The Removal of Victoria Blue from Aqueous Solution by Adsorption on a Low-Cost Material

OZKAN DEMİRBAŞ, MAHIR ALKAN AND MEHMET DOĞAN
Department of Chemistry, Faculty of Science and Literature, Balikesir University, 10100 Balikesir, Turkey
ozkan@balikesir.edu.tr
malkan@balikesir.edu.tr
mdogan@balikesir.edu.tr

Abstract. The use of perlite for the removal of victoria blue from aqueous solution at different concentration, ionic strength, pH and temperature has been investigated. Adsorption process is attained to the equilibrium within 1 h. It is found that the adsorption capacity of perlite samples for the removal of victoria blue increased by increasing pH and temperature, and decreased by expansion and ionic strength. The adsorption isotherms are described by means of the Langmuir and Freundlich isotherms. The adsorption isotherm was measured experimentally at different conditions, and the experimental data were correlated reasonably well by the adsorption isotherm of the Langmuir, and the isotherm parameters (Q_m and K) have been calculated for perlite samples as well. It is concluded that victoria blue is physically adsorbed onto the perlite. The removal efficiency (P) and dimensionless seperation factor (R) have shown that perlite can be used for removal of victoria blue from aqueous solutions, but unexpanded perlite is more effective.

Keywords: adsorption, adsorption isotherms, victoria blue, perlite, dye

1. Introduction

Some specific effluents from industrial production processes may be difficult to purify by traditional wastewater treatment technology, as a result of the complexity of some of their components. These specific components, which are not easily degradable, are commonly toxic, so their discharge can cause serious problems to the environment, and achieving legal purification levels is often very difficult. Wastewater from textile industries creates a great problem of pollution due to the dyes contained therein. Often, dyes are recalcitrant organic molecules that cause strong colour in the wastewater. They contribute to organic load and toxicity of the wastewater. Moreover, textile industry effluents can pose serious problems either wastewater treatment plants, which are often incapable of obtaining satisfactory color elimination with current conventional biological treatment processes, so the combination of different processes is necessary. Color is the first contaminant to be recognized, and environmental regulation in most European countries has made it mandatory to decolorize the dye wastewater prior to discharge (Aretxaga et al., 2001).

Color elimination in wastewater is, today, the principal problem concerning the textile industry. The adsorption characteristics of dyes on various adsorbents have previously been extensively investigated. Pelekani and Snoeyink (2000) investigated the competitive adsorption between atrazine and methylene blue on activated carbon. Wang et al. (1998) studied the adsorption characteristics of dye onto sludge particulates, and found that dye adsorption is a fast process and can reach equilibrium in 30 min. Moreover, their results have shown that pH is the most important factor determining the dye adsorption. The mechanism of adsorption of dyes and phenols from water using activated carbons prepared from plum kernels has been

investigated by Juang et al. (2000). Khattri and Singh (2000) used a bioadsorbent to removal the synthetic dye wastewater, and showed that the equilibrium data followed the Langmuir model of adsorption. The cost of the adsorbent minerals indicate 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 healt has led to research into processes using alternative adsorbents and also some biological treatment of textile wastewater. Suitable candidate would be perlite.

Perlite, a glassy volcanic rock, expands to about 20 times its original volume upon heating within its softening temperature range of 760° to 1090°C (Bolen, 1991, 1993; Alkan and Doğan, in press). As most perlites have a high silica content, usually >70% and are adsorptive, they are chemically inert in many environments and, hence, are excellent filter aids and fillers in various processes and materials (Chesterman, 1975). Along the Aegean Coast, Turkey possesses about 70% (70×10^9 tons) of the world's known perlite reserves (Holroyd, 1995). The main consumption of perlite is in construction related fields, so the production of new construction materials and investigation of properties of those materials have been the subject of researches (Erdem, 1997; Özdeniz, 1996). Özdeniz (1996) studied the hygrothermal performance of a new briquette design. Akin-Öktem and Tinçer (1993, 1994a, 1994b, 1995) prepared and characterized the perlite-filled high density polyethylenes in a series of articles. Only a limited number of studies on the use of perlite as an adsorbent has been found in literature. Antonacci et al. (1976) studied the adsorption of some organic solutes which were 1,2,3,4-tetrahydronapthalene, o-dichlorobenzene, methylnonylketone, and borneol on modified perlite and found that it was quite effective in the extraction of organic solutes from water. In another work of Conti et al. (1978), modified perlite-active charcoal mixture was used for the adsorption organic solutes. They used a modified perlite-active charcoal mixture as the adsorbent for removing organic compounds from drinking water and wastewater and found that a 50% modified perlite-active charcoal mixture is a suitable adsorbent for organic contaminates for analytical work as well as for water purification.

In our previous works, we investigated the electrokinetic properties (Doğan et al., 1997) and surface titrations of perlite suspensions (Alkan and Doğan, 1998), and also the adsorption of copper (II) from aqueous solutions onto perlite samples (Alkan and Doğan, 2001). In the present study, removal of victoria blue from aqueous solutions by adsorption has been studied. The effects of solution pH, ionic strength, and temperature on victoria blue adsorption have been evaluated, and parameters for Langmuir adsorption isotherm have been reported. The results obtained have been applied to a batch design for the removal of victoria blue from aqueous media by using perlite samples.

2.   Materials and Methods

2.1.   Material

The unexpanded and expanded perlite samples were obtained from Cumaovasi Perlite Processing Plants of Etibank (İzmir, Turkey). The chemical composition of the perlite found in Turkey is given in literature (Uluatam, 1991). The unexpanded and expanded perlite samples were treated before using in the experiments as follows (Doğan et al., 1997): the suspension containing 10 g/dm^3 perlite was mechanically stirred for 24 h, after waiting for about two minutes the supernatant suspension was filtered through a white-band filter paper ($\Phi = 12.5$ cm). The solid sample was dried at 110°C for 24 h, then sieved by 100-mesh sieve. The particles under 100-mesh are used in further experiments.

The cation exchange capacity (CEC) of the various perlite samples was determined by the ammonium acetate method, density by the piknometer method. The specific surface area of the samples of expanded (EP) and unexpanded (UP) perlite were measured by BET N$_2$ adsorption. The results are summarised in Table 1 (Doğan et al., 1997). All chemicals were obtained from Merck.

2.2.   Method

Adsorption experiments were carried out by shaking 0.5 g perlite samples with 50 cm^3 aqueous solution of victoria blue of desired concentrations at various pH, ionic strength and temperatures for 1 h. Prior to adsorption experiments the solution was kept under N$_2$ for 10 min. A preliminary experiment revealed that about 1 h is required for victoria blue to reach the equilibrium concentration. A thermostated shaker bath was used to keep the temperature constant. For obtaining an

Table 1. Some physicochemical properties of perlite samples used in the study.

| Sample	Nomenclature	CEC (meg/100 g)	Density (g/cm^3)	Specific surface area (m^2/g)	Zeta potential (mV)
Expanded, purified in water	EP	33.30	2.24	2.30	−46.8
Unexpanded, purified in water	UP	25.97	2.30	1.22	−23.5

adsorption isotherm, the concentration of victoria blue used was varied in the range of 1×10^{-5}–6×10^{-4} M for unexpanded and expanded perlite samples. All adsorption experiments were performed at 30°C and pH 6 except those in which the effect of pH of victoria blue solution was investigated. The pH of the solution was adjusted with NaOH or HNO$_3$ solution by using a Orion 920A pH-meter equipped with a combined pH electrode. 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 concentration of the residual victoria blue, C_e, was determined with the aid of a Cary |1E| UV-Visible Spectrophotometer (Varian). The measurements were made at the wavelength $\lambda = 616$ nm, which corresponds to maximum absorbance. Blanks containing no victoria blue were used for each series of experiments. After 1 h, the victoria blue uptake onto perlite was calculated from the difference between the victoria blue concentration before and after adsorption onto perlite. Each experimental point was an average of three independent adsorption tests (Doğan et al., 2000).

3. Results and Discussion

3.1. Effect of Heat Treatment

Perlite can be considered as a mixed oxide consisting mainly of SiO$_2$ and Al$_2$O$_3$ such as kaolinite, so the structures of silica and alumina may give a picture of perlite. The surface hydroxyl groups of the adsorbent have the main effect on the adsorption of victoria blue onto the perlite, so it would be useful to review the surface hydroxyl groups. The silicon atoms at the surface tend to maintain their tetrahedral coordination with oxygen. They complete their coordination at room temperature by attachment to monovalent hydroxyl groups, forming silanol groups. Theoretically, it is possible to use a pattern in which one silicon atom bears two or three hydroxyl groups, yielding silanediol and silanetriol groups, respectively. It is stated as improbable

that silanetriol groups exist at the silica surface. The types of silanol groups are shown below (Scott, 1993; Karakaş, 1996; Doğan, 1997):

Hydroxyl or silanol groups   Silanediol groups   Silanetriol groups
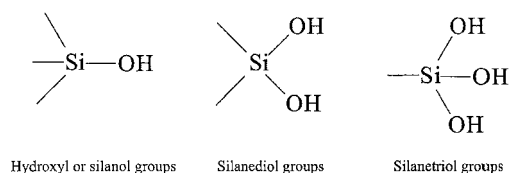
The hydrous oxide surface groups in alumina are given as following (Hohl and Stumm, 1976):
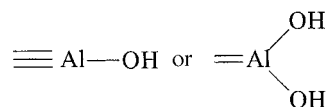
Adsorption of dyes by hydrous metal oxides is frequently found to be extremely rapid, most of the exchange occurring within a matter of minutes. This rapid adsorption reflects the fact that the adsorption is a surface phenomenon and that the surfaces are readily accessible to the dyes in solution. In microporous oxide systems, especially those obtained by heating, equilibrium is achieved somewhat more slowly. The rate of exchange is generally controlled by the rate of diffusion within the particle and this is related to the size, shape and spatial distribution of the pores. The size distribution of microporous in hydrous oxides is frequently found to be very sensitive to heat treatment. The porosity and specific surface are generally found to reach a maximum at some particular temperatures, and then the specific surface area decreases with increasing temperature as a result of sintering (Alkan and Doğan, 2001). The adsorption isotherms of victoria blue onto unexpanded and expanded perlite samples are shown in Fig. 1. The adsorbed amount of victoria blue for unexpanded perlite is greater than that for expanded perlite. The decrease in the amount of adsorption by expansion may be a result of several events occurring during the
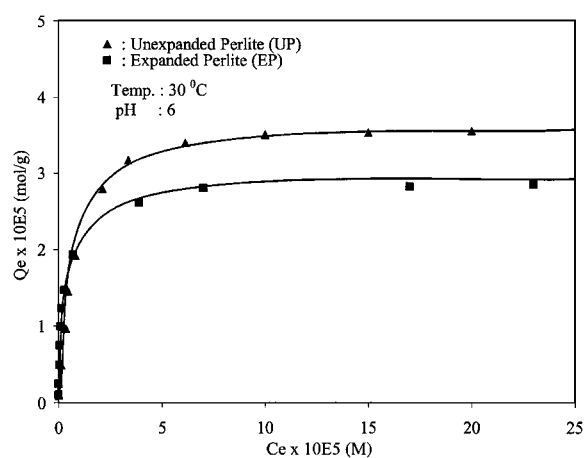
Figure 1. The effect of thermal treatment on the adsorption of victoria blue on perlite.

calcination: (i) the decrease in the amount of hydroxyl groups, (ii) the removal of most of the micropores due to heating the sample (Doğan, 1997). Infrared spectra of the unexpanded and expanded perlite samples show that the amount of hydroxyl groups is decreased by the thermal treatment in the production of expanded perlite from unexpanded perlite (Karakaş, 1996; Doğan, 1997). The decrease in the amount of hydroxyl groups of the adsorbent, which are mainly effective sites for adsorption, during the expansion of perlite is thought to cause a decrease in adsorption capacity, although expanded perlite has greater values of cation exchange capacity (CEC), zeta potential (ZP) and specific surface area than unexpanded perlite.

3.2. Effect of pH of Solution

The tendency for dyes to be adsorbed on solid surfaces has also been put to use for scavenging impurities from solution. The hydrous metal oxides, particularly those of iron, aluminium and manganese, have been used most frequently for these purposes, and the efficiency of removal of the dyes from solution has invariably been found to be strongly pH dependent. To study the influence of pH on the adsorption capacity of perlite samples for victoria blue, experiments were performed using various initial solution pH values, changing from 3 to 6 (Fig. 2). The curves in this figure clearly show that the adsorption capacity of perlite samples increase with increased pH. It has been shown by Doğan et al. (1997) that the perlite samples have no point of zero charged and exhibits negative zeta potential value at the
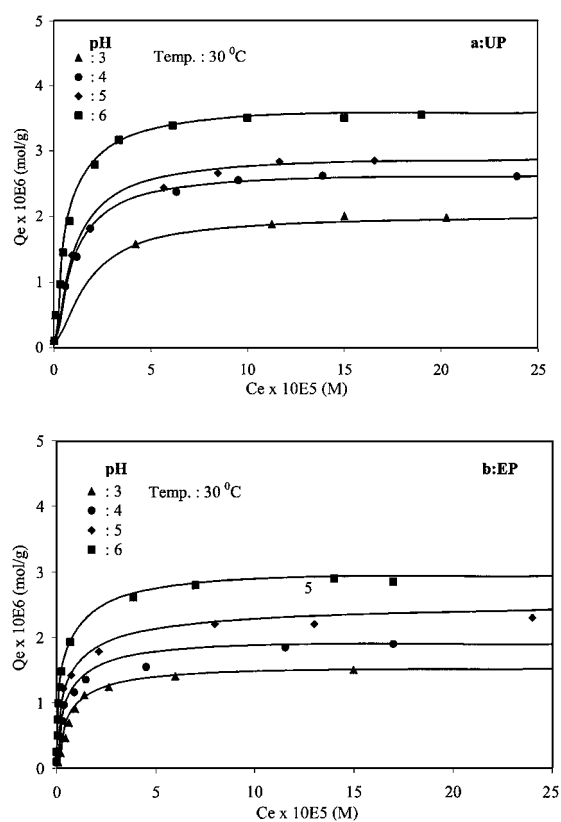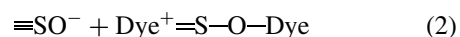

Figure 2. The effect of pH of the solution on the adsorption of victoria blue on perlite: (a) Unexpanded perlite and (b) expanded perlite.

pH range 3–11. As the pH of the dye solution becomes higher (Eq. (1)), the association of dye cations with negatively charged perlite surface can more easily take place as follows (Eq. (2)):

$$\equiv S-OH + OH^- = SO^- + H_2O \qquad (1)$$

$$\equiv SO^- + Dye^+ = S-O-Dye \qquad (2)$$

3.3. Effect of Temperature

A study of the temperature dependence of adsorption reactions gives valuable information about the enthalpy change during adsorption. The effect of temperature on the adsorption isotherm was studied by carrying out a series of isotherms at 30, 40, 50 and 60°C for both of the perlite samples (unexpanded perlite and expanded perlite) and shown in Fig. 3. Results indicate that the adsorption capacity of unexpanded and expanded perlite for adsorption of victoria blue
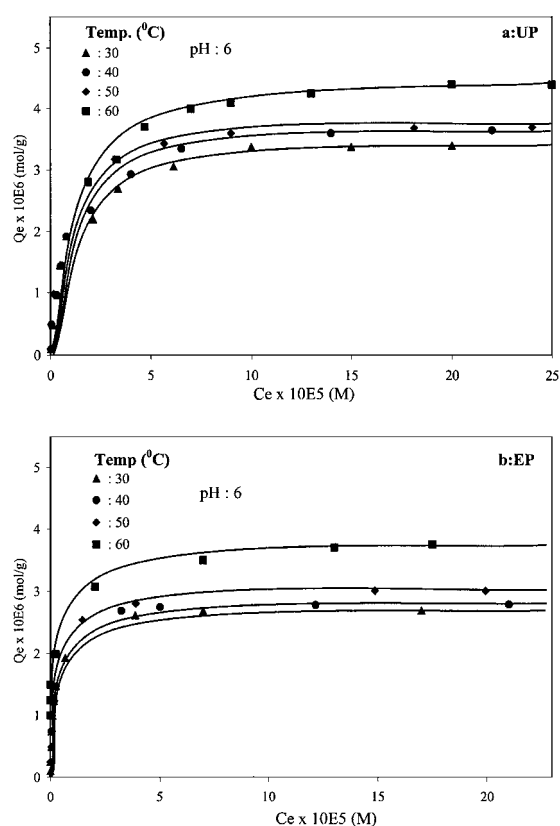
Figure 3. The effect of temperature on the adsorption of victoria blue on perlite: (a) Unexpanded perlite and (b) expanded perlite.

Figure 4. The effect of ionic strength on the adsorption of victoria blue on perlite: (a) Unexpanded perlite and (b) expanded perlite.

increases with increasing temperature which is typical for the adsorption of most organics from their solutions. The effect of temperature is fairly common and increasing the temperature must increase the mobility of the large dye cation. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the perlite enabling large dyes to penetrate further. The R values at different temperatures were also determined (Table 3), and were less than unity. This indicates that the adsorption process becomes more favourable with increasing temperature (Asfour et al., 1985).

3.4. Effect of Ionic Strength

Ionic strength affects the activity coefficients for OH^-, H_3O^+ and specifically adsorbable dye ions. As seen in Fig. 4, the increasing the ionic strength of solution causes the decrease in adsorption of victoria blue onto perlite surface. This indicates that the negative
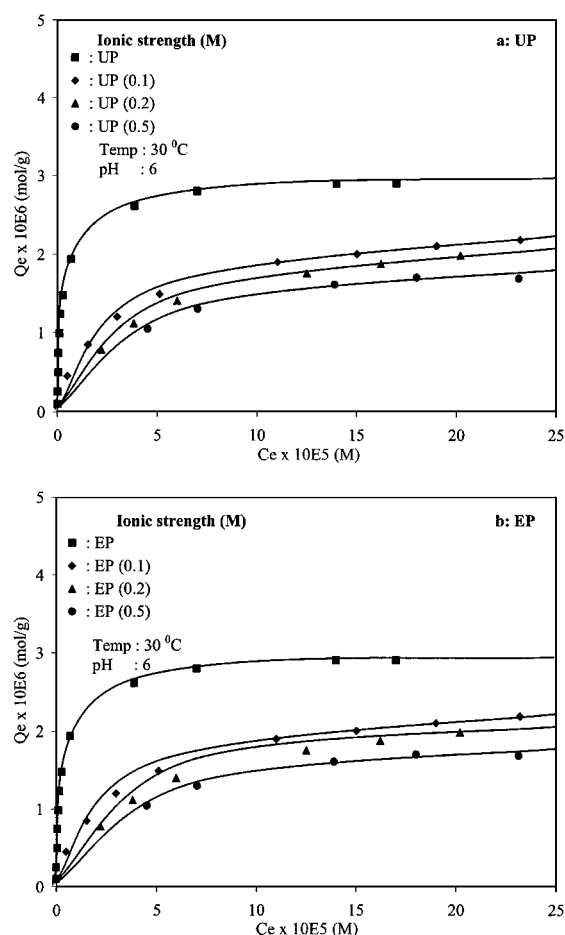
charge of the surface of perlite, which has no pH_{pzc} in the range of pH 3–11 (Doğan et al., 1997; Alkan and Doğan, 1998, 2001), decreases with increasing ionic strength, resulting in reducing the adsorption capacity.

3.5. Isotherm Analysis

The purpose of the adsorption isotherms is to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface (Eastoe and Dalton, 2000). The analysis of the isotherm data is important to develop an equation which accurately represents the results and which could be used for design purposes (McKay et al., 1985). Several isotherm equations are available. Two of them have been selected in this study: Langmuir and Freundlich isotherms.

The linear form of the Langmuir equation can be written in the following form:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K} + \frac{C_e}{Q_m} \qquad (3)$$

where Q_e is equilibrium dye concentration on adsorbent (mol g^{-1}), Q_m is monolayer capacity of the adsorbent (mol g^{-1}), K is adsorption constant (dm^3 mol^{-1}) and C_e is equilibrium dye concentration in solution (mol dm^{-3}). According to the Eq. (3), a plot of C_e/Q_e versus C_e should be a straight line with a slope $1/Q_m$ and intercept $1/Q_m K$ when adsorption follows the Langmuir equation (Alkan and Doğan, 2001).

Freundlich equation in logarithmic form can be written as following:

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e \qquad (4)$$

If Eq. (4) applies, a plot of $\log Q_e$ against $\log C_e$ will give a straight line, of slope $1/n$ and intercept $\log K_F$ (Alkan and Doğan, 2001).

Adsorption isotherms were obtained in terms of Eqs. (3) and (4) by using experimental adsorption results in these equations. Values for Q_m, K, n and K_F are summarised in Tables 2–4. The isotherm data were calculated from the least square method and the related correlation coefficients (r values) are given in the same tables. As seen from the Tables 2–4, the Langmuir equation represents the adsorption process very well; the r values were almost all higher than 0.99, indicating a very good mathematical fit. The fact that the Langmuir isotherm fits the experimental data very well may be due to homogenous distribution of active sites on the perlite surface; since the Langmuir equation assumes that the surface is homogenous (Alkan and Doğan, 2001).

The removal efficiencies, P, defined as (Alkan and Doğan, 2001):

$$P = \frac{C_0 - C_e}{C_0} \times 100 \qquad (5)$$

are given in Tables 2–4. As can be seen from Table 3, the removal efficiency ranged from 98.4–69.5% at 30°C, 99.8–76.4% up to at 60°C for unexpanded perlite and from 99.6–80.0% at 30°C up to 99.9–68.2% at 60°C for expanded perlite.

The shape of the isotherm may also be considered with a view to predicting if an adsorption system is "favourable" or "unfavourable". The essential characteristics of a Langmuir isotherm can be expressed in terms of a dimensionless separation factor or equilibrium parameter R (Alkan and Doğan, 2001), which is defined by

$$R = \frac{1}{1 + K C_e} \qquad (6)$$

According to the value of R the isotherm shape may be interpreted as follows:

Value of R	Type of adsorption
$R > 1.0$	Unfavourable
$R = 1.0$	Linear
$0 < R < 1.0$	Favourable
$R = 0$	Irreversible

The results given in Tables 2–4 show that the adsorption of victoria blue on the perlite is favourable.

Table 2. Isotherm constants for different solution pH and the values of the removal efficiency and separation factor.

Sample	pH	Temp. (°C)	Langmuir isotherm			Freundlich isotherm			%P	R
			$Q_m \times 10^6$ (mol g^{-1})	$K \times 10^{-4}$ (dm^3 mol^{-1})	r	n	$K_F \times 10^5$	r		
UP	3	30	2.052	1.873	0.998	0.264	9.068	0.977	99.98–40.86	0.999–0.017
UP	4	30	2.714	1.264	0.999	0.471	10.629	0.922	98.41–52.20	0.980–0.032
UP	5	30	3.004	1.031	0.997	0.469	8.756	0.965	99.12–63.19	0.991–0.055
UP	6	30	3.683	1.546	0.999	0.563	5.240	0.907	98.39–69.50	0.815–0.043
EP	3	30	1.606	1.056	0.998	0.633	21.050	0.833	91.86–50.04	0.920–0.059
EP	4	30	1.905	1.812	0.997	0.317	0.844	0.886	97.03–61.57	0.788–0.045
EP	5	30	2.268	2.755	0.998	0.192	0.141	0.974	98.34–62.85	0.686–0.027
EP	6	30	2.836	4.371	0.999	0.502	1.590	0.899	99.58–80.00	0.978–0.026

Table 3. Isotherm constants for different temperatures and the values of the removal efficiency and separation factor.

			Langmuir isotherm			Freundlich isotherm				
Sample	pH	Temp. (°C)	$Q_m \times 10^6$ (mol g^{-1})	$K \times 10^{-4}$ (dm^3 mol^{-1})	r	n	$K_F \times 10^5$	r	%P	R
UP	6	30	3.683	1.546	0.999	0.563	5.240	0.907	98.39–69.50	0.815–0.043
UP	6	40	3.762	1.407	0.999	0.551	4.487	0.915	98.58–72.04	0.980–0.048
UP	6	50	3.816	1.609	0.999	0.506	2.982	0.930	99.25–67.03	0.985–0.028
UP	6	60	4.248	1.961	0.992	0.340	0.781	0.919	99.75–76.36	0.883–0.098
EP	6	30	2.836	4.371	0.999	0.502	1.590	0.899	99.58–80.00	0.978–0.026
EP	6	40	2.812	4.721	0.999	0.228	0.743	0.942	99.40–69.65	0.827–0.017
EP	6	50	3.042	4.987	0.999	0.333	0.213	0.885	99.95–60.14	0.996–0.014
EP	6	60	3.762	6.976	0.999	0.143	0.010	0.941	99.89–68.18	0.984–0.018

Table 4. Isotherm constants for different solution ionic strength and the values of the removal efficiency and separation factor.

				Langmuir isotherm			Freundlich isotherm				
Sample	pH	Temp. (°C)	Ionic strength (M)	$Q_m \times 10^6$ (mol g^{-1})	$K \times 10^{-4}$ (dm^3 mol^{-1})	r	n	$K_F \times 10^5$	r	%P	R
UP	6	30	–	3.683	1.546	0.999	0.563	5.240	0.907	98.39–69.50	0.815–0.043
UP	6	30	0.1	3.238	0.413	0.999	0.501	13.600	0.944	91.95–62.22	0.864–0.124
UP	6	30	0.2	2.578	0.315	0.999	0.434	18.272	0.946	83.99–55.30	0.669–0.136
UP	6	30	0.5	2.252	0.245	0.998	0.320	27.220	0.964	73.43–42.49	0.505–0.136
EP	6	30	–	2.836	4.371	0.999	0.502	1.590	0.899	99.58–80.00	0.978–0.026
EP	6	30	0.1	2.410	0.356	0.998	0.414	26.070	0.967	90.01–48.44	0.849–0.108
EP	6	30	0.2	2.364	0.234	0.999	0.395	33.404	0.956	78.02–44.40	0.660–0.145
EP	6	30	0.5	1.952	0.292	0.997	0.294	34.106	0.921	69.91–38.07	0.431–0.109

From the adsorption data at various temperatures for victoria blue, the enthalpy of adsorption, ΔH_{ads}, as a function of coverage fraction ($\theta = Q_e/Q_m$) can be estimated from van't Hoff isochore (Alkan and Doğan, 2001):

$$\left[\frac{\partial \ln K}{\partial T} \right]_\theta = \frac{\Delta H_{ads}}{R_g T^2} \tag{7}$$

The subscript θ means that the equilibrium constant at each temperature is measured at constant coverage. Under these conditions from Langmuir equation at $\theta = 0.5$, $K = 1/C_e$ and so

$$\left[\frac{\ln C_e}{(1/T)} \right]_{\theta = 0.5} = \frac{\Delta H}{R_g} \tag{8}$$

where R_g is the gas constant.

This value of ΔH_{ads} is called the isosteric heat of adsorption referring to the fact that it applies to a certain value of the coverage. The Langmuir model implies that ΔH_{ads} should be constant but it is more likely to be a function of coverage ($\theta = Q_e/Q_m$) (Alkan and Doğan, 2001).

The values of ΔH_{ads} were calculated as 12.1 kJ/mol for expanded perlite and 14.6 kJ/mol for unexpanded perlite from the data given in Fig. 5 according to Eq. (8). The results show that the interactions between surface and adsorbate molecules are a physical interaction. Since adsorption is an endothermic process, it would be expected that an increase in solution temperature woult result in an increase in adsorption capacity (Alkan and Doğan, 2001).

3.6. Designing Batch Adsorption from Isotherm Data

Adsorption isotherms can be used to predict the design of single stage batch adsorption systems (Alkan
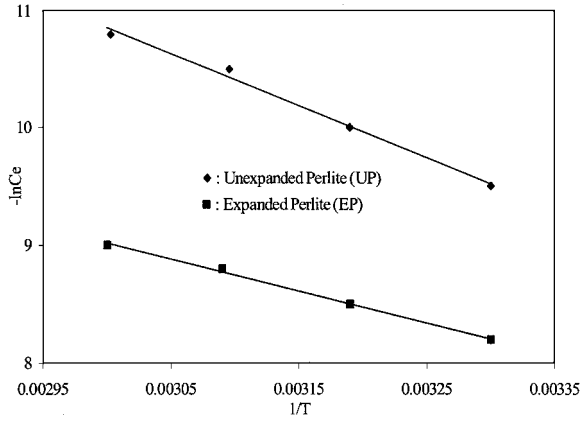
Figure 5. Plot of $-\ln C_e$ versus $1/T$ for adsorption of victoria blue on perlite.
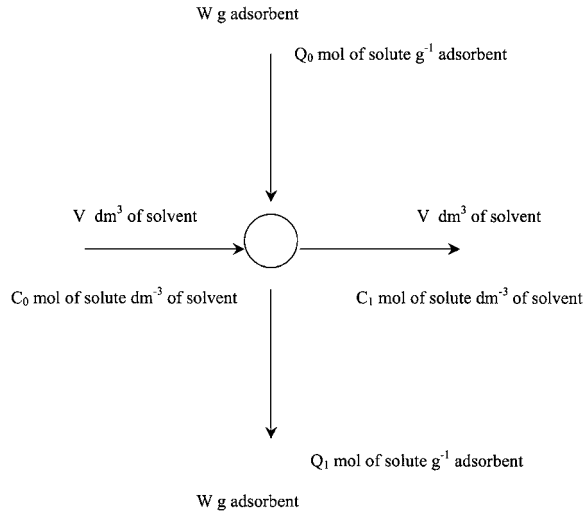

Figure 6. Single stage batch adsorber.

and Doğan, 2001). A schematic diagram is shown in Fig. 6 where the effluent contains V dm^3 of water and an initial victoria blue concentration C_0, which is to be reduced to C_1 in the adsorption process. In the treatment stage W g perlite (dye free) is added and the dye concentration on the solid changes from $Q_0 = 0$ (initially) to Q_1. The mass balance that equates the dye removed from the liquid effluent to that accumulated by the solid is

$$V(C_0 - C_1) = W(Q_1 - Q_0) = WQ_1 \qquad (9)$$

In the case of the adsorption of victoria blue on unexpanded and expanded perlite samples the Langmuir isotherm gives the best fit to experimental data. Consequently equation can be best substituted for Q_1 in the rearranged form of Eq. (9) giving adsorbent/solution ratios for this particular system

$$\frac{W}{V} = \frac{C_0 - C_1}{Q_e} \equiv \frac{C_0 - C_e}{\left(\frac{Q_m K C_e}{1 + K C_e}\right)} \qquad (10)$$

Figures 7(a) and (b) show a series of plots derived from Eq. (10) for the adsorption of victoria blue on unexpanded and expanded perlite. An initial dye concentration of 1.0×10^{-4} mol/dm^3 at 30°C and pH 6 is assumed and figures show the amount of effluent which can be treated to reduce the victoria blue content by 50, 60, 70, 80 and 90% using various masses of adsorbent.
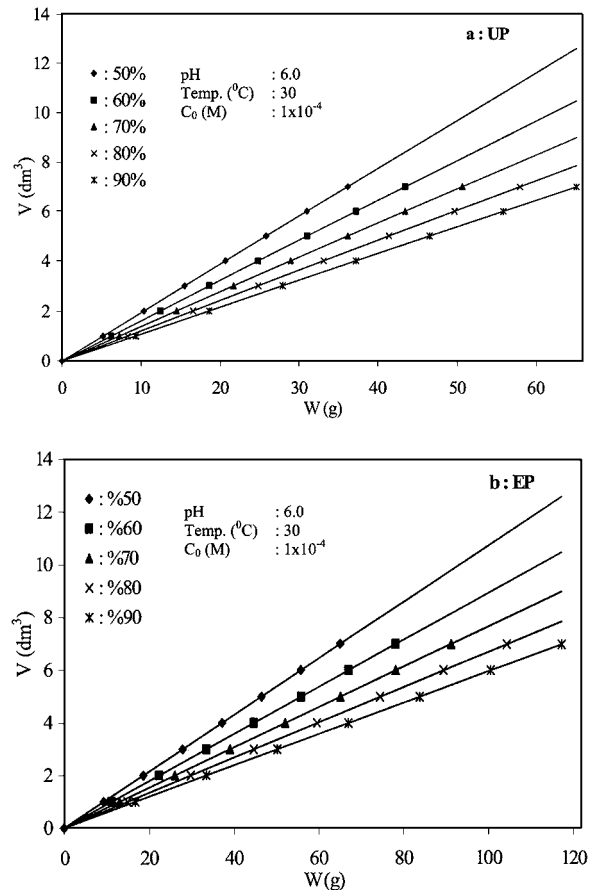

Figure 7. Volume of effluent (V) treated against adsorbents mass (W) for different percentage color removal: (a) Unexpanded perlite and (b) expanded perlite.

4. Conclusions

The experimental data were correlated reasonably well by the Langmuir adsorption isotherm and the isotherm parameters (Q_m and K) have been calculated. The adsorbed amounts of victoria blue increased with increasing pH for both of perlite samples. The adsorbed amount of victoria blue increased with increase in temperature for both of perlite samples. The adsorbed amount of victoria blue slightly decreased with increasing ionic strength for both of perlite samples. The dimensionless separation factor (R) showed that perlite can be used for removal of victoria blue from aqueous solutions, but unexpanded perlite is more effective. Its adsorption capacity is greater than that of expanded perlite. The values of ΔH_{ads} for unexpanded and expanded perlite samples were calculated as 14.6 and 12.1 kJ/mol, respectively. Perlite has a considerable potential as an adsorbent of dyes in a commercial system because of being cheap.

As can also understood from the present work, the studies about the adsorbent properties of perlite have been quite limited, so it was considered to be important to investigate wheather or not perlite could be used for removal of dyes from industrial wastewater. As the adsorbent properties of perlite become better known, it will be find more fields on the environmental problems such as the removing of dyes, heavy metals and other pollutants as an adsorbents due to its natural occurrence and inexpensive cost.

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