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COMPARATIVE EVALUATION OF COMMERCIAL AND SEWAGE SLUDGE BASED ACTIVATED CARBONS FOR THE REMOVAL OF TEXTILE DYES FROM AQUEOUS SOLUTIONS

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ABSTRACT

The sorption of dyes from aqueous solutions on to sludge-based activated carbon have been studied and compared with commercial activated carbon. Adsorption parameters for the Langmuir and Freundlich isotherms were determined and the effects of effluent pH, adsorbent dosage, contact time and initial dye concentration were studied. A pseudo-second order kinetic model has been proposed to correlate the experimental data.

Key words: Kinetics, textile dye, sorption, sludge based activated carbon, commercial activated carbon

INTRODUCTION

Almost every industry uses coloring matter to color their products and the residual and unspent substances are discharged in to the environment, particularly aquatic environment. Discharge of such colored effluents imparts color to the receiving water and interferes with its intended beneficial use. In addition, recent reports suggest that several color-causing substances are micro toxic to the aquatic biota. Moreover if effort is not made to remove this visible pollution, public assumes that little effort is being expanded in removing other pollutants also. It is thus imperative that color must be removed from the effluent before disposal. Among different industrial wastewaters with different types of color causing substances, synthetic textile organic dye color wastes occupy a prominent position. Over 7 x 10^5 tons and approximately 10000 different types of dyes and pigments are produced worldwide annually. It is estimated that 10 to 15% of the dye is lost in the effluent during the dyeing process (Young *et al*., 1997; Danis *et al*., 1998). Various physical, chemical, physico-chemical and bio-chemical processes like sedimentation, equalization, neutralization, flotation, chemical precipitation, coagulation and flocculation, adsorption, ion-exchange, reverse osmosis, electro-chemical coagulation etc., have been investigated for the removal of dyes from aqueous solutions (McKay, 1982; Barton, 1987; Karithikeyan, 1990; karthikeyan, 1988; Venkatamohan, 1997). The stringent and rigid national effluent disposal standards call for an advanced treatment among which liquid-phase adsorption has been found highly efficient for removal of color in terms of initial cost, simplicity of design, ease of operation and insensitivity to toxic substances. Activated carbon is the most widely used adsorbent because it has excellent adsorption efficiency for organic compounds but its use is usually limited due to its high cost (Y. S. Ho *et al*., 1999; Rozada *et al*., 2003). In recent years this has prompted a growing research interest in the production of carbon based adsorbents from a range of residues – mainly industrial or agricultural by products (Malik, 2003; Chiou and Li, 2003; Garg *et al*., 2003; Ozacar and Sengil, 2003; Shukla *et al*., 2002; Ramakrishna and Viraraghavan, 1997). The production of activated carbon from residues may allow waste producers to offset their increasing waste disposal cost

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against the cost of carbon production, while saving the non renewable natural resources and producing a valuable product with potential applications in pollution control.

The main focus of this research is to evaluate the adsorption potential of sewage sludge derived activated carbon (SAC) for the removal of dyes from aqueous solution. The effects of dose of SAC, aqueous solution pH and initial dye concentration in the aqueous solution on sorption of dye were investigated. The Langmuir and Freundlich isotherms were used to fit the equilibrium data. Similar experiments were carried out with commercial grade activated carbon (CAC) to compare the results.

MATERIALS AND METHODS

Sorbate and sorbents

The anaerobically digested and dewatered sewage sludge collected form Koyambedu metro sewage treatment plant, Chennai, TamilNadu, India, was used for making activated carbon as per the procedure suggested by Sreedharreddy, S. (2004). Characteristics of SAC in comparison with CAC are presented in Table: 1. Surface area of activated carbon was measured as per guide lines of Poots *et al.*, (1976). The textile dye laden aqueous solution was prepared as a mixture of three reactive dye formulations (Table: 2), as per the procedure suggested by Sreedhar reddy *et al.*, (2005). According to the information obtained from the local dyeing industries, usually 20% of the dyestuffs (hydrolyzed and / or unfixed form) and 100% of all assisting chemicals remain in the exhausted dye-bath. Dye-bath was suitably diluted to represent the typical strength of effluents from the washing and rising stages in the above-mentioned textile manufacturing plant. Composition of suitably diluted exhausted dye-bath is presented in Table 3.

Table 1: Properties of activated carbons used in the investigation

Activated carbon	Apparent density (gm/L)	Iodine number	Surface area (m^2/g of activated carbon)
SAC	873.2	6.9211	928.26
CAC	291.7	1249.753	1083.42

Table 2: The properties of dyes used in the investigation

Properties	Procion brilliant Blue M-R	Procion brilliant Red H-8G	Procion turquoise Blue M-G
Form	Powder	Powder	Powder
Color Index	Bright Blue (Rb4)	Red	Bright Greenish Blue (Rb140)
Odor	Odorless	Odorless	Odorless
Chromophore	Anthraquinone	Anthraquinone	Copper Pthalocyanine
Color Index Number	61205	-	74180
Reactive Group	Dichlorotriazine	Monochlorntriazine	Dichlorotriazine
pH Value	7 (10 g/L @ 25 ^{0}C)	7 (10 g/L @ 25 ^{0}C)	7 (10 g/L @ 25 ^{0}C)

Table 3: Characteristics and composition of synthetic textile dyeing unit effluent

Sample No	Parameter	Concentration
1	Dye	120
2	pH	8.0
3	Total solids	7500
4	Total suspended solids	320
5	Total dissolved solids	7180
6	BOD_3 at 27 ^{0}C	560
7	COD	3200
8	Chlorides (as Cl^-)	3800
9	Sodium (as Na^+)	7900

*All the values except pH are in mg/L

Batch adsorption studies

Batch experiments were conducted to evaluate the adsorption potential of SAC and CAC for dyes from aqueous solutions. 100 mL of dye solution of known concentration (C_o) and initial pH was taken in a 300 mL capacity borosil made reagent bottle, with a required amount of adsorbent and was agitated at a speed of 100 rpm in a horizontal bench shaker (laboratory net scientific) at room temperature, for a specified period of contact time. Then the solution was filtered through a Whattman filter paper. The initial pH of the solution was adjusted by using either 0.1N NaOH or H_2SO_4. Spectornic made spectrophotometer was used to determine the concentration of dye at a wavelength corresponding to maximum absorbance. The filtrate was analyzed for the remaining dye concentration. The amount of dye adsorbed in mg/g at time, t was computed by using the following equation:

$$qt = \frac{(Co - Ct)V}{m_S} \qquad (1)$$

Where C_o and C_t are the dye concentrations in mg/L initially and at a given time t, respectively. V is the volume of the dye solution in mL, and m_s is the weight of activated carbon in grams.

Kinetic model theory

The reaction rate of a chemical reaction is defined as the change in concentration of a reactant or product per unit time. The concentrations of the products do not appear in the rate law because the reaction rate is being studied under conditions where the reverse reactions do not contribute to the overall rate. The reaction order and rate constant must be determined by experiment. In 1995, Ho presented a sorption pseudo-second order rate law expression showed how the rate depends on the sorption capacity on solid phase but not the concentration of the sorbate (Ho, Y.S., 1995; Ho, Y.S. and McKay, G., 2000).

$$\frac{dq_t}{dt} = K(q_e - q_t)^2 \qquad (2)$$

Integrated pseudo-second order rate law can be obtained from Eq. (2) for the boundary conditions t=0–t and q_t=0– q_t, gives:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + Kt \qquad (3)$$

Eq. (3) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{Kq_e^2} + \frac{1}{q_e^2}t \qquad (4)$$

Where q_e is the amount of sorbate sorbed at equilibrium, (mg/g); t is the reaction time, (min); q_t is the amount of sorbate sorbed at time t, (mg/g); k is the equilibrium rate constant of pseudo-second order sorption, (g/mg min). Ho's pseudo-second order rate law shows how the sorption capacity of sorbate depends on time. If the equilibrium sorption capacity of sorbate and the rate constant *k* are known, then the sorption capacity of sorbate at any time can be calculated. Using the linear form equation, Eq. (4), plotting t / q_t, versus time,t will give a straight line with slope $1/ q_e$, and intercept $1/kq_e^2$.

RESULTS

Equilibrium studies

The equilibrium studies conducted at a fixed initial concentration and varying adsorbent dose have been fitted in to the linearized Freundlich adsorption isotherm, which is of the form,

$$\text{Ln }(X/M) = \ln K_f + 1/n \ln C_e \qquad (5)$$

Where X is the amount of dye removed (mg), m is the weight of adsorbent used (g), C_e is the equilibrium concentration and K_f and n are constants incorporating all factors affecting the adsorption process such as adsorption capacity and intensity, respectively.

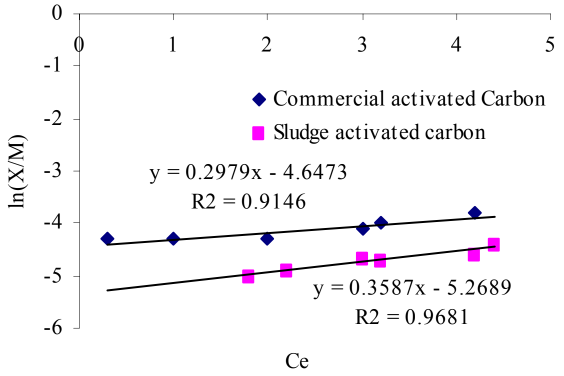

Fig.1: Freundlich isotherm for commercial and sludge derived activated carbons

From Fig.1 Freundlich constants K_f and n were found to be 9.2 mg/g and 2.45 for CAC, 4.6 mg/g and 2.116 for SAC respectively. The Langmuir isotherm, which is in the form of Eq. 6, was applied for adsorption equilibrium.

$$C_e/q_e = 1/Q_o b + C_e/Q_o \qquad (6)$$

Where C_e is the equilibrium concentration (mg/L), and q_e in the amount of dye adsorbed per gram at equilibrium (mg/g). Q_o and b are Langmuir constants. Q_o and b were determined from the slope and intercept of the plot (Fig.2) and found to be 33.5 mg/g and 0.070 l/mg, for CAC and 25mg/g and 0.44 l/mg, for SAC, respectively.

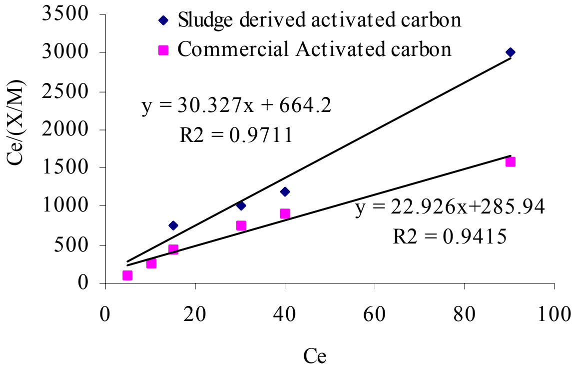

Fig. 2: Langmuir isotherm for commercial and sludge derived activated carbons

Effect of initial concentration

The experimental results of the sorption of dye from aqueous solutions at various initial concentrations on to SAC and CAC are shown in Table 4. The equilibrium sorption capacity of SAC and CAC increased from 20 to 32.03 mg/g and 20.12 to 42 mg/g respectively with an increase in the concentration of reactive dye from 20 mg/L to 120 mg/L with an adsorbent dose of 1 g/L and at a solution pH of 3. Fig. 3 shows a plot of t/q_t against t, for the sorption on to SAC and CAC, for the pseudo – second order model. The correlation coefficients (R^2), the pseudo-second order rate parameter (k_e), the initial sorption rate (h) and the equilibrium sorption capacity (q_e) are shown in Table: 4. The solid line curves in Fig. 4 are the theoretical plots of equation (4), and the values of R^2 are all extremely high (>0.997).

Table 4: Amount of reactive dye sorbed on to SDAC and CAC at various reactive dye concentrations

Time (Min)	q_t(mg/g)									
	Co=20mg/L		Co=50mg/L		Co=75mg/L		Co=100mg/L		Co=120mg/L	
	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC
30	18.072	19.011	24	30	25	31.57	26.54	33	27	36
60	20	20	27.64	32.5	27.52	37.5	30	39.63	31	42
90	20	20.12	27.67	33	28.037	39.13	31.57	39.64	32.028	42
120	20	20.12	28	33	28.037	39.13	31.57	39.6	32.028	42

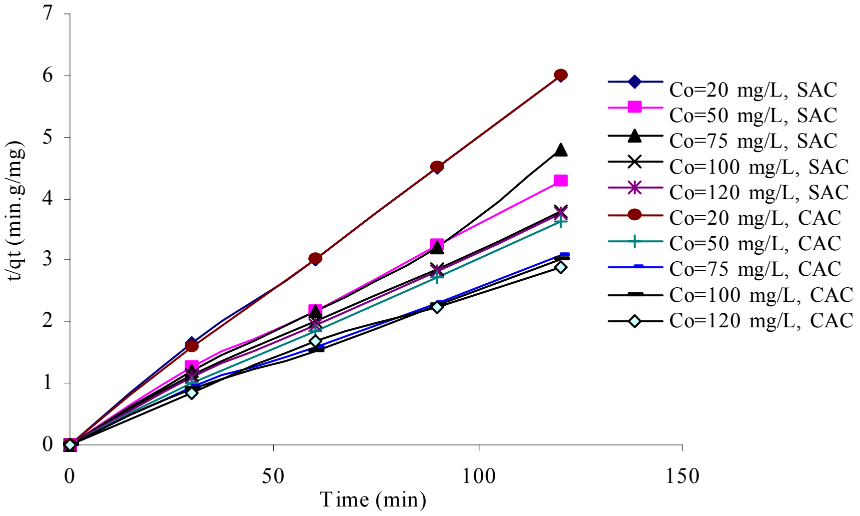

Fig. 3: Pseudo-second order sorption kinetics of reactive dye on to SAC and CAC at various initial dye concentrations

Table 5: Pseudo-second order sorption kinetic model constants at different initial dye concentrations

Initial dye Concentration (mg/L)	q_e(mg/g)		K(g/mg.min)		h(mg/g.min)		R^2	
	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC
20	20	20.12	0.038	0.079	15.2	31.98	0.999	0.999
50	28.40	33.33	0.018	0.021	14.5	23.32	0.998	0.999
75	30.12	40.16	0.015	0.0107	13.60	17.25	0.999	0.996
100	32.15	40.48	0.0136	$7.14\text{x}10^{-3}$	11.74	11.69	0.997	0.997
120	32.67	41.84	0.0105	$5.88\text{x}10^{-3}$	11.20	1029	0.997	0.991

Effect of adsorbent dosage:

The sorption kinetics of reactive dye on to SAC and CAC are shown in Table 6. The Table shows that by that by increasing the adsorbent dose, the percentage dye removal increases, but adsorption density, i.e. the amount of dye adsorbed per unit mass, decreased.

Table 6: Amount of reactive dye sorbed on to SDAC and CAC at various SDAC and CAC loadings

Time (min)	q_t(mg/g)									
	Co=120 mg/L									
	SAC (1g/L)	CAC (1g/L)	SAC (3 g/L)	CAC (3 g/L)	SAC (5 g/L)	CAC (5 g/L)	SAC (7 g/L)	CAC (7 g/L)	SAC (9 g/L)	CAC (9 g/L)
30	27	36	12	16	9.6	12	8.57	9.42	7.33	8
60	31	39	22	26	15.6	18	12	13.71	10	11.33
90	32.028	39.4	24	28	16.8	19.2	12.85	14.57	10.66	12
120	32.028	39.6	25.2	29.2	17.52	19.92	14.22	15.94	12	12.93

Fig.4 shows a plot of t/q_t against t for the sorption on to SAC and CAC, at various masses of adsorbent for the pseudo second order model. The correlation coefficients (R^2), the pseudo-second order rate parameter (k_e), the initial sorption rate (h) and the equilibrium sorption capacity (q_e) as a function of adsorbent dosages are shown in Table: 7.When increasing the adsorbent dosage from 1 g/L to 10 g/L, at pH=3, the equilibrium sorption capacity (q_e) decreased form 38.46 to 14.22 mg/g for sludge derived activated carbon and from 40.81 to 14.20 mg/g for commercial activated carbon.

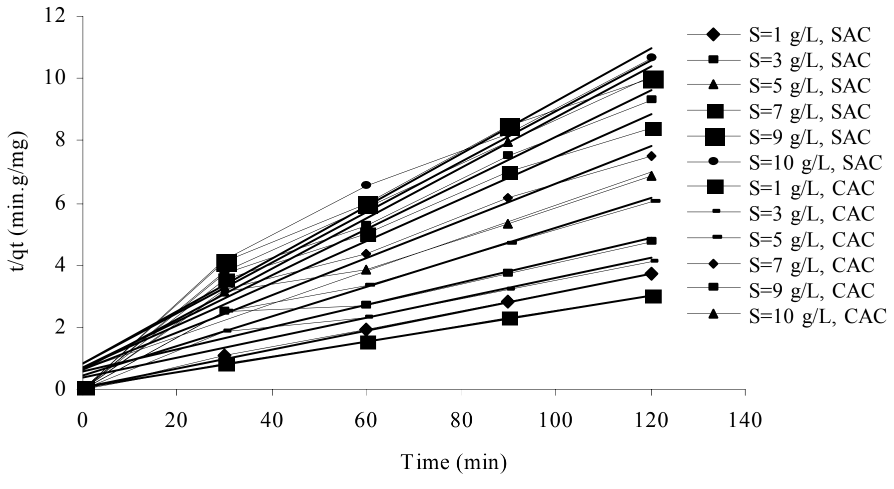

Fig. 4: Pseudo-second order sorption kinetics of reactive dye on to SAC and CAC at various adsorbent dosages

Table 7: Pseudo–second order sorption kinetic model constants at different adsorbent dosages

Adsorbent dosage (g/L)	$q_{e,}$(mg/g)		K(g/mg.min)		h (mg/g.min)		R^2	
	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC
1	38.46	40.81	$4.359\text{x}10^{-4}$	$6.56\text{x}10^{-4}$	0.648	1.092	0.99	0.99
3	35.24	39.52	$5\text{x}10^{-4}$	$6.84\text{x}10^{-4}$	0.63	1.068	0.99	0.97
5	23.69	25.81	$1.093\text{x}10^{-3}$	$1.36\text{x}10^{-3}$	0.613	0.90	0.99	0.98
7	17.84	20.24	$1.756\text{x}10^{-3}$	$1.517\text{x}10^{-3}$	0.55	0.621	0.99	0.99
9	14.92	15.94	$2.145\text{x}10^{-3}$	$2.24\text{x}10^{-3}$	0.47	0.569	0.99	0.99
10	14.22	14.20	$2.32\text{x}10^{-3}$	$2.62\text{x}10^{-3}$	0.46	0.52	0.99	0.99

Effect of pH

To investigate the role of surface chemistry of SDCAC and CAC, on the adsorbent capacity of the reactive dyes, the effect of pH at equilibrium was studied. The experiments were carried out by varying the initial pH from 3 to 11, under a constant initial dye concentration of 120 mg/L, and a carbon dosage of 10 g/L. The dependence of the equilibrium adsorption capacity of SAC and CAC for reactive dye on equilibrium pH is presented in Table 8. Equilibrium sorption capacity of both the activated carbons decreased with increasing pH values of reactive dye solution. The maximum removal of reactive dye with sludge derived activated carbon and commercial activated carbon was hence observed at pH: 3 when the negative charge on the surface is very much reduced due to the excess of protons in solution. For SAC and CAC, the amount of dye adsorbed at equilibrium increased from 5.21 to 11.3 mg/g and 5.5 to 12.8 mg/g respectively with decrease in pH from 11 to 3. Results from a series of kinetic experiments at different pH conditions are shown in Table: 9.

Table 8: Amount of reactive dye sorbed on to SDAC and CAC at varying pH conditions of reactive dye effluent

Time (min)	q_t(mg/g)									
	pH =3		pH =5		pH =7		pH =9		pH =11	
	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC
30	7.2	7.92	5.28	6.48	4.08	5.28	3	4.2	2.52	3.12
60	9.12	10.32	7.68	9	6.48	7.7	5.4	6.6	3.72	4.2
90	10.94	11.28	8.88	10.08	7.68	8.9	6.96	8.04	5.64	5.88
120	11.304	11.88	10.32	10.92	9	9.96	7.92	9.12	7.2	8.28

Table 9: Pseudo–second order sorption kinetic model constants at various pH conditions

pH	$q_{e,}$(mg/g)		K(g/mg.min)		h (mg/g.min)		R^2	
	SAC	CAC	SAC	CAC	SAC	CAC	SAC	CAC
3	14.26	14.99	$2.29\text{x}10^{-3}$	$2.22\ \text{x}10^{-3}$	2.14	2	0.995	0.997
5	12.77	13.75	$1.781\text{x}10^{-3}$	$1.42\ \text{x}10^{-3}$	3.44	3.76	0.996	0.993
7	12.59	12.5	$1.42\ \text{x}10^{-3}$	$1.4\ \text{x}10^{-3}$	4.42	4.5	0.999	0.996
9	12.30	11.11	$1.038\ \text{x}10^{-3}$	$1.045\ \text{x}10^{-3}$	8.38	8	0.996	0.999
11	9.85	10	$7.88\ \text{x}10^{-4}$	$9.9\ \text{x}10^{-4}$	9.92	10.095	0.992	0.991

The data shows a good compliance with the pseudo-second-order equation and the regression coefficient (R^2) for the linear plots of Fig. 5. It is found that the equilibrium sorption capacity (q_e), the rate constant, k and the initial sorption rate, h are a function of pH. The ultimate sorption capacity of sludge derived activated carbon and commercial activated carbon has decreased from 14.26 mg/g to 9.85 mg/g and 14.99 mg/g to 10 mg/g respectively with increase in pH from 3 to 11.

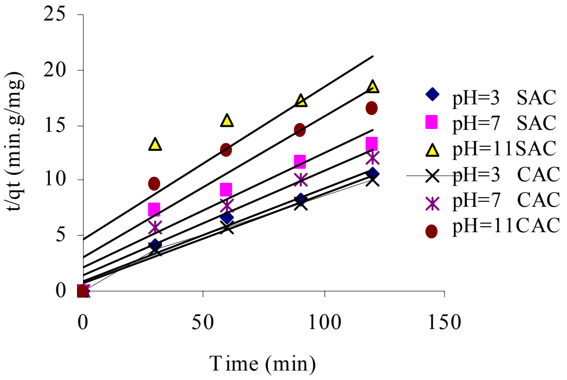

Fig. 5: Pseudo-second order sorption kinetics of reactive dye on to SAC and CAC at various pH conditions

DISCUSSION

Adsorption studies performed on the adsorbents, SAC and CAC revealed the ability of both the carbons to remove reactive dye from cotton dyeing plant effluents. The equilibrium data have been analyzed using Langmuir and Freundlich isotherms. Linear plot of log (X/M) versus log C_e showed that the adsorption obeyed a Freundlich isotherm (Fig. 1). McKay *et al.*, (1982) have shown that n value between 2 and 10 indicate beneficial adsorption. The linear plot of C_e/q_e versus C_e showed that the adsorption obeyed a Langmuir isotherm model (Fig. 2). The adsorption capacity obtained in this experiment is in agreement with the results reported in the literature. The adsorption data obeyed both Freundlich and Langmuir models exhibiting heterogeneous surface conditions and monolayer adsorption (Lee *et al.*, 1995). The favorable nature of adsorption can be expressed in terms of a dimensionless parameter R_L that is given by the equation.

$$R_L = 1/\ 1+bC_o \qquad (7)$$

Where b is the Langmuir constant (l/mg) and C_o is the initial dye concentration (McKay *et al.*, 1982). All R_L values obtained using equation (7), for dye adsorption are greater than zero and less than unity, showing favorable adsorption of dye on to sewage sludge derived activated carbon and commercial activated carbon. The equilibrium sorption capacity of the SAC anc CAC has increased with an increase in the concentration of dye in the aqueous solution.This is due to the acceleration of dye diffusion from dyeing solutions at higher concentrations (Ozacar, 2003a; Sun and Xu, 1997). It is observed that by increasing the dose of SAC and CAC, the percentage of dye removal from aqueous solution increased but adsorption density, ie the amount of dye adsorbed per unit mass decreased. It is readily understood that, the number of available adsorption sites increases by increasing the adsorbent dose and it therefore, results in the increase of percentage of dye adsorbed. The decrease in adsorption density with increase in the adsorbent dosage is mainly because of unsaturation of adsorption sites through the adsorption process (Yu *et al.*, 2003; Shukla *et al.*, 2002). Another reason may be due the interparticle interaction, such as aggregation resulting from high adsorbent dosage. Such aggregation would lead to decrease in total surface area of the adsorbent and an increase in diffusional path length. The equilibrium sorption capacity of SAC and CAC has increased by varying the pH value of dye laden aqueous solution from 11 to 3. At acidic pH the positively charged species start dominating and the surface tends to acquire positive charge, while the adsorbate species are still negatively charged. As the adsorbent surface is positively charged, the increasing electrostatic attraction between negatively charged adsorbate species and positively charged adsorbent particles would lead to increased adsorption of reactive dye (Shukla *et al.*, 2002). From Table: 9 it is found that the equilibrium sorption capacity (q_e), the rate constant (k) and the initial sorption rate (h) of SAC and CAC depends on pH of dye laden aqueous solution.

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