Adsorption of cationic dyes on cotton stalk based activated carbon from aqueous solutions: Equilibrium and kinetic studies

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Abstract : Activated carbon prepared from cotton stalk waste by chemical activation using phosphoric acid as the activating agent was used as adsorbent for the removal of Methylene blue and Rhodamine B from aqueous solutions. By batch adsorption method, the effects of various parameters such as solution pH, contact time, initial dye concentration and adsorbent dosage have been investigated. Equilibrium adsorption data were modeled using the Langmuir and Freundlich adsorption isotherms and Pseudo-first-order, Pseudo-second-order and intraparticle diffusion models were used to analyse the kinetic data. The kinetic data was found to be best represented by the pseudo-second-order kinetic model.

Keywords : Activated carbon, Cotton stalk, Adsorption, Methylene blue, Rhodamine B.

Introduction

Dyes are being widely used in broad industrial sectors such as textile manufacturing, leather tanning, cosmetics, paper and food processing. The annual production of synthetic dyes and dying stuffs are generally exceeding 700,000 tones as reported in the literature[1]. Dyes discharged into the waterbodies causes environmental damage to aquatic organisms by blocking sunlight, retarding photosynthetic activity and disturbing the re-oxygenation capacity, which creates anaerobic condition that limits aquatic plant growth[2]. Dyes can cause allergic dermatitis, skin irritation, cancer, mutation, etc. In addition, biodegradation of some of them produce aromatic amines, which are highly carcinogenic[3,4]. The continuous exposure of workers in the textile industries is linked to a higher bladder cancer risk[5].

Methylene blue (MB) is a cationic dye commonly used for coloring. It is generally used for dyeing cotton, wool, and silk. MB can cause eye burns and irritation to the skinin humans and animals and if ingested, irritation to the gastrointestinal tract, nausea, vomiting, and diarrhea[6].Rhodamine B(RhB) is widely used as a colouring paper, temporary hair colourant, dyeing cottons, wools, leather and coating for paper stock[7].

The most widely used methods for removing dyes from water include coagulation[8], electrochemical[9], oxidation[10], ozonation[11], solvent extraction[12], adsorption[1,13]and photocatalytic degradation[14] and the adsorption technique is considered to be superior to other techniques. Activated carbon is the most popular adsorbent for the removal of dyes by adsorption. Its adsorption capacity depends on its surface characteristics and the functional groups present on pore surface which are in turn depend on precursor used and method of preparation[15]. Activated carbon prepared from agricultural wastes has shown very promising prospects as

adsorbents for pollution control due to their natural abundance, renewability, cost effectiveness and eco-friendliness[7].

This study reports the preparation of activated carbon from cotton stalk waste by chemical activation by using H_3PO_4 as activating agent and its use as an adsorbent for the removal of methylene blue and rhodamine B from synthetic aqueous solutions. The effects of different parameters such as pH, adsorbent dosage, contact time and initial dye concentration on the adsorption process have been investigated. Langmuir and Freundlich models were used to fit the equilibrium isotherm data. The kinetic data were analyzed using the pseudo-first-order, pseudo-second-order and intra-particle diffusion models.

Materials and methods

Cotton Stalk (CS) wastes were collected from local agriculture area in Tranquebartaluk in Nagapattinam district, Tamilnadu, India. The CS was thoroughly washed several times with distilled water to remove the impurities and was dried in oven at 110°C for 24 h. The dried material was then ground and sieved to obtain precursors of particle sizes less than 2 mm.

Preparation of cotton stalk activated carbon (CSAC)

The powdered cotton stalk was impregnated in phosphoric acid solution in a ratio of 1:2 (weight of precursor: volume of H_3PO_4) and the mixture was stirred continuously using a magnetic stirrer at ambient temperature for 5 h. The precursors were then kept soaked in for 24 h prior to being filtered and dried in hot air oven at 110°C for 24 h. The dried mix was heated at a heating rate of 20°C min^{-1} in a muffle furnace to 600°C and after 2 h, the mix was cooled down to room temperature. The mix was then washed sequentially with 0.1M NaOH solution, hot distilled water and finally with cold distilled water to remove residual acids until washing solution becomes neutral. The washed sample was then dried in hot air oven at 110°C for 6h and cooled to room temperature to obtain the cotton stalk activated carbon.

Preparation of adsorbates

Methylene blue and Rhodamine B were purchased from Loba and SD fine chemicals,Mumbai, India respectively. Stock dye solutions of 1000 mg/L were prepared by dissolving an appropriate quantity of dyes. Working solutions at appropriate concentrations were prepared by diluting the stock solution. Distilled water was used throughout the experiments.

Adsorption studies

Adsorption experiments were carried out by batch method at room temperature (28°C ± 2). In 250 ml Erlenmeyer flasks, 100 ml of MB and RhB dye solutions of different initial concentrations (10-50 mg/L) with 0.15 g of adsorbent (CSAC) were taken and the pH of the solutions was adjusted with 0.1 M HCl and 0.1 M NaOH. The solutions were stirred continuously at room temperature for 110 min. At the end of the adsorption period, the supernatant solutions were separated by centrifugation and the residual dye concentration was measured using Shimadzu UV-1800 UV-Vis spectrophotometer at wavelength of 664nm and 554nm for MB and RhB respectively. All the experiments were duplicated under identical conditions and the percentage of dye removal was calculated using the following equation:

$$\% \, Dye \, Removal = \frac{C_o - C_t}{C_o} \times 100$$

where C_o (mg/L) and C_t (mg/L) are the initial dye concentration and at time t respectively.

Equilibrium studies

For equilibrium studies, both dye solutions with different initial concentrations (10-50 mg/L) were agitated with known amounts of adsorbent until the adsorption equilibrium was reached. The amount of dye adsorption at equilibrium, q_e (mg/g) was calculated by the following equation:

$$q_e = \frac{(C_o - C_e)V}{W}$$

where C_o and C_e (mg/L) are the liquid-phase concentrations of dye at the initial and equilibrium concentrations, respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent used (g). The equilibrium data were then fitted using two different isotherm models, namely the Langmuir and Freundlich isotherm models.

Kinetic studies

Using the same procedure stated above, at predetermined time intervals, aqueous samples were taken from the solution and the concentration of MB and RhB were measured. The amount of dye adsorbed at time t, q_t(mg/g) was calculated by following equation:

$$q_t = \frac{(C_o - C_t)V}{W}$$

where C_o and C_t(mg/L) are the liquid-phase concentrations of dyes initially and at any time t, respectively. V is the volume of the solution (L) and W is the mass of adsorbent used (g). The adsorption kinetics of dyes was investigated using pseudo-first order model, pseudo-second-order model and intra-particle diffusion models.

Results and discussion

Effect of contact time

The effect of contact time on the removal of MB and RhB on 0.15 g/L CSAC in 100 mL dye solutions of different initial concentrations (10 to 50 mg/L) at room temperature is shown in Fig.1. The removal of both the dyes was rapid initially (30-50 min) but it gradually decreased over time until the equilibrium adsorption is reached in about 110 min for almost all cases. The percentage removal is higher in the beginning due to adsorption on large surface area of CSAC available initially. The rate of dye uptake is then controlled by the rate of dye transportfrom the surface to the interior sites of the CSAC particles.
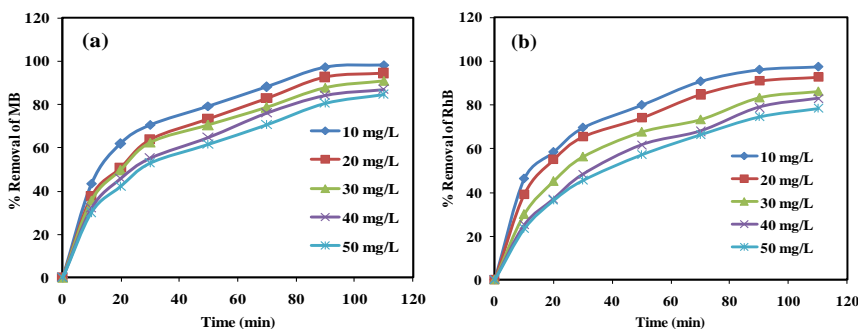

Fig.1. Effect of contact time on the adsorption of dyes (a) MB and (b) RhB.

Effect of solution pH

The effect of solution pH on dye adsorption was investigated using 100 mL of 10 mg/L MB and RhB dye solutions with 0.15 g CSAC by varying the pH from 2 to 12 and the result is presented in Fig.2. The percent removal of MB increased from 51.24 % to 98.09% for an increase in pH from 2 to 12. The maximum MB removal was attained at pH 12. Lower adsorption capacity at lower pH is mainly due to the protonation of MB in acidic medium and the presence of excess H^+ ions that can compete with the cationic dye molecules for adsorption sites[16]. For the RhB, the maximum percentage removal occured at pH 3 and it decreased gradually above pH 3. Hence 12 and 3 were chosen as optimum pH for the removal of MB and RhB respectively.
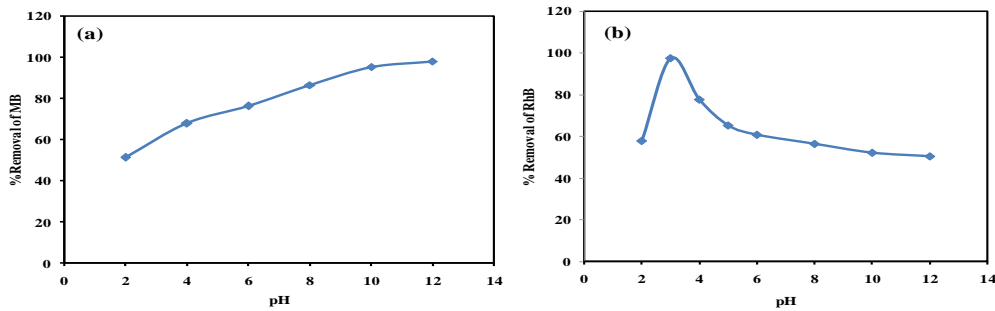
Fig.2. Effect of pH on % removal of (a) MB and (b) RhB.

Effect of adsorbent dosage

The effect of adsorbent dosage on removal of MB and RhB was studied by varying the CSAC dosage from 0.05 to 0.25 g/L. The experiments were carried out at fixed dye concentration of 10 mg/L in 100 mL of MB and RhB solutions of pH 12 and 3 respectively, at room temperature. Results depicted in Fig.3 showthatthe percentage removal of both dyes increasedwith increasingadsorbent dosage upto 0.15g but remained unchanged with further increase in adsorbent dosage (Fig.3). At equilibrium time, the percentage removal of MB increased from 67.85 to 98.09 and of RhB from 64.48 to 97.33 for an increase of dosage from 0.05 to 0.25 g. The rapid increase in adsorption with the increasing adsorbent dosagecan be attributed to the greater surface area and initial availability of more adsorption sites.
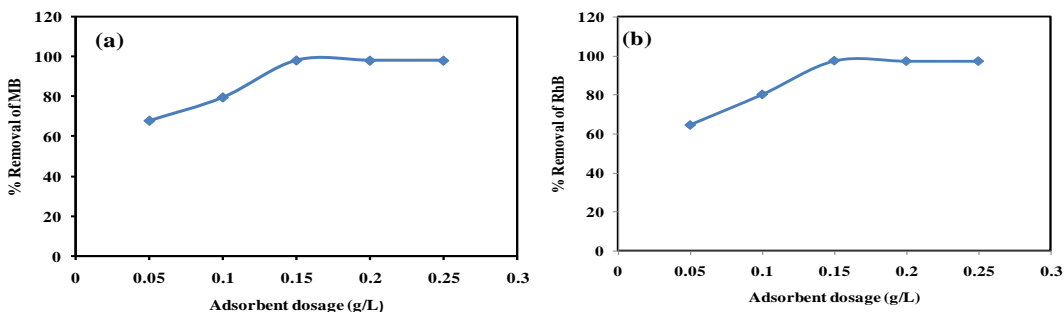

Fig.3. Effect of adsorbent dosage on the % removal of (a) MB and (b) RhB.

Effect of initial dye concentration

Experiments were carried out at optimum adsorbent dose 0.15g in100mL dye solutions of different concentrations (10, 20, 30,40, 50 mg/L). The results reported in Fig.4reveal that the percent removal of the MB and RhB dyes decreased from 98.09 and 97.33 to 84.63 and 78.23 respectively. This behavior may be attributed to the saturation of available active sites above a certain concentration of dyes.The initial dye concentration provides an important driving force to overcome the mass transfer resistance of dyes between the aqueous solution and solid phase (CSAC) surface.
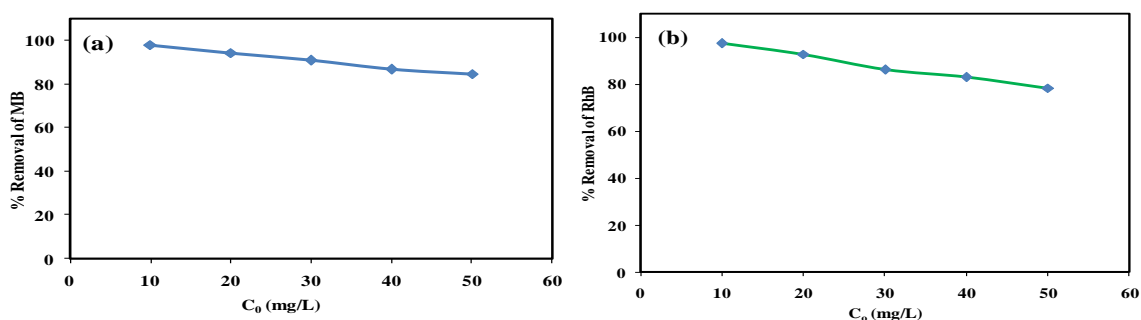

Fig.4. Effect of initial dye concentration on % removal of dyes (a) MB and (b) RhB.

Adsorption isotherms

Adsorption isotherms were obtained from the results (Fig.5) of batch adsorption experiments at room temperature at optimum adsorbent dose (0.15 g/L), optimum pH (12 for MB and 3 for RhB) for 110 minutes, varying dye concentration from 10 to 50 mg/L. The amount of dye adsorbed at different time intervals were determined and these data were fitted to equilibrium isotherms and kinetic models.
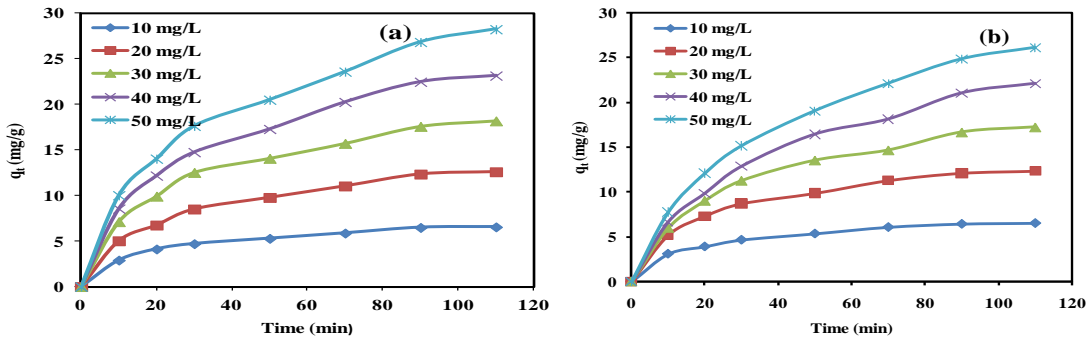

Fig.5. Adsorption capacity Vs contact time at different initial concentrations of (a) MB and (b) RhB.

Langmuir isotherm

Langmuir isotherm is based on the assumption of monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface[17]. The linear form of Langmuir isotherm equation is given as:

$$\frac{C_e}{q_e} = \frac{1}{Q_o K_L} + \frac{1}{Q_o} C_e$$

where C_e (mg/L) is the equilibrium concentration of the dye, q_e (mg/g) is the amount of dye adsorbed per unit mass of adsorbent, Q_o and K_L are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. When C_e/q_e is plotted against C_e, a straight line with slope of $1/Q_o$ and intercept of $1/Q_o K_L$ is obtained (Fig.6), indicating that the adsorption of MB and RhB on CSAC follows the Langmuir isotherm. The Langmuir constants K_L and Q_o were calculated from this isotherm model and their values are listed in Table.1. Higher correlation coefficient (R^2) indicates that the equilibrium adsorption data fits this model well for both dyes.
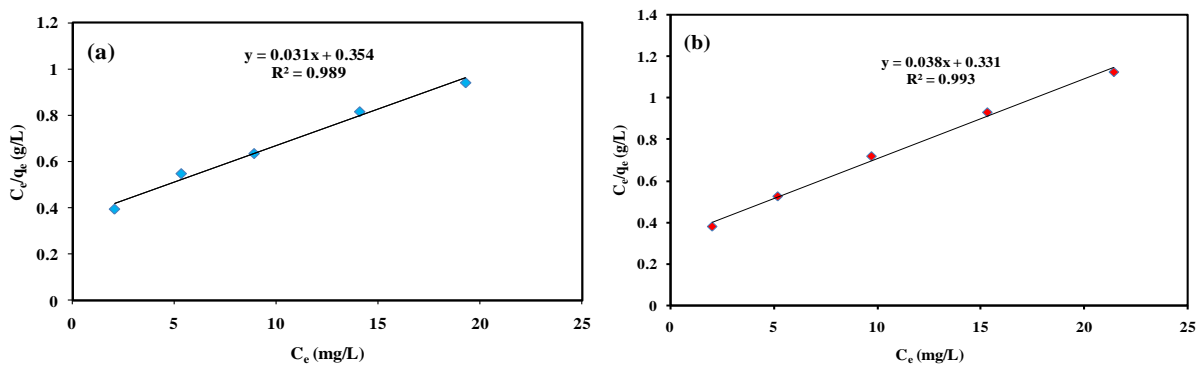

Fig.6. Langmuir isotherm model for the adsorption of dyes (a) MB and (b) RhB.

Table.1. Langmuir and Freundlich isotherms parameters for the adsorption of MB and RhB onto CSAC.

Dyes	Langmuir isotherm			Freundlich isotherm		
	Q_o (mg/g)	K_L (L/mg)	R^2	K_F (mg/g)	1/n (L/mg)	R^2
MB	32.2580	0.0875	0.989	3.4593	1.6339	0.994
RhB	26.3157	0.1148	0.993	3.7670	1.8484	0.993

The essential characteristic of Langmuir equation can be expressed in terms of a dimensionless separation factor R_L[18], which is defined as

$$R_L = \frac{1}{(1 + K_L C_o)}$$

Where K_L is the Langmuir constant and C_o(mg/L) is the initial dye concentration. The value of R_L indicates the type of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$).

The values of separation factor R_L for MB and RhB adsorption onto CSAC in different initial concentrations are reported in table 2. Respective plots are shown in Fig.7. R_L values obtained are between 0 and 1, showing that the adsorption of both dyes is favorable. For both dyes, as the initial concentration increased from 10 to 50 mg/L, the R_L values were found to decrease, indicating that the adsorption was more favorable.

Table 2. Separation factor R_L at different initial concentrations of MB and RhB.

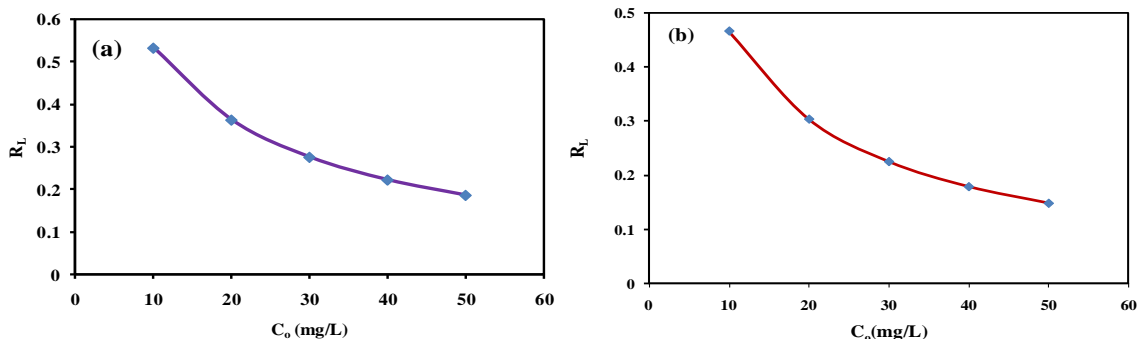| Initial dye concentrations (mg/L)	R_L for MB	R_L for RhB
10	0.5333	0.4655
20	0.3636	0.3033
30	0.2758	0.2250
40	0.2222	0.1788
50	0.1860	0.1483

Fig.7.Effect of initial dye concentration on separation factor R_L for (a) MB and (b) RhB.

Freundlich isotherm

The Freundlich isotherm assumes heterogeneous surface energies. The well-known logarithmic form of the Freundlich isotherm[19] is given by the following equation:

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e$$

where C_e is the equilibrium concentration of the adsorbate (mg/l), q_e is the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g), K_F and n are Freundlich constants with n giving an indication of how favourable is the adsorption process and K_F (mg/g (l/mg)$^{1/n}$) is the adsorption capacity of the adsorbent.Freundlich isotherms for both dyes were plotted and are shown in Fig.8. The $1/n$ and K_F values were determined from the slope and intercept and are presented in Table 1.
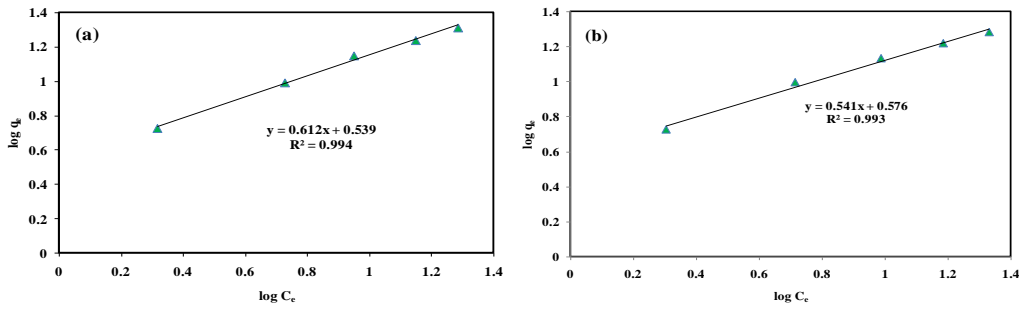
Fig.8. Freundlich isotherms for the adsorption of dyes (a) MB and (b) RhB.

Then values between 1 and 10 represent that the dye is favourably adsorbed onto the adsorbent[20]. The Langmuir and Freundlich isotherms for both dyes were found to be linear over the whole range of concentration. Both Langmuir and Freundlich isotherms models describes the nature of adsorption with high correlation coefficient.

Adsorption kinetics

The kinetics of dye adsorption was studied for different initial concentrations of the both dye solutions ranging from 10 to 50 mg/L.Three kinetic models such as pseudo-first-order, pseudo-second-order and intra-particle diffusion were applied to the experimental data to analyze the adsorption kinetics.

Pseudo first-order kinetic model

The Pseudo first-order kinetic model was proposed by Lagergren[21].The integrated form of the model is

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

where, q_e is the amount of dye adsorbed at equilibrium (mg/g), q_t is the amount of dye adsorbed at time t (mg/g), K_1 is the first-order rate constant (min^{-1}) and t is the time (min). A plot of log (q_e-q_t) and t, will give a linear trace provided the adsorption follows first order kinetics. The values of k_1 and q_e can be determined from the slope and intercept.

The pseudo first-order kinetic plots at various initial dye concentrations of both dyes are shown in Fig.9 and the results are reported in Table 3. The calculated equilibrium adsorption capacity $q_{e(cal)}$ values are too low compared with the experimental q_e values and the correlation coefficient R^2 are relatively low for most adsorption data indicating that the adsorption of MB and RhB onto CSAC is not a pseudo first-order reaction.
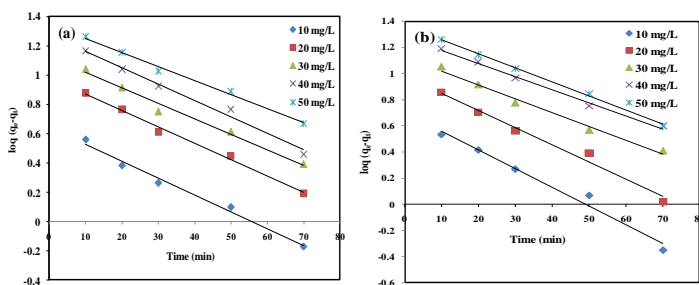

Fig.9. Pseudofirst-order kinetic model for adsorption of dyes (a) MB and (b) RhB

Pseudo second-order model

The pseudo second-order model model[22] is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

where k_2 (g mg^{-1}min^{-1}) denotes the pseudo second-order rate constant. The values of k_2 and q_e were calculated from the slope and intercept of plots of t/q_t versus t. The initial adsorption rate denoted as h (mg g^{-1}min^{-1}) is given as:

$h = k_2 q_e^2$

The k_2, q_e and h are reported in Table 3.

The pseudo second-order kinetic plots at various initial dye concentrations of both dyes are shown in Fig.10 and the results are reported in Table 3. The correlation coefficient R^2 for pseudo-second-order kinetic model was higher than that for pseudo first-order kinetic model and the calculated q_e values are much closer to the experimental data. Thus, adsorption process obeyed pseudo second-order model for both dyes.
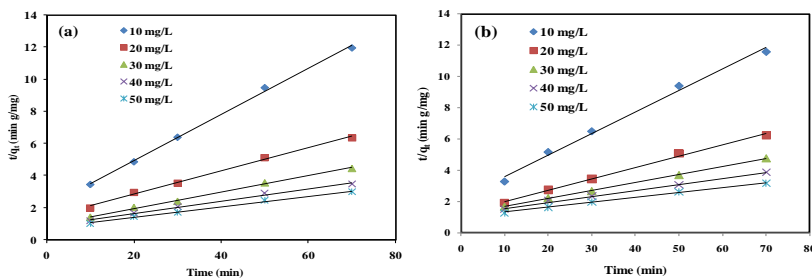

Fig.10.Pseudo-second-order kinetic model for adsorption of dyes (a) MB and (b) RhB onto CSAC

Intra-particle diffusion model

Intra-particle diffusion model based on the theory proposed by Weber and Morris[23] was tested to identify the diffusion mechanism. According to this theory:

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

Wherek_p, the intraparticle diffusion rate constant (mg/gmin$^{1/2}$),can be determined from the slope of the linear plot of $q_t$versus $t^{(1/2)}$(Fig.11). The values of k_p and correlation coefficient R^2 for intra-particle diffusion model are reported in Table 3. The R^2 values were low compared to those obtained from pseudo-first order and pseudo-second order kinetic models.
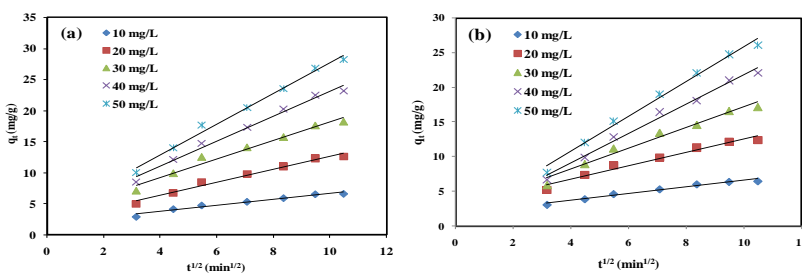

Fig.11. Intra-particle diffusion model for adsorption of (a) MB and (b) RhB at different initial concentrations.

The plots of q_t versus $t^{1/2}$ are multilinear and there are three different portions, indicating the different stages in adsorption. The first, sharper portion represents the external mass transfer. The second portion is the gradual adsorption stage where intra-particle diffusion is rate-limiting. The third portion is the final equilibrium stage where intra-particle diffusion starts to slow down due to the extremely low adsorbate concentration left in the solutions[24]. The linear plots at each concentration did not passthrough the origin. This indicates that the intra-particle diffusion wasnot only a rate controlling step.

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Table 3.Pseudo-first-order, Pseudo-second-order and Intra-particle diffusion model parameters for adsorption of MB and RhB dyes onto CSAC at different initial concentrations.

C_o (mg/L)	q_e, exp (mg/g)	pseudo-first-order model			pseudo-second-order model				Intra-particle diffusion model	
		q_e, cal (mg/g)	k_1 (min^{-1})	R^2	q_e, cal (mg/g)	k_2 (g/mg min)	h	R^2	k_p (mg/g min$^{1/2}$)	R^2
MB										
10	6.5397	4.3853	0.0253	0.986	6.9930	9.9505	0.4866	0.998	0.483	0.963
20	12.5820	9.5940	0.0253	0.992	13.8888	3.7050	0.7147	0.995	1.045	0.979
30	18.1587	13.2129	0.0230	0.984	19.6078	2.7846	1.0706	0.997	1.479	0.974
40	23.1217	18.7931	0.0253	0.986	26.3157	1.6848	1.1668	0.992	2.005	0.986
50	28.2116	22.0292	0.0207	0.992	31.25	1.4104	1.3774	0.994	2.468	0.990
RhB										
10	6.4889	5.0118	0.0322	0.979	7.2992	8.4987	0.4528	0.992	0.480	0.971
20	12.3555	9.5279	0.0299	0.982	13.8888	4.0689	0.7849	0.995	0.966	0.966
30	17.2222	13.3967	0.0230	0.987	19.6078	2.2519	0.8658	0.999	1.504	0.973
40	22.10	18.9670	0.0230	0.993	26.3157	1.2278	0.8503	0.995	2.119	0.988
50	26.0777	23.3345	0.0230	0.997	32.2580	0.0094	0.9832	0.997	2.510	0.990

Conclusions

Activated carbon prepared from cotton stalk was used as adsorbent for the removal of Methylene blue and Rhodamine B from aqueous solutions. The effects of experimental parameters such as initial pH, contact time, initial dye concentration and adsorbent dosage on adsorption were studied. The optimum pH value was determined to be 12 for MB and 3 for RhB. Equilibrium adsorption studies revealed that both Langmuir and Freundlich isotherm models described the nature of adsorption with good correlation coefficient. The kinetic studies suggested that the adsorption of both dyes followed the pseudo-second-order model.

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