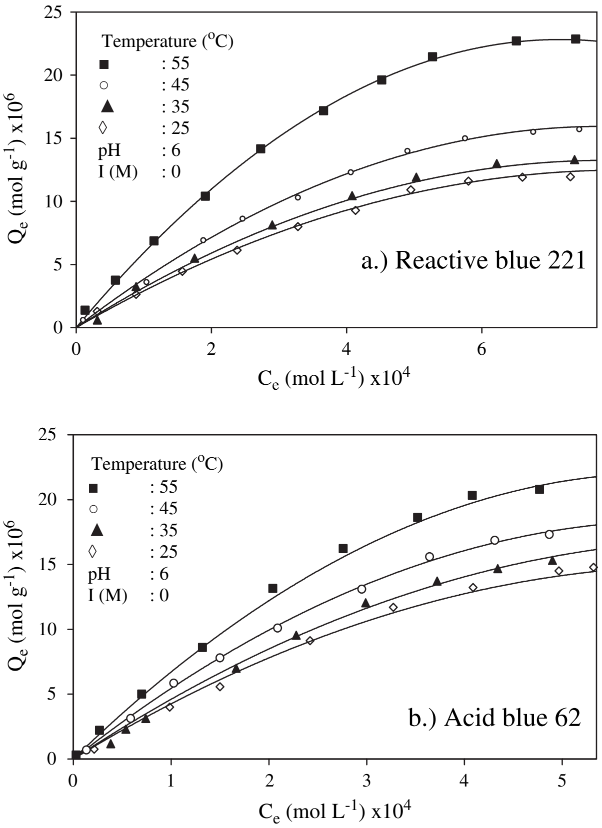

Fig. 7. The adsorption isotherms of reactive blue 221 and acid blue 62 with different temperatures on natural sepiolite.

temperature is higher than that of lower temperature. This may be a result of increase in the mobility of the large dye with increasing temperature [23,24]. An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the sepiolite enabling large dyes to penetrate further as found by Asfour et al. [25]. Increase in raising temperature of the adsorption capacity of sepiolite implies that the enthalpy change (ΔH) has positive values, indicating that the adsorption process of reactive blue 221 and acid blue 62 on sepiolite adsorbent was endothermic, which was opposite to the exothermic adsorption usually encountered, and thereby suggesting the possibility of bonding between the adsorbate and the sepiolite products. Thus, it should be pointed out that sulfonic ($-SO_3^-$) and/or (NH_2) groups of reactive blue 221 and acid blue 62 may interact with the acidic oxide sites on the surface of the sepiolite.

4. Adsorption isotherms

The relationship between the amount of a substance adsorbed at constant temperature and its concentration in the equilibrium solution is called the adsorption isotherm. The adsorption isotherm is important from both a theoretical and a practical point of view. In order to optimize the design of an adsorption system to remove the dye, it is important to establish the most appropriate correlations of the equilibrium data of each system. Two isotherm equations have been tested in the present study: Langmuir and Freundlich models. The applicability of the isotherm equations is compared by judging the correlation coefficients, R^2.

4.1. Langmuir isotherm

Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites. The model assumes uniform energies of adsorption on the surface and no transmigration of adsorbate in the plane of the surface [2]. The Langmuir equation is commonly expressed as

$$Q_e = \frac{Q_m K C_e}{1 + K C_e}$$
$$\frac{C_e}{Q_e} = \frac{1}{Q_m K} + \frac{1}{Q_m} C_e \tag{6}$$

In Eq. (6), C_e and Q_e are as defined in Eq. (1), K is a direct measure for the intensity of the adsorption process, and Q_m is a constant related to the area occupied by a monolayer of adsorbate, reflecting the adsorption capacity. From a plot of C_e/Q_e vs. C_e, Q_m and K can be determined from its slope and intercept.

Table 3a and b presents the correlation coefficient results for Langmuir isotherm, which has not a satisfactorily good correlation between the model predictions and the experimental data, giving the correlation coefficients for Langmuir isotherm in the range of 0.532–0.990.

4.2. Freundlich isotherm

The Freundlich isotherm is an empirical equation and is shown to be satisfactory for low concentrations. The equation is commonly given by

$$Q_e = K_F C_e^{1/n}$$
$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{7}$$

where K_F is a constant for the system, related to the bonding energy. K_F can be defined as the adsorption or distribution coefficient and represents the quantity of dye adsorbed onto adsorbent for an unit equilibrium concentration (i.e., $C_e = 1\ \text{mol L}^{-1}$). The slope $1/n$, ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for $1/n$ below one indicates a normal Langmuir isotherm

Table 3
Isotherm constants for reactive blue 221 and acid blue 62 adsorption onto sepiolite

(a) Reactive blue 221

No	Cal. Temp. (°C)	Temp.(°C)	pH	Ionic strength (mol L^{-1})	Langmuir isotherm			Freundlich isotherm		
					$Q_m \times 10^6$ (mol g^{-1})	$K \times 10^{-5}$ (L mol^{-1})	R^2	$K_F \times 10^3$	n	R^2
1	–	25	3	–	23.8	0.17	0.967	6.2	1.20	0.992
2	–	25	5	–	21.0	0.08	0.853	5.0	1.15	0.985
3	–	25	7	–	18.2	0.08	0.889	4.5	1.09	0.994
4	–	25	9	–	55.9	0.05	0.822	2.4	1.07	0.995
5	–	25	11	–	30.2	0.06	0.861	1.7	1.11	0.993
6	–	25	6	0.1	22.3	0.05	0.830	5.1	1.08	0.996
7	–	25	6	0.2	24.3	0.07	0.816	6.2	1.14	0.991
8	–	25	6	0.5	28.6	0.23	0.977	9.5	1.43	0.990
9	–	25	6	–	12.1	0.14	0.948	3.1	1.31	0.997
10	–	35	6	–	13.5	0.17	0.995	3.7	1.47	0.994
11	–	45	6	–	15.2	0.13	0.821	13.8	1.24	0.997
12	–	55	6	–	22.7	0.41	0.965	7.7	1.71	0.998
13	105	25	6	–	13.87	0.103	0.942	5.89	1.22	0.990
14	200	25	6	–	16.22	0.150	0.945	4.68	1.31	0.996
15	300	25	6	–	12.40	0.125	0.968	4.01	1.27	0.993
16	500	25	6	–	10.85	0.113	0.976	10.00	1.11	0.990
17	700	25	6	–	9.84	0.125	0.975	10.47	1.05	0.989

(b) Acid blue 62

No	Temp.(°C)	pH	Ionic strength (mol L^{-1})	Langmuir isotherm			Freundlich isotherm		
				$Q_m \times 10^6$ (mol g^{-1})	$K \times 10^{-5}$ (L mol^{-1})	R^2	$K_F \times 10^3$	n	R^2
1	25	3	–	20.9	0.09	0.630	7.1	1.23	0.994
2	25	5	–	20.0	0.07	0.532	3.0	0.81	0.996
3	25	7	–	12.7	0.04	0.643	2.2	0.88	0.998
4	25	9	–	12.3	0.08	0.885	1.7	1.20	0.999
5	25	6	0.1	26.9	0.30	0.987	6.5	1.70	0.998
6	25	6	0.2	27.3	0.44	0.967	7.8	1.89	0.996
7	25	6	0.5	32.9	0.34	0.996	8.5	1.80	0.991
8	25	6	–	8.7	0.09	0.870	4.0	1.08	0.995
9	35	6	–	10.3	0.04	0.818	3.7	1.06	0.998
10	45	6	–	12.6	0.07	0.974	4.4	1.08	0.998
11	55	6	–	21.0	0.21	0.971	6.6	1.27	0.996

while $1/n$ above one is indicative of cooperative adsorption. 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 slope of the linear regression [2].

Table 3a and b presents the results of the Freundlich isotherm fit of reactive blue 221 and acid blue 62 to the measured adsorption capacity data for sepiolite at various calcination temperature, pH, ionic strength and temperature. Freundlich isotherm appears to fit the data better than the Langmuir isotherm, as reflected with correlation coefficients (R^2) in the range of 0.985–0.999. As also illustrated in Table 3a and b, the values of n are in the range of 1.06–1.88, which indicates favorable adsorption.

5. Single-stage batch adsorption

The schematic diagram for a single-stage adsorption process is shown in Fig. 8. The solution to be treated contains V solvent (L), and the dye concentration is reduced from C_0 to C_1 (mol L^{-1}) in the adsorption process. The adsorbent is added to the extent of W adsorbate-free sepiolite (g), and the solute dye concentration increases from Q_0 to Q_1 (mol g^{-1}). If fresh

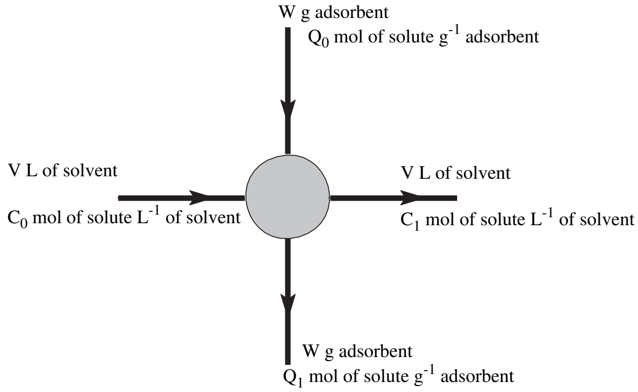

Fig. 8. A single-stage batch adsorber.

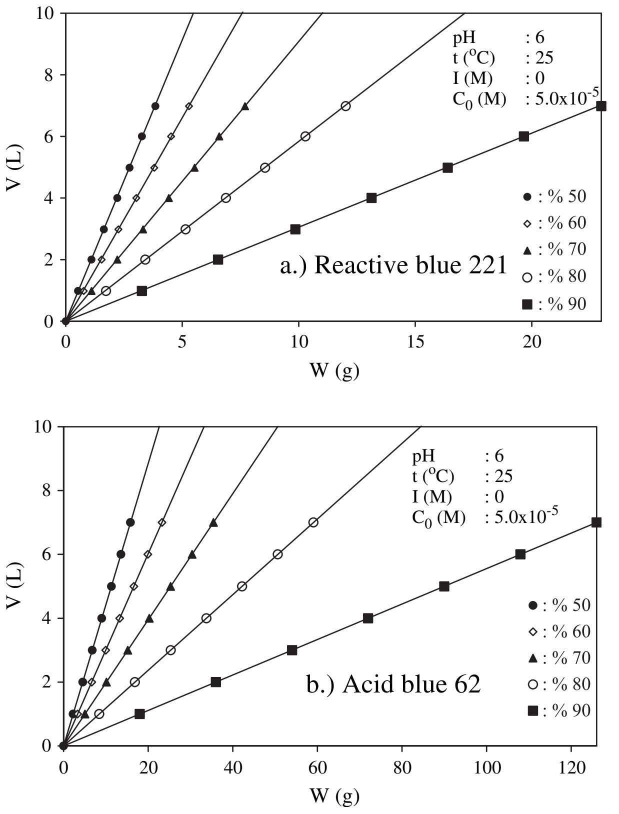

Fig. 9. Volume of effluent (V) treated against adsorbent mass (W) for different percentages of reactive blue 221 and acid blue 62 removals.

adsorbent is used, $Q_0 = 0$. The mass balance equates the dye removed from the liquid to that picked up by the solid.

$$V(C_0 - C_1) = W(Q_1 - Q_0) = WQ_1 \tag{8}$$

The Freundlich data may now be applied to Eq. (8) and substituting for Q_1 from Eq. (6) and rearranging gives

$$\frac{W}{V} = \frac{C_0 - C_1}{Q_e} = \frac{C_0 - C_e}{\left(K_F C_e^{1/n}\right)} \tag{9}$$

Eq. (9) permits analytical calculation of the adsorbent solution ratio for a given change in solution concentration, C_0 to C_e. A series of plots are shown in Fig. 9a and b. Fig. 9a and b shows a series of plots derived from Eq. (9) for the adsorption of reactive blue 221 and acid blue 62 on sepiolite. An initial dye concentration of 5×10^{-5} mol L^{-1} at 25 °C and pH 6 is assumed, and the figures show the amount of effluent which can be treated to reduce the dyes content by 50, 60, 70, 80 and 90% using various masses of adsorbent [1].

6. Conclusions

Sepiolite, a natural clay mineral, can be effectively used as an adsorbent for the removal of anionic dyes such as reactive blue 221 and acid blue 62 from wastewater. The amount adsorbed of reactive blue 221 and acid blue 62 on sepiolite increased with increasing ionic strength under the pH_{iep} and temperature, and decreasing pH. For calcinated sepiolite samples, the adsorbed amount increased until 200 °C calcination temperature, and then decreased with increasing calcination temperature due to sintering effect. The experimental data were correlated reasonably well by the Freundlich adsorption isotherm.

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