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Sorption Studies of Acid Dye by Mixed Sorbents

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Abstract. The sorption of Acid Blue 9 onto the mixture of activated clay and activated carbon has been studied in terms of pseudo-first order and pseudo-second order chemical sorption processes. The batch sorption model, based on the assumption of a pseudo-second order mechanism, has been developed to predict the rate constant of sorption and the equilibrium capacity with the effect of initial dye concentration, mass of mixed sorbent, temperature and initial solution pH. The rates of sorption were found to conform to pseudo-second order kinetics with good correlation. Batch isotherm studies showed that the sorption of Acid Blue 9 by the mixed sorbent from aqueous solution was described by the Langmuir isotherm equation. A comparison of the evaluated equilibrium capacity of sorption has been made by the pseudo-second order rate equation as well as by the Langmuir isotherm and operating line method. In addition, an activation energy of sorption has also been determined based on the pseudo-second order rate constants.

Keywords: sorption, kinetics, acid dye, mixed sorbent

Introduction

In modern industrial society, dyes are used everywhere. Because they are inert and nontoxic when discharged into waste streams, people always overlook their undesirable nature. Some are harmful to aquatic life in rivers where they are discharged. Decolourizing of textile and dye-manufacturing wastewater is currently a major problem for environmental managers. Coloured effluent treatment has not received much attention as it was thought to be of only aesthetic concern. Sorption techniques produce high quality treated effluents and sorption processes have been investigated as a method to remove dyes from wastewater. The most widely used sorbent for industrial applications is activated carbon (Walker and Weatherley, 1997; Khalil and Girgis, 1998; Porter et al., 1999). There are still problems associated with its use; namely, activated carbon is expensive and the higher the quality the greater the cost. Extensive investigations are being carried out to identify suitable relatively cheap sorbents capable of removing significant quantities of acid dyes. Table 1 shows a number of cheap sorbents available for acid dye sorption.

In recent years, clay has been accepted as one of the most appropriate low cost sorbents. For the treatment of colours, such as carpet effluents containing mainly Metomega Chrome Orange GL (Gupta and Shukla, 1996), the popular chrome dye, Omega Chrome Red ME (Gupta et al., 1992) and the acid dye (Khattri and Singh, 1998) as well as the treatment of heavy metals, such as lead(II) (Yadava et al., 1991) and cesium (Sawhney, 1966), the application of sorption techniques using clay has been tested. In addition, the mixed sorbent systems, such as the sorption of lead(II) by

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Table 1. A number of cheap available sorbents for sorption of acid dyes.

Sorbents	References
Peat	Poots et al. (1976)
Wood	Ho and McKay (1998)
Woodmeal	McKay and McConvey (1985)
Wood sawdust	Ibrahim et al. (1997)
Chitin	McKay et al. (1987)
Linked chitosan fiber	Yoshida et al. (1993)
Biomass	Laszlo (1994)
Slag	Ramakrishna and Viraraghavan (1998)
Clay	Khattri and Singh (1998)
Water hyacinth root	Lee et al. (1999)
Pith	Ho and McKay (1999)
White rot fungu	Nagarajan and Annadurai (1999)

adsorption on china clay and wollastonite (Yadava et al., 1991), and the heterogeneous mixture of alumina and clay to remove the colour of dyes (Khattri and Singh, 1998) have also been investigated.

In this paper, experiments have been carried out to expect the sorption capacity and kinetics of Acid Blue 9 from aqueous solution using the mixture of activated clay and activated carbon. The factors studied include the influence of initial dye concentration, mass of mixed sorbent, temperature and initial solution pH on the sorption kinetics of the system. A rate parameter, k, has been defined and used to describe the sorption of acid dye onto mixed sorbent. In addition, a comparison of a pseudo-first order rate parameter, k_1, and a pseudo-second order sorption parameter, k, has also been discussed.

Materials and Methods

Acid Blue 9, AB9, (was provided by Taiwan I-Hwa Co. and Bayer Chemical Co.), solutions were prepared with reverse osmosis treated water to make 500 ml of solutions at pH 3.0, which were maintained at a desired temperature prior to the experiment (Solution A). Activated carbon and activated clay (was purchased from Laporte Industries Singapore Pte., Ltd.) had been placed in an oven for more than 24 hours, that were accurately weighed for a required amount and were placed in the stirring chamber containing 400 ml of reverse osmosis treated water with pH adjusted to 3.0, and temperature was preset to a desired value. The ionic strength of the dye solution at pH 3.0 was adjusted to 0.02 M with sodium sulfate (Solution B). Before mixing, 100 ml of Solution A was drawn for the measurement of the initial dye concentration, C_0, while the remaining 400 ml was immediately decanted into Solution B for the sorption experiment. At intervals of 0.5, 1, 2, 5, 30, 60 and 120 minutes, about 2 ml of the sample was drawn using a 20 ml syringe. It was then filtered through a membrane filter (it was place in a filter with a diameter of 25 mm, provide by Gelman Co.). The filtrate was diluted with reverse osmosis treated water for optical measurement. To study the effects of experimental parameters such as the initial dye concentration, the ratio of activated carbon to activated clay, pH, and temperature on AB9 sorption, we proceeded by changing one parameter at a time while holding the others constant.

Results and Discussion

Sorption Isotherm

The equilibrium isotherm for the sorption of AB9 on mixed sorbent was determined with 6.5 g mixed sorbent, activated clay 6 g and carbon 0.5 g, with 0.8 dm^3 of dye solutions of various concentrations. The systems were agitated for 2.5 hours in a constant temperature using water bath. The sorption process was confirmed to fit with the following Langmuir isotherm:

$$q_e = \frac{C_e}{\frac{1}{K_L} + \frac{a_L}{K_L} C_e} \tag{1}$$

The experimental points are shown in Fig. 1 and compared with a solid Langmuir isotherm line, having a correlation coefficient of 0.996 and equation:

$$q_e = \frac{C_e}{9.75 \times 10^{-2} + 1.62 \times 10^{-2} C_e} \tag{2}$$

Figure 1 also shows the operating line which is generated with a slope of solution volume/mass of mixed sorbent. The final equilibrium solution concentration is predicted from the point of intersection of the operating line on the Langmuir isotherm, for the sorption of AB9. The operation line is the line which predicts the time dependence of a sorption process and integrates this dependence into the equilibrium isotherm. The dye material balance equation for batch dynamic studies is:

$$(C_0 - C_t)V = (q_t - q_0)m \tag{3}$$

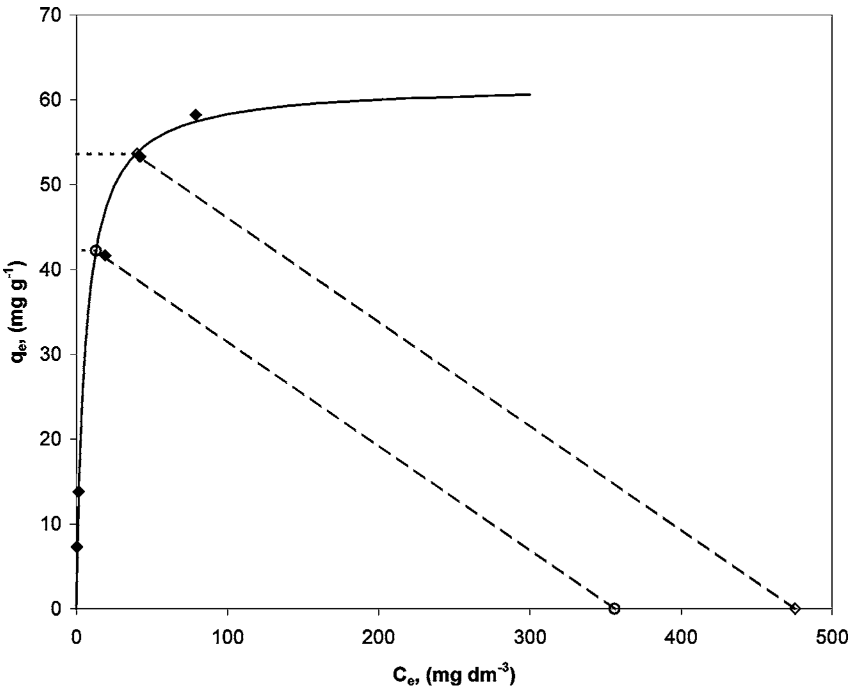

Figure 1. Langmuir equation of AB9 onto mixed sorbent. (◆) Experimental date, (○----○) $C_0 = 356$ mg dm^{-3}, (◇----◇) $C_0 = 475$ mg dm^{-3} and (—) Langmuir isotherm. Conditions: pH 3.0, $T = 303$ K and $S = 500$ rpm.

At equilibrium, this becomes:

$$(C_0 - C_t)V = (q_e - q_0)m \qquad (4)$$

For fresh mixed sorbend $q_0 = 0$. It is can be expressed as follows:

$$q_e = -\frac{V}{m_s}C_e + \frac{V}{m_s}C_0 \qquad (5)$$

where V is solution volume (dm^3) and m_s is mass of mixed sorbent (g).

The equilibrium sorption capacity, q_m, can be obtained from the operating line and the Langmuir equation (Fig. 1). The results are in reasonable agreement with the equilibrium sorption capacity, q_e, obtained from the pseudo-second order model. A comparison is made between the equilibrium sorption capacity evaluated from Langmuir equation and the pseudo-second order model in Table 2, which shows the equilibrium sorption capacities evaluated are reasonable. Again a comparison is made between the equilibrium sorption capacity evaluated from Langmuir equation and the pseudo-second order model in Table 3, which shows the equilibrium sorption capacities evaluated are reasonable.

Sorption Dynamics

Predicting the rate at which sorption takes place for a given system is probably the most important factor

Table 2. Parameters for effect of initial concentration on the AB9 activated clay/carbon system.

C_0, (mg dm^{-3})	r^2	k ($\times 10^{-2}$ g mg^{-1}min^{-1})	q_e, (mg g^{-1})	q_m, (mg g^{-1})	r_1^2	q_1, (mg g^{-1})
59.7	1.000	134	7.28	7.25	0.963	0.253
114	1.000	55.3	13.8	13.8	0.913	0.632
356	1.000	6.68	41.0	42.2	0.877	4.26
475	1.000	2.79	52.9	53.6	0.723	9.35

Table 3. Parameters for effect of ratio of carbon and clay on the AB9 activated clay/carbon system.

m_s, (g)								
Clay	Carbon	r^2	k ($\times 10^{-3}$ g mg^{-1}min^{-1})	q_e (mg g^{-1})	q_m (mg g^{-1})	R_e (%)	r_1^2	q_1 (mg g^{-1})
2	0.5	1.000	11.9	64.7	59.6	58.4	0.949	20.4
3	0.5	0.999	9.8	58.9	58.0	47.5	0.987	21.7
4	0.5	1.000	20.8	52.3	54.1	85.1	0.946	12.7
6	0.5	1.000	68.7	39.9	41.1	93.6	0.877	4.2

for sorber design, with sorbate residence time and the reactor dimensions controlled by the system's kinetics. However, sorption kinetics show a large dependence on the physical and/or chemical characteristics of the sorbent material which also influences the sorption mechanism. In order to investigate the mechanism of sorption, the constants of sorption of dye were determined to use equations of Lagergren (1898), a pseudo-first order mechanism and equations of Ho (1995), a pseudo-second order mechanism, respectively which are as follows:

Pseudo-First Order Model. The sorption kinetics may be described by a pseudo-first order (Lagergren, 1898). The differential equation is the following:

$$\frac{dq_t}{dt} = k_1(q_1 - q_t) \tag{6}$$

Integrating Eq. (6) for the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, gives:

$$\log\left(\frac{q_1}{q_1 - q_t}\right) = \frac{k_1}{2.303}t \tag{7}$$

which is the integrated rate law for a pseudo-first order reaction. Where q_1 is the amount of dye sorbed at equilibrium, (mg g^{-1}); q_t is amount of dye sorbed at time t, (mg g^{-1}) and k_1 is the equilibrium rate constant of pseudo-first sorption, (min^{-1}). Equation (7) can be rearranged to obtain a linear form:

$$\log(q_1 - q_t) = \log(q_1) - \frac{k_1}{2.303}t \tag{8}$$

In order to obtain the rate constants, the straight line plots of log $(q_1 - q_t)$ against t for different experimental conditions have been analysed. The rate constant, k_1 and correlation coefficients, r_1^2, values of the dye under different conditions were calculated from these plots.

Pseudo-Second Order Model. The sorption kinetics may be described by a pseudo-second order (Ho, 1995). The differential equation is the following:

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \tag{9}$$

Integrating Eq. (9) for the boundary condition $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, gives:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt \tag{10}$$

which is the integrated rate law for a pseudo-second reaction. Where q_e is amount of dye sorbed at equilibrium (mg g^{-1}) and k is the equilibrium rate constant of pseudo-second order (g mg^{-1}min^{-1}). Equation (10) can be rearranged to obtain a liner form:

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t \tag{11}$$

The straight line plots of t/q_t against t have been tested to obtain rate parameters. The values of k, q_e and correlation coefficients, r^2, of dye under different conditions were calculated from these plots.

Effect of Initial Dye Concentration. A series of sorption using mixed sorbent, activated clay 6 g and carbon 0.5 g, were carried out to study the influence of initial dye concentration. Figure 2 shows the removal of dye increased rapidly in the beginning and then more slowly until the equilibrium. This was caused by strong attractive forces between the dye molecules and the mixed sorbent, fast diffusion onto the external surface was followed by fast pore diffusion into the intraparticle matrix to attain rapid equilibrium (Asfour et al., 1985). Similar results were reported in the removal of chrome dye by mixed fly ash and coal (Gupta et al., 1990). Linear plots of the t/q_t against t in Fig. 3, show the applicability of the pseudo-second order equation for the system of

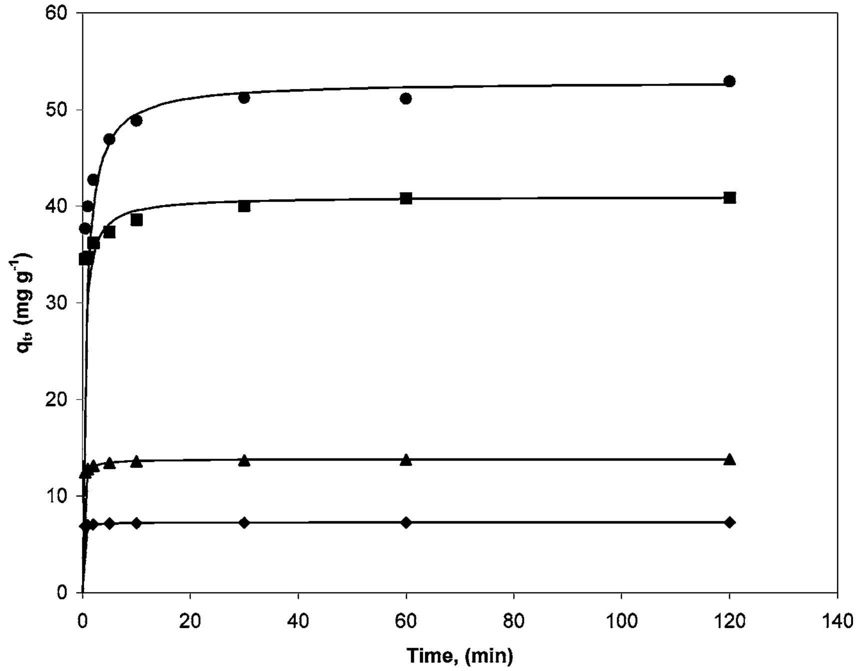

Figure 2. Plot of sorbed amount versus time for AB9 at various initial concentrations. (◆) $C_0 = 59.7$ mg dm^{-3}, (▲) $C_0 = 114$ mg dm^{-3}, (■) $C_0 = 356$ mg dm^{-3}, (●) $C_0 = 475$ mg dm^{-3} and (—) pseudo-second order model. Conditions: pH 3.0, $T = 303$ K, $S = 500$ rpm and $m_s = 6.5$ g dm^{-3}.

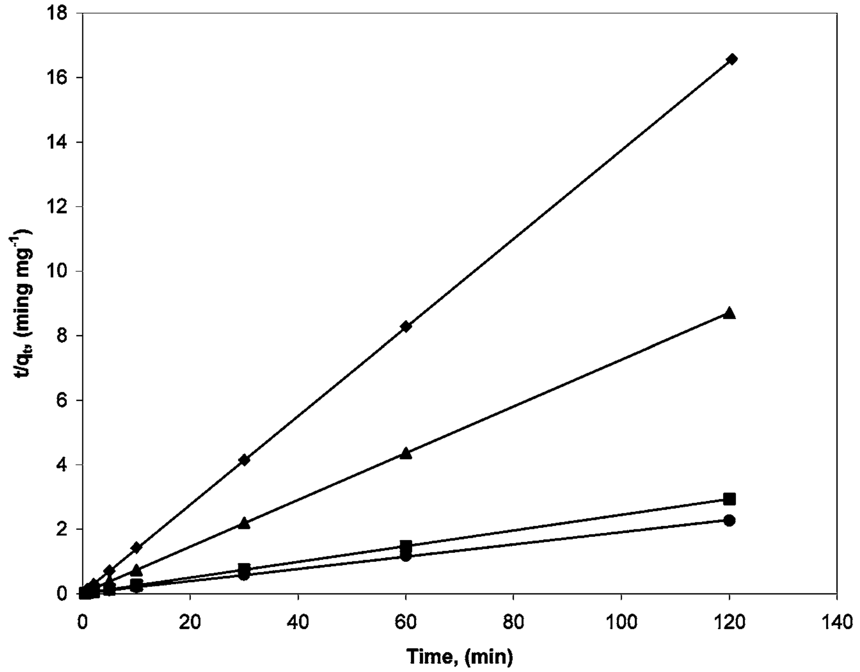

Figure 3. Pseudo-second order sorption kinetics of AB9 onto mixed sorbent at various initial concentration. (◆) $C_0 = 59.7$ mg dm^{-3}, (▲) $C_0 = 114$ mg dm^{-3}, (■) $C_0 = 366$ mg dm^{-3}, (●) $C_0 = 475$ mg dm^{-3} and (—) pseudo-second order model. Conditions: pH 3.0, $T = 303$ K, $S = 500$ rpm and $m_s = 6.5$ g dm^{-3}.

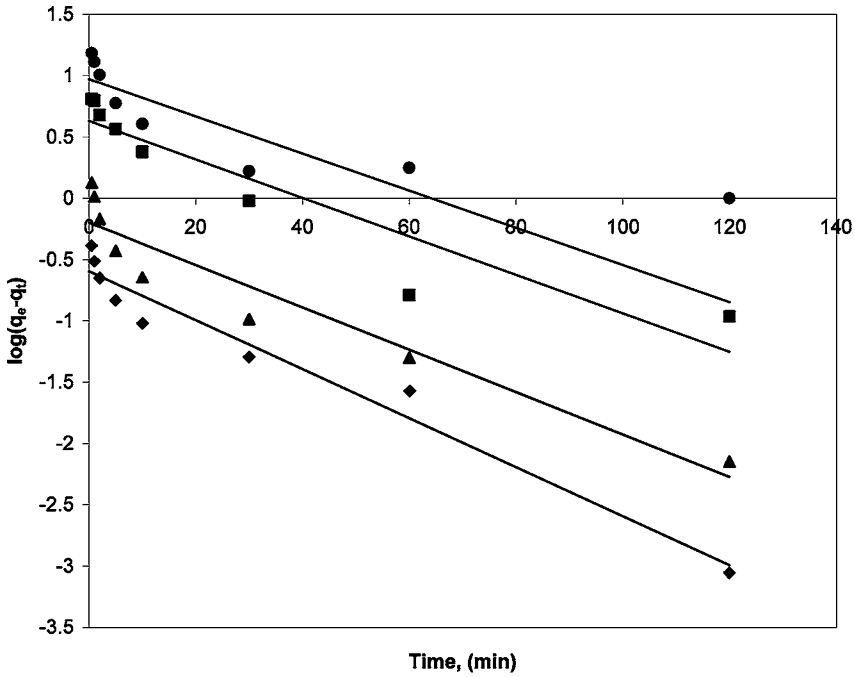

Figure 4. Pseudo-first order sorption kinetics of AB9 onto mixed sorbent at various initial concentrations. (◆) $C_0 = 59.7$ mg dm^{-3}, (▲) $C_0 = 114$ mg dm^{-3}, (■) $C_0 = 366$ mg dm^{-3}, (●) $C_0 = 475$ mg dm^{-3} and (—) pseudo-first model. Conditions: pH 3.0, $T = 303$ K, $S = 500$ rpm and $m_s = 6.5$ g dm^{-3}.

AB9 with the mixed sorbent for initial concentrations ranging from 59.7 to 475 mg dm^{-3}. Figure 4 shows plots of q_t against t for the pseudo-first order equation for the sorption of AB9. The correlation coefficients, r^2, and the pseudo-second order rate parameters, k, are shown and compared with r_1^2 and k_1 values for the pseudo-first order reaction mechanism (Table 2). The data show a good compliance with the pseudo-second order equation and the regression coefficients for the linear plots were higher than 0.999 for all the systems.

The kinetics of AB9 sorption on the mixed sorbent follows the pseudo-second order rate expression. However, the mechanism of dye sorption has usually been described by the pseudo-first order Lagergren equation (Low et al., 1995; Mittal and Gupta, 1996; Walker and Weatherley, 1999). The equilibrium sorption capacity increased from 7.28 to 52.9 mg g^{-1} when the initial concentration of AB9 increased from 59.7 to 475 mg dm^{-3}, indicating that the dye removal is dependent on initial concentration. However, the values of the rate constant were found to decrease from 1.34 to 2.79×10^{-2} g mg^{-1}min^{-1} for an increase in the initial concentration from 59.7 to 475 mg dm^{-3}.

Effect of Mass of Mixed Sorbent. The kinetic data obtained from batch studies have been analysed using pseudo-first order model proposed by Lagergren (1898) and the results shown in Table 3. The correlation coefficients, r^2, the pseudo-second order rate constant, k, and equilibrium sorption capacity, q_e, were calculated and presented in Table 3. It is evident that the kinetic of AB9 sorption on the mixed sorbent follows a pseudo-second order rate expression. The results show that the sorption capacity for AB9 increases from 39.9 to 64.7 mg g^{-1} with the decrease in mass of the mixed sorbent from 6.5 g to 2.5 g. It can be seen that removal increases from 58.4% to 93.6% with the increase of the mixed sorbent doses from 2.5 g to 6.5 g for a given initial dye concentration (Table 3). This leads to the conclusion that, in order to obtain the optimum sorbent dose, higher dye concentrations should be tested in conjunction with high doses. This indicates that with an increased mass of mixed sorbent, more surface area is made available and therefore the total number of sites increases.

Effect of Temperature. The sorption experiments were repeated at various temperatures in the range of

Table 4. Parameters for effect of temperature on the AB9 activated clay/carbon system.

T (K)	r^2	k ($\times 10^{-2}$ g mg^{-1}min^{-1})	q_e (mg g^{-1})
293	1.000	6.68	41.0
303	1.000	5.84	40.6
313	1.000	4.91	40.1

293 to 313 K with the mixed sorbent, activated clay 6 g and carbon 0.5 g. Table 4 shows that the equilibrium sorption capacity of AB9 slightly decreases with increasing temperature that indicates a low temperature favors AB9 removal by sorption onto the mixed sorbent. This may be due to a tendency for the dye molecules to escape from the solid phase to the bulk phase with an increase in temperature of the solution (Panday et al., 1984; Gupta et al., 1998). A similar observation was also reported in the study on the sorption of Basic Blue 3 on silica (Ahmed and Ram, 1992) and the sorption of lead onto china clay and wollastonite (Yadava et al., 1991). The results in Table 4 show the correlation coefficients for the linear plots from the pseudo-second order equation are extremely high.

The values of rate constant, k, were found to slightly decrease from 6.68×10^{-2} to 4.91×10^{-2} g mg^{-1}min^{-1} for an increase in solution temperature from 293 to 313 K for the sorption of AB9. A linear relationship between the pseudo rate constant and temperature was observed with a correlation coefficient of 0.992. The k values were plotted as a function of the reciprocal of the Kelvin temperature. Linear variations were observed as shown in Fig. 5. Therefore, the sorption rate constant may be expressed as a solution function of temperature by the following relationship:

$$k = k_0 \exp\left(\frac{-E}{RT}\right) \tag{12}$$

where k is the rate constant of sorption (g mg^{-1} min^{-1}); k_0 is the temperature independent factor (g mg^{-1}min^{-1}); E is activation energy of sorption (kJ mol^{-1}); R is the gas constant (8.314 J mol^{-1} K^{-1}); T is solution temperature (K).

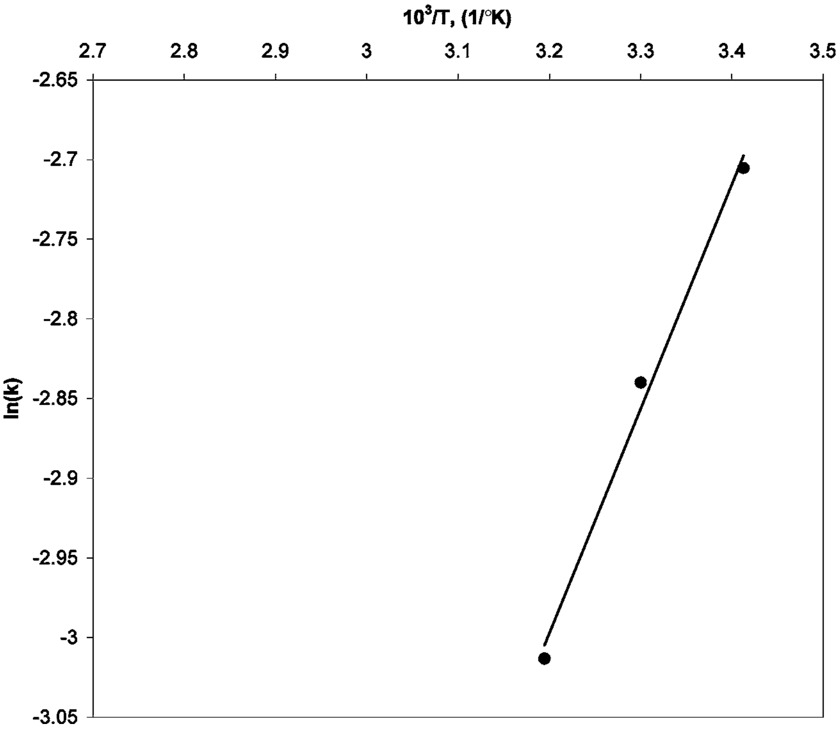

Figure 5. Plot of ln (k) against reciprocal temperature for AB9 sorption onto a mixed sorbent. Conditions: C_0 = 300 mg dm^{-3}, pH 3.0, S = 500 rpm and m_s = 6.5 mg dm^{-3}.

Thus, the relationship between k and T can be represented in an Arrhenius form as:

$$k = 5.50 \times 10^{-4} \exp\left(\frac{11.7}{8.314T}\right) \qquad (13)$$

From this equation, the rate constants of sorption, k_0, is 5.50×10^{-4} g mg^{-1}min^{-1} and the activation energy of sorption, E, is -11.7 kJ mol^{-1}. Since sorption is an exothermic process, it would be expected that an increase in solution temperature would result in a decrease in sorption capacity.

Effect of Initial pH. It is expected that the rate of AB9 sorption onto the mixed sorbent will be varied with the available pH values of solution when ion exchange development and applications is one of the sorption processes. The pH values used in these studies are 2.0, 3.0, 5.0, 7.0 and 9.0 for the sorption of AB9 with the mixed sorbent, activated clay 6 g and carbon 0.5 g. The rate constant, k, and the equilibrium capacity, q_e, of sorption at various pH values have been determined and are listed in Table 5.

The removal of AB9 from aqueous solution by sorption is highly dependent on pH of the solution which affects the surface properties of the sorbent and dye structure. The sorption capacity for AB9 of the mixed sorbent decreased from 41.1 to 29.7 mg g^{-1} when the pH of the initial dye solution was increased from 2.0 to 9.0 (Table 5). The extent of dye removal decreased rapidly as pH was increased from pH 3.0 to 7.0. However, the dye removal remained essentially constant within the pH range of 2.0 to 3.0 and 7.0 to 9.0. The trend was similar to the sorption of chrome dye on china clay (Gupta et al., 1992) and the sorption of lead on kaolinitic clay (Orumwense, 1996). In addition, the sorption capacity of As(III) on china clay increases with an increase in pH within the range of 4.0 to 8.0 (Yadava et al., 1988).

Table 5. Parameters for effect of initial pH on the AB9 activated clay/carbon system.

pH	r^2	k ($\times 10^{-2}$ g mg^{-1}min^{-1})	q_e (mg g^{-1})
2.0	1.000	5.95	41.1
3.0	1.000	6.68	41.0
5.0	1.000	3.97	35.3
7.0	1.000	3.28	30.1
9.0	1.000	3.73	29.7

Conclusions

The kinetics of sorption of AB9 onto mixed sorbent, activated clay and carbon (12:1) is studied on the basis of the pseudo-first and pseudo-second order rate mechanism. The kinetics of AB9 sorption on the mixed sorbent follows the pseudo-second order rate expression. A pseudo-second order model has been developed to predict the rate constant of sorption and the equilibrium capacity. The activation energy of sorption can be evaluated using the pseudo-second order rate constants. The sorption of AB9 onto the mixed sorbent is favoured at higher concentrations of dye solution, low temperatures and solution pH. The equilibrium data fit well in the Langmuir model of sorption, showing the formation of monolayer coverage of dye molecules at the outer surface of the sorbent. An equilibrium capacity of sorption has also been evaluated with the pseudo-second order rate equation as well as the Langmuir isotherm and operating line.

Nomenclature

a_L	Constant in Langmuir isotherm (cm^3 g^{-1})
C_0	Initial dye concentration (mg dm^{-3})
E	Activation energy of sorption (kJ mol^{-1})
k	Equilibrium rate constant of pseudo-second order (g mg^{-1}min^{-1})
k_0	Temperature independent factor (g mg^{-1}min^{-1})
K_L	Constant in Langmuir isotherm (m^3 g^{-1})
m_s	Mass of mixed sorbent (g)
q_e	Amount of dyes sorbed at equilibrium (mg g^{-1})
q_m	Equilibrium sorption capacity (mg g^{-1})
q_t	Amount of dyes sorbed at time t (mg g^{-1})
R	Gas constant (8.314 J mol^{-1} K^{-1})
r^2	Correlation coefficient
R_e	Percentage of dye removal at equilibrium (%)
S	Agitation speed (rpm)
T	Solution temperature (K)
t	Time (min)
V	Solution volume (dm^3)

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