

COMPARATIVE STUDY ON THE REMOVAL OF ACID VIOLET BY ADSORPTION ON VARIOUS LOW COST ADSORBENTS

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ABSTRACT

The kinetic studies on the removal of Acid Violet-17 (AV) by adsorption on various adsorbents such as fly ash (FA), iron chromium oxide (ICO), lignite coal (LC), kapok fruit shell carbon (KC) and cashew nut shell carbon (CC) have been made and the results have been compared with that of commercial activated carbon (CAC). Effect of various experimental parameters has been investigated using batch adsorption technique at room temperature ($30\pm 1^\circ\text{C}$). The percentage removal of AV increases with decrease in the initial concentration of AV, initial pH and particle size of adsorbent and increases with increase in the contact time and dose of adsorbent. Adsorption data were modeled with the Freundlich and Langmuir adsorption isotherms and various first order kinetic equations at $30\pm 1^\circ\text{C}$. The kinetics of adsorption is found to be first order with intra particle diffusion as one of the rate determining steps. The adsorbent materials like iron-chromium oxide (ICO), kapok fruit shell carbon (KC) and cashew nut shell carbon (CC) could be employed as low cost adsorbents as alternative materials to CAC for the removal of AV.

KEYWORDS: Wastewater treatment, acid violet, Freundlich and Langmuir isotherms, kinetics of adsorption, intra particle diffusion.

INTRODUCTION

Industrial effluents largely affect the ground water and pollute the environment. Colour is the first contaminant to be recognized, since it is visible to human eye. So, effluent treatment for removal of colour is necessary to protect the environment and the aquatic life. Removal of dyes by conventional chemical and biological methods is not effective. Adsorption technique is followed for the removal of dyes. Activated carbon is widely used for the removal of dyes from effluent due to its effectiveness and versatility. Despite of its prolific use in water and wastewater industries commercial AC (CAC) remains an expensive material. This has led researchers to search for low cost materials as alternative adsorbent materials such as nut shell, fly ash, fruit stones and scrap tyres *etc.*, as alternative to CAC (Pollard *et al.*, 1992).

Attempts have been made to prepare AC from agricultural and industrial wastes. Preparation of ACs from a wide range of agro wastes for water purification has recently been reported by Pollard *et al.* (1992). ACs prepared from agricultural wastes have been used as adsorbents (Pollard *et al.*, 1992; Deo and Ali, 1997; Kannan and Rajakumar, 2001; 2002; Kannan and Meenakshisundaram, 2001; 2002).

The present paper aims at assessing the ability of fly ash (FA), iron-chromium oxide (ICO), kapok fruit shell carbon (KC), cashew nut shell carbon (CC), and lignite coal (LC), as economically attractive low cost adsorbents for the removal of acid violet-17 (AV) and to study the kinetics of removal of AV. The objectives of this paper is to study the effect of various

process parameters on the extent of removal of AV, to optimise the process parameters and to apply various adsorption isotherms and first order kinetic equations to the adsorption data.

MATERIALS AND METHODS

Materials

Commercial activated carbon (CAC) was procured from E Merck (India). Raw materials for the preparation of ACs such as kapok fruit shells and cashew nut shells were collected locally. They were washed, dried and cut into small pieces. The raw materials were carbonised (at 250°C), thermally activated with NaHCO₃ (at 700°C), acid digested with 2N HNO₃ solution (at 80°C for 2 h), washed, dried and sieved into discrete particle size (Jayant sieve, India). FA and ICO were procured from Tuticorin Thermal power station and SPIC industry, Tuticorin, Tamil Nadu, respectively and lignite coal (LC) samples were collected from coal mines (Neyveli Lignite Corporation, Neyveli, Tamil Nadu). The materials were acid digested, washed, dried and sieved into discrete particle size (Jayant sieve, India) and stored. Adsorbent samples were thermally activated at 120°C for 2h. The dye (AV), supplied by BDH (India) was used as an adsorbate. Double Distilled (DD) water was used for preparing all the solutions and reagents. Thermostatic incubator shaker (Neolab, India) was used to maintain the temperature (30±1°C).

Adsorption studies

A stock solution of AV (500 mg l⁻¹) was prepared and suitably diluted accordingly to the various required initial concentrations. Adsorption experiments were carried out at room temperature (30±1°C) under batch mode (Kannan, 1991). The concentrations of the AV solutions before and after adsorption were estimated by measuring O.D. at 545 nm (λ_{max}) with the help of UV-Visible spectrophotometer (ELICO, Model SL-159, India) and then interpolated into the standard curves. Exactly 50 ml of AV solution of known initial concentration (C_i) was shaken at constant agitation speed (200 rpm) with required dose of adsorbent of a fixed particle size (75 microns) for a specific period of contact time (Table 1). The initial pH of the dye solution was adjusted to the required value by adding either 1M HCl or 1M NaOH solution. Initial pH values of dye solutions were noted with digital pen pH meter (Hanna instruments, Portugal). After equilibration, the final concentration (C_f) of AV was measured by using a UV-Visible spectrophotometer (ELICO, Model SL-159, India). The values of percentage removal and the amount of dye adsorbed were calculated using following relationships:

$$\text{Percentage removal} = \frac{C_i - C_f}{C_i} \times 100$$

$$\text{Amount adsorbed } q_e = \frac{C_i - C_f}{m}$$

where, C_i = initial concentration (ppm)

C_f = final concentration (ppm)

m = mass of adsorbent (g l⁻¹)

Control experiments were carried out and the average values of duplicate runs were obtained and analysed (Error: ±1-2% for percentage removal and ±0.005-0.01 mg g⁻¹ for amount adsorbed).

RESULTS AND DISCUSSION

The adsorption experiments were carried out at different experimental conditions (Table 1) and the results obtained are discussed below:

Effect of initial concentration

The effect of initial concentration of Acid Violet-17 (AV) on the extent of removal of AV (in terms of percentage removal and amount adsorbed (q) on various adsorbents were studied. The relevant data are given in Table 2. The percentage removal was found to decrease exponentially, while the amount of dye adsorbed increased exponentially with the increase in initial concentration of AV. This indicates a decrease in adsorption, which is attributed due to

the lack of available active sites required for the high initial concentration of AV. Similar results have been reported in literature on the extent of removal of dyes (Annadurai and Krishnan, 1996; Kannan and Meenakshisundaram, 2001; 2002; Deo and Ali, 1997; McKay *et al.*, 1985), metal ions (Kannan and Rajakumar, 2001; 2002) and carboxylic acids (Kannan and Karuppasamy, 1998; Kannan and Xavier, 2001). The optimum initial concentration of dye solution (AV) was fixed for various adsorption systems and given in Table 1.

Table 1. Experimental conditions of adsorption experiment for the removal of AV by various adsorbents at 30°C

S.No.	Variation	Adsorbents	Initial dye concentration (ppm)	Dose (g l ⁻¹)	Contact time (min)	Initial pH	Particle size of adsorbents (µm)
I	Initial dye concentration	CAC	115 - 145	FA - 10	30	solution pH*	75
		FA,ICO,KC,CC,LC	6 - 12.	Other adsorbents - 2			
II	Contact time	CAC	CAC - 140	FA - 10	5 - 60	solution pH*	75
		FA,ICO,KC,CC,LC	Other adsorbents - 20	Other adsorbents - 2			
III	Dose	CAC	CAC - 140	CAC - 2 - 3.2	40	solution pH*	75
		FA,ICO,KC,CC,LC	Other adsorbents - 20	FA - 8 - 20 ICO,KC,CC,LC - 1 - 7			
IV	Initial pH	CAC	CAC - 140	FA - 10	40	4 - 8	75
		FA,ICO,KC,CC,LC	Other adsorbents - 20	Other adsorbents - 2			
V	Particle size of adsorbents	Other than CAC	FA,ICO,KC,CC, LC - 20	FA - 10	40	solution pH*	75 - 250
		FA,ICO,KC,CC,LC		Other adsorbents - 2			
VI	Ionic Strength Range (0.05 - 0.3)	CAC	CAC - 140	FA - 10	40	solution pH*	75
		FA,ICO,KC,CC,LC	Other adsorbents - 20	Other adsorbents - 2			

*Solution pH: 6.6

Adsorption isotherms

The adsorption data were analysed with the help of the following linearised forms of Freundlich and Langmuir isotherms (Adamson, 1960):

$$\text{Freundlich isotherm: } \log q_e = \log K + \frac{1}{n} \log C_e$$

$$\text{Langmuir isotherm : } \frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o}$$

where, K = adsorption capacity

(1/n) = order/intensity of adsorption

q_e = amount of AV adsorbed per unit mass of adsorbent (mg g⁻¹)

C_e = equilibrium concentration of dye (ppm)

Q_o = monolayer (maximum) adsorption capacity (mg g⁻¹)

b = Langmuir constant related to energy of adsorption (l mg⁻¹)

The values of Freundlich and Langmuir parameters have been obtained, respectively, from the linear correlation between the values of (i) $\log q_e$ and $\log C_e$ and (ii) (C_e/q_e) and C_e (Table 3).

Table 2. Effect of Process parameters on the extent of removal of AV by various adsorbent at 30°C

Process Parameter varied	Parameter*	CAC	FA	ICO	KC	CC	LC
Initial concentration (ppm.)	%R	96.94-91.93	68.55-37.11	59.12-51.26	33.96-21.38	16.14-13.00	27.67-18.76
	q	55.74-66.65	0.411-0.445	1.774-3.075	1.019-1.283	0.484-0.780	0.830-1.126
Contact time (min.)	%R	79.78-95.55	9.120-27.99	38.36-42.77	22.01-31.45	3.460-15.41	15.09-27.04
	q	55.85-66.89	0.182-0.560	3.836-4.277	2.201-3.145	0.346-1.541	1.509-2.704
Dose (g l ⁻¹)	%R	92.41-98.56	79.20-89.50	24.84-84.91	18.24-49.37	10.38-31.76	14.47-58.49
	q	64.69-43.12	0.865-0.670	4.969-2.426	3.648-1.411	2.075-0.907	2.893-1.671
Initial pH	%R	94.16-92.14	71.70-0.630	59.12-22.01	42.77-24.53	23.27-9.430	24.21-19.81
	q	65.91-64.50	1.434-0.013	5.912-2.201	4.277-2.453	2.327-0.943	2.421-1.981
Particle size (in micron)	%R	22.64-2.200	42.45-18.87	28.93-4.720	12.58-5.660	20.13-4.400
	q	0.453-0.044	4.245-1.887	2.893-0.472	1.258-0.566	2.013-0.440
Ionic Strength Range (0.05 – 0.3)	%R	92.59-98.97	22.01-33.33	42.45-63.52	28.30-63.21	13.52-36.48	20.75-36.48
	q	64.81-69.28	0.440-0.667	4.245-6.352	2.830-6.321	1.352-3.648	2.075-3.648

* percentage removal (%R) and amount adsorbed (q in mg g⁻¹)

Table 3. Freundlich and Langmuir parameters of adsorption isotherms for the removal of AV by various adsorbents at 30°C

Model	Parameter	CAC	FA	ICO	KC	CC	LC
Freundlich isotherm	K-values	47.00	0.396	1.007	0.592	0.157	0.513
	Slope (1/n)	0.140	0.051	0.624	0.354	0.680	0.357
	Correlation coefficient (r)	0.981	0.935	0.995	0.964	0.993	0.950
	Δq(%)	0.006	0.003	0.014	0.036	0.016	0.044
Langmuir isotherm	Q ₀	72.01	0.454	6.493	1.780	1.975	1.523
	b	0.917	3.803	0.148	0.285	0.061	0.303
	Correlation coefficient (r)	0.999	0.999	0.976	0.994	0.963	0.991
	Δq(%)	0.009	0.010	0.026	0.046	0.022	0.030
	10 ² R _L	0.837	2.983	43.60	27.50	64.90	27.60

In order to compare the validity of each model (isotherm) more efficiently a normalised standard deviation, Δq(%) is calculated using the following equation:

$$\Delta q(\%) = 100 \times \left[\frac{\sum \left(\frac{q_t^{\text{exp}} - q_t^{\text{cal}}}{q_t^{\text{exp}}} \right)^2}{n-1} \right]^{\frac{1}{2}}$$

where, the superscripts, exp. and cal. are the experimental and calculated values of q_t viz., the amount adsorbed at different time t and n is the number of observations.

The Δq(%) values are also given in Table 3. Based on the low values of Δq(%), it is concluded that the adsorption of AV can be best described by the Langmuir adsorption isotherm. Langmuir isotherm plots are shown in Figure 1. The monolayer adsorption capacities (Q₀) of the adsorbents are found to be of the order given below:

$$FA < LC < KC < CC < ICO \ll CAC$$

Further, the essential characteristics of the Langmuir isotherm can be described by a Separation factor, R_L , which is defined by the following equation (Hall, *et al.*, 1966):

$$R_L = \frac{1}{1 + bC_i}$$

where, C_i = optimum initial concentration of AV (mg l^{-1})

b = Langmuir constant (l mg^{-1}).

The values of separation factor, R_L , indicate the nature of the adsorption process as given below:

R_L values	Nature of adsorption process
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

In the present study, the values of R_L (Table 3) are observed to be fraction *i.e.*, in the range of zero to one (0.0298-0.649), which indicate that the adsorption process is favourable for all these adsorbents.

Effect of contact time

The effect of contact time on the amount of dye adsorbed was observed at the optimum initial concentration of dye. The relevant data are also given in Table 2. The extent of removal of AV by these adsorbents is found to increase and reach a maximum value with increase in contact time. The relative increase in the extent of removal of dye after 40min. of contact time is not significant and hence it is fixed as the optimum contact time. Similar results have been reported in literature for the removal of dyes (Deo and Ali, 1997; Kannan and Meenakshisundaram, 2002; McKay, 1985) metal ions (Kannan and Rajakumar, 2001; 2002) and carboxylic acids (Kannan and Karuppasamy, 1998; Kannan and Xavier, 2001).

Kinetics of adsorption

The kinetics and dynamics of adsorption of AV on various adsorbents have been studied by applying the various first order kinetic equations (Kannan and Rajakumar, 2001; 2002; Kannan and Meenakshisundaram, 2001; 2002):

Natarajan and Khalaf equation:
$$\log \frac{C_i}{C_t} = \frac{k}{2.303} t$$

Lagergren equation:
$$\log(q_e - q_t) = \log q_e - \frac{k}{2.303} t$$

Bhattacharya and Venkobachar equation:
$$\log[1 - U(T)] = -\frac{k}{2.303} t$$

where:

C_i and C_t = concentration of AV at time zero and time t , respectively (mg l^{-1})

q_e and q_t = amount of AV adsorbed at equilibrium time and time t , respectively (mg g^{-1})

$U(T) = [(C_i - C_t) / (C_i - C_e)]$

C_e = equilibrium AV concentration (mg l^{-1})

k = first order adsorption rate constant (min^{-1}).

The values of (i) $\log(C_i/C_t)$, (ii) $\log(q_e - q_t)$ and (iii) $\log[1 - U(T)]$ were linearly correlated with time (t). The values of first order rate constant, $\Delta q(\%)$ and correlation coefficient (r -values) are given in Table 4. All the linear correlations were found to be statistically significant, indicating the applicability of these kinetic equations and the first order nature of the adsorption process of AV on these low cost adsorbents. The rate of adsorption is found to be high in ICO (0.107 min^{-1}) and low in LC (0.061 min^{-1}) as evidenced by k -values. Lagergren plots are shown in Figure 2.

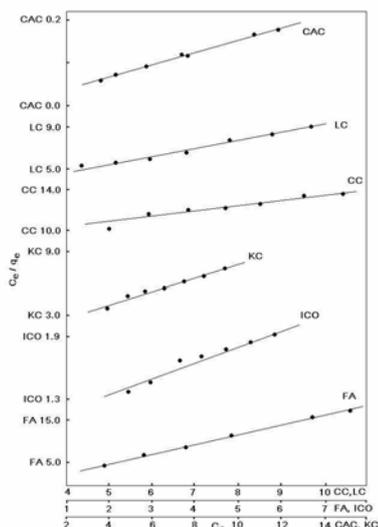


Figure 1. Langmuir isotherm plots for the removal of AV by adsorption on various adsorbents

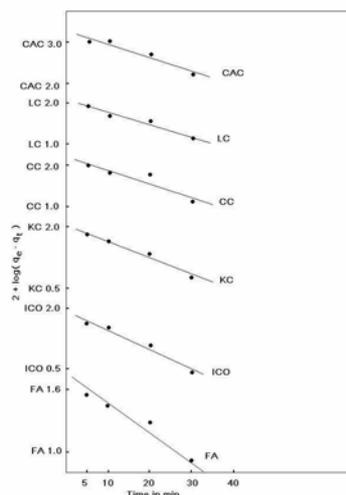


Figure 2. Lagergren plots for the removal of AV by adsorption on various adsorbents

Intra particle diffusion model

The possibility of intra particle diffusion process was explored by using the Weber and Morris intra particle diffusion model (Weber and Morris, 1964):

$$q_t = k_p t^{1/2} + C$$

where:

q_t = amount of AV adsorbed at time, t (mg g^{-1})

C = intercept

k_p = intra particle diffusion rate constant ($\text{mg g}^{-1} \text{min}^{-1/2}$).

Table 4. Statistical results of the application of the kinetic equations and models for AV dye by various adsorbent systems at 30°C

Parameter	Carbon					
	CAC	FA	ICO	KC	CC	LC
<i>I. Natarajan and Khalaf equation</i>						
$10^2 k$	3.311	0.452	0.133	0.227	0.222	0.244
r-value	0.959	0.972	0.880	0.960	0.911	0.984
$\Delta q(\%)$	22.90	22.80	60.01	50.35	27.37	43.72
<i>II. Lagergren equation</i>						
$10^2 k$	7.472	5.566	10.71	9.477	8.398	6.112
r-value	0.966	0.975	0.989	0.992	0.991	0.978
$\Delta q(\%)$	9.437	1.734	6.332	5.577	3.757	9.355
<i>III. Bhattacharya and Venkobachar equation</i>						
$10^2 k$	7.472	5.566	10.71	9.477	8.398	6.112
r-value	0.966	0.975	0.989	0.992	0.991	0.978
$\Delta q(\%)$	12.67	17.42	7.929	9.400	10.99	15.83
<i>IV. Intra particle diffusion model</i>						
$10^2 k_p$	239.5	7.488	8.490	17.05	21.16	19.50
Intercept	50.15	0.012	3.699	1.896	0.065	1.113
r-value	0.965	0.989	0.938	0.983	0.957	0.988
$\Delta q(\%)$	0.027	0.123	0.016	0.033	5.576	0.062
<i>V. log (% removal) vs log (time)</i>						
Slope	0.088	0.485	0.049	0.144	0.570	0.213
Intercept	1.831	0.608	1.550	1.245	0.246	1.031
r-value	0.967	0.993	0.974	0.992	0.952	0.985
$\Delta q(\%)$	64.77	16.45	54.03	54.01	54.43	53.93

The values of q_t were found to be linearly correlated with values of $t^{1/2}$. The k_p values were calculated by using correlation analysis (Table 4). The r -values are found to be close to unity, indicating the application of this model. This reveals the presence of intra particle diffusion process (Weber and Morris, 1964; Deo and Ali, 1997). The calculated values of k_p is maximum for CC (0.212) and minimum for ICO (0.008). This result indicates that the CC is more porous than the other low cost adsorbents. The intra particle diffusion plots are given in Figure 3. The values of intercept (Table 4) give an idea about the boundary layer thickness *i.e.*, the larger the intercept, the greater is the boundary layer effect (Deo and Ali, 1997; McKay, 1983). The intercept (C) value is maximum in ICO (3.699) and minimum in CC (0.065) indicating that the boundary layer effect is maximum in ICO and minimum in CC, among the indigenously prepared carbons.

The correlation of the values of \log (% removal) and \log (time) also resulting in linear relationship, which indicates that the process of intra particle diffusion is taking place in these adsorption system (Table 4). The divergence in the values of slope from 0.5 (min. – max. =0.049 – 1.012) indicates the presence of intra particle diffusion process as one of the rate limiting steps, besides many other processes controlling the rate of adsorption, all of which may be operating simultaneously (Deo and Ali, 1997).

Effect of dose of adsorbent

The effect of dose of adsorbent on the amount of AV adsorbed was studied (Table 2). The equilibrium value of amount adsorbed was observed to decrease with increase in dose of adsorbent. The percentage removal (Table 2) increases with increase in dose of adsorbent. This may be due to the increase in availability of surface active sites resulting from the increased dose and conglomeration of the adsorbent (Kannan and Karuppasamy, 1998). The amount of AV adsorbed was observed to vary exponentially in accordance with the fractional power term of the dose of adsorbent *i.e.* (dose)⁻ⁿ, where n = fraction. The n -values (r -values) are CAC=0.848 (0.999), FA=0.27 (0.990), ICO=0.3602 (0.990), KC=0.498 (0.995), CC=0.3608 (0.944) and LC=0.302 (0.909). The plots of $\log q_e$ vs \log (dose) were found to be linear (r = unity). This suggests that the adsorbed species/solutes may either block the access to the internal pores or cause particles to aggregate and there by resulting in decrease in the availability of active sites for adsorption. Similar results have been noticed in literature for the removal of dyes (Deo and Ali, 1997; Kannan and Meenakshisundaram, 2002; McKay, 1985) metal ions (Kannan and Rajakumar, 2001; 2002) and carboxylic acids (Kannan and Karuppasamy, 1998; Kannan and Xavier, 2001).

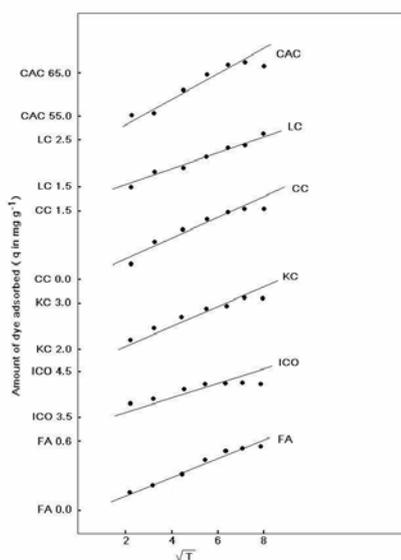


Figure 3. Intra particle diffusion plots for the removal of AV by adsorption on various adsorbents

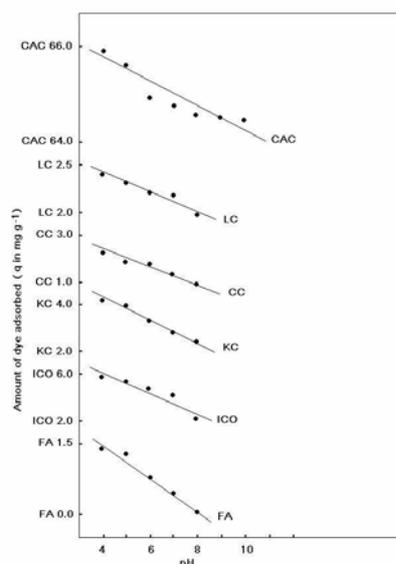


Figure 4. Effect of pH on the removal of AV by adsorption on various adsorbents

Effect of initial pH

The effect of initial pH of the dye solution on the amount of AV adsorbed was studied by varying the initial pH, under constant conditions of other process parameters (Table 2). The pH value changes slightly after adsorption and a decrease in pH value is noted ($\Delta\text{pH} = \text{pH}_{\text{final}} - \text{pH}_{\text{initial}}$). The optimum pH value is fixed as 6.6. The results are shown in Figure 4. The increase in initial pH, decrease the amount of AV adsorbed. The change in initial pH values of dye solution significantly affect the adsorption characteristics of acidic dye indicating that removal of AV (Acidic dye) is enhanced by acidic solution.

Effect of particle size of the adsorbent

The amount of AV adsorbed increases with the decrease in particle size of the adsorbent. This is due to the increase in available surface area with the decrease in particle size. There exists a linear relationship between the amount of dye adsorbed and particle size, as evidenced by the r-value close to unity (r-value for FA=0.938, ICO=0.924, KC=0.912, CC=0.983 and LC= 0.934). Similar observations have been reported for the adsorption of dyes (Deo and Ali, 1997; Kannan and Meenakshisundaram, 2001; McKay *et al.*, 1985; McKay, 1983).

Effect of ionic strength

The increase in the percentage of removal with increase in the ionic strength of electrolytes (NaCl, NaNO₃ and Na₂SO₄) for the adsorbents (CAC, FA, ICO, KC, CC and LC) indicate that the adsorption is sensitive to the change in ionic strength, which indicate that electrostatic attraction is a significant mechanism for sorption process. The increase in adsorption with ionic strength may be due to the compression of the thickness of the diffused double layer (Vinod, 2004). Such compression may help the sorbent particle and sorbate species to approach each other more closely, by then the attractive forces become significant, leading to increased adsorption. These results indicate that electrostatic attraction plays a significant role in the removal of AV (Vinod, 2004).

CONCLUSIONS

Acid Violet-17 (AV) is found to adsorb strongly on the surface of carbons. The percentage removal of AV increases with decrease in the initial concentration of the dye (AV), initial pH and particle size of adsorbent, and increases with increase in the contact time and dose of adsorbent. Adsorption data were modeled with the Freundlich and Langmuir adsorption isotherms and various first order kinetic equations at $30 \pm 1^\circ\text{C}$. The results suggest that pore diffusion *i.e.* intra particle diffusion is more important. Adsorption behaviour is described by a monolayer Langmuir type isotherm. The adsorption process is found to be first order with intra particle diffusion, as one of the rate determining steps. The present study concludes that ICO and CC could be employed as low cost adsorbents as alternative to CAC for the removal of AV.

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