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Adsorption of Rhodamine-B Dye by Raw and Activated Bael Leaves: Isotherms, Kinetics and Thermodynamics Perspectives

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The agricultural waste and their derived products to remove dyes from wastewater have attracted ample attention due to its availability and economic advantages with high removal efficiency. In this study, raw bael leaves and their prepared carbon were used to remove Rhodamine-B (RhB) dye present in wastewater. The effects of different factors, including concentration (50-100 mg/L), contact time (20-140 min), adsorbent dosage (5-50 mg), pH range (1-10) and temperature (20-60 ºC) were investigated. The maximum percentage removal of RhB dye were found to be 77.4 and 89.9, respectively using raw bael leaves (RBL) and activated carbon of bael leaves (ACBL) with optimum conditions. Different characterization techniques used to characterize the adsorbents (RBL and ACBL) *via* SEM, FTIR, XRD and BET. Adsorption process obeyed Langmuir isotherm model for both cases *viz.* adsorption of RhB on RBL and adsorption of RhB on ACBL. The maximum adsorption capacities 86.89 mg/g and 113.82 mg/g were found for RBL and ACBL, respectively. The kinetics adsorption study for adsorption of RhB on RBL and ACBL had a great correlation with pseudo-second order model and confirmed the processes are chemisorption. Therefore, the RBL and ACBL adsorbents could be encouraging adsorbents for the removal of dyes.

Keywords: Raw bael leaves, Bael leaves, Activated carbon, Rhodamine-B dye, Adsorption.

INTRODUCTION

The effluent discharged from the different type of industries like textile industries, pharmaceutical industries, agriculture industries food industries, plastic and paper industries consist of different dyes [1]. The colour of these dyes put the adverse effect on the environment. Rhodamine-B dye is a cationic dye used pulp and paper, textile, leather industries, *etc.* causes the hepatic and lung tumor [2]. Therefore, various techniques *viz.* reverse osmosis, chemical oxidation, photodegradation, electro-coagulation, electro floatation and adsorption are used for the removal of these colours from effluents [3]. Among them, adsorption is the best method for wastewater treatment as low initial cost, easy in operation and design is simple. Many adsorbents used for dye removal as agro based industrial byproduct fibres and nanocomposites but most of them are not cost effective [4]. Activated carbon as an adsorbent gives best result for dye removal. However, its cost increased as it is regenerated and reused. Many researchers reviewed a properties of lowcost adsorbents from different kinds of agriculture wastes [5-8]. Much of these agriculture solid wastes are used to prepare activated carbon as rice husk, maize cobs, tamarind fruit shell, cashew nut shell, sunflower seed hull, *etc.* [9-15].

In present work, the activated carbon prepared from the powdered raw bael leaves is applied as an adsorbent in treating the textile wastewater containing Rhodamine-B dye. Moreover, the adsorption capacity of the prepared activated carbon towards Rhodamine-B dye is high when compared to raw bael leaves.

EXPERIMENTAL

Analytical grade chemical and solvents were used throughout the experiments and procured from Merck, India, whereas Rhodamine-B (RhB) dye was purchased from LobaChemie, India.

Preparation of adsorbents

Preparation of raw bael leaves powder (RBL): Bael leaves were collected from the campus of Deenbandhu Chhotu Ram University of Science and Technology, Murthal, India and washed with double distilled water to remove the foreign impurities and then dried in oven for 24 h at 60 ºC. The dried bael leaves were grind with home grinder machine (Make: Bajaj, India) to make fine powder. Powdered bael leaves were sieved into particles of 300 µ using laboratory sieve shaker and then stored for further use.

Preparation of activated carbon from bael leaves (ACBL): Raw bael leaf powder (300 µ size) was soaked in conc. H_2SO_4 acid in a ratio of 1:1 (w/v) for 48 h. Then, the slurry was heated to 600 ºC in a muffle furnace for 2 h and then washed twice with distilled water. The carbonized matter was dried in an oven at 105 ºC and stored.

Characterization: The adsorbent morphology was examined with the help of scanning electron microscopy (SEM, JEOL-6380, Japan). The infrared spectra were captured between 4000 and 400 cm^{-1} using Fourier transform infrared spectroscopy (Perkin-Elmer Frontier BSEN60825, USA). The X-Ray diffraction was used to study the adsorbents diffraction patterns (XRD, Rigaku Miniflex 600, Japan). Surface area and particle size of adsorbents were measured using a Brunauer-Emmett-Teller (BET, Quanta Chrome Novae-2200, USA).

Batch studies: The pH of solution was varied from pH 1 to 10 by adding of 0.1 N HCl and 0.1 N NaOH to the solution. The experiments were investigated for the time range 20 to 140 min with time interval (20 min), initial concentration range between 50 to 100 mg/L with concentration gap 10 mg/L, adsorbents dosage range 5-50 mg and temperature variation 20 to 60 ºC with temperature interval of 10 ºC. All solutions were shaking at 120 rpm *via* orbital shaker. Dye concentrations suspended in the solution after the adsorption of reaction were determined by UV spectrophotometer (Rigol-3660, India). The percentage removal of dye was calculated using eqn. 1:

$$\text{Removal } (\%) = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

whereas C_o and C_e is the initial concentration and equilibrium concentration of RhB dye, respectively.

RESULTS AND DISCUSSION

FTIR studies: Several characteristics band were observed in the FTIR spectra of RBL and ACBL (Fig. 1). A peak at 3849 $cm^{-1}$was observed in RBL is subsequent to H-bonded O-H stretching, while the absorbance peaks at 2917 and 2407 cm^{-1} were due to the C-H stretching of methyl group. The absorbance band found at ~1646 cm^{-1} revealed the C=C stretches of amine group and the peak at 1038 cm^{-1} is due to the -C-O-C strong stretching group. A band in the range 850-550 cm^{-1} confirmed the presence the C-Cl stretching group. When bael leaves were carbonized and used to make activated carbon (ACBL), it was found that some peaks were shifted, while others disappeared. A peak at 1646 cm^{-1} was disappeared after the carbonization of bael leaves. Similar results were also reported by various researchers [16-20].

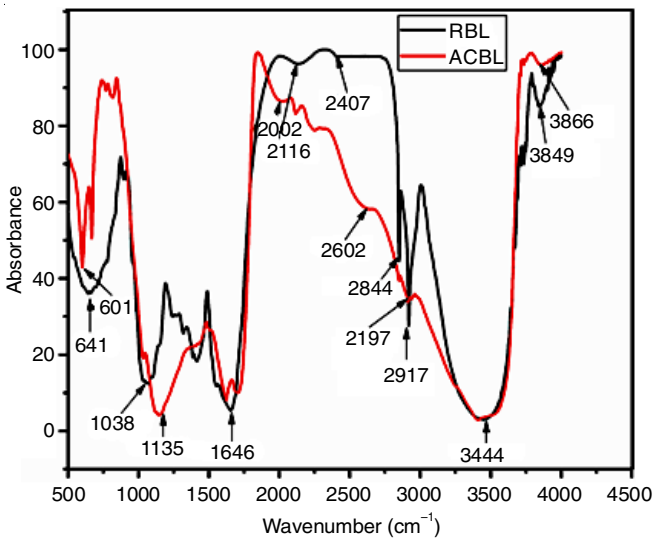

Fig. 1. FTIR spectra of RBL and ACBL

XRD studies: Fig. 2 represents the XRD spectrum of RBL and ACBL adsorbents. No characteristic peaks between 20º and 70º were observed in either adsorbent (RBL or ACBL), making their amorphous nature unassailable.This is happened, since after the preparation of activated carbon of adsorbents, the adsorbents contain quite crystalline and amorphous type resulting from the arrangement of the glycosidic chain, which is hindered by hydrogen bonding in the crystalline nature while such hydrogen bonding in the amorphous nature is insufficient [21,22].

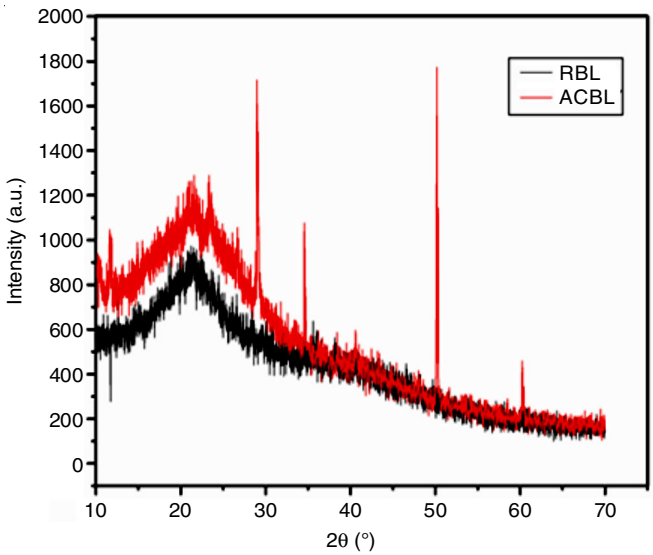

Fig. 2. XRD spectra of RBL and ACBL

Morphology studies: The surface morphology and microstructure of RBL and ACBL adsorbents were investigated using SEM analysis. The consequential surface transformation was detected in the activation of natural biomass. Limited number of pits, rough, tightly bound surface with macropores was replicated in RBL, while heterogeneous, micropores and porous structure, with different sizes was developed after the carbonization of RBL (Fig. 3).

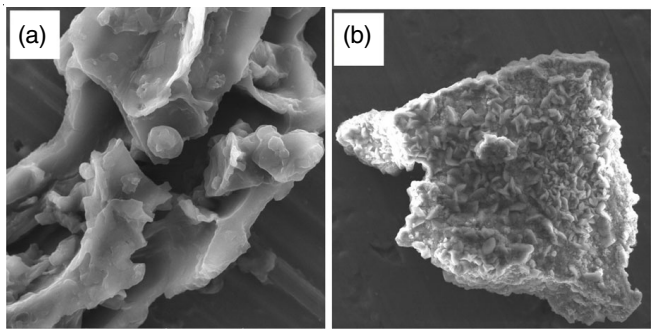

Fig. 3. (a) SEM images of RBL (b) ACBL

Adsorption behaviours

Effects of initial RhB dye concentration: Percentage removal of RhB dye was determined for both adsorbents (RBL and ACBL) in the concentration range of 50 to 100 mg/L. For both adsorbents, the percentage of RhB dye removal decreased as dye concentration increased. The percentage elimination decreases from 77.4 to 63.5 in case of RBL and 89.9 to 81.7 in case of ACBL. The maximum removal was found to be 77.4% and 89.9% for RBL and ACBL, respectively at 50 mg/L RhB concentration (Fig. 4a). This is because the ratio of available active sites of both adsorbents to the number of dye molecules is enormous at low dye concentrations, limiting the proportion of removal of the dye negligible. Furthermore, at higher concentrations of RhB dye, the ratio of available active sites of both adsorbent and dye concentration decreased [23,24]. As a result, the removal percentage of RhB dye decreases as dye concentration increases.

Effect of contact time: Adsorption behaviour based on contact time of RhB dye *via* RBL and ACBL adsorbents was conducted in the time range of 20 to 140 min (Fig. 4b). Other conditions were temperature (30 ºC), concentration (50 mg/L), adsorbent dose (50 mg) and pH 7 for RBL and 5 for ACBL, respectively. At initial stage rate of adsorption of RhB dye fast due to the abundant available active sites of both adsorbents surface. As the contact time increases, the adsorption rate decreases, and eventually both lines become constant, indicating equilibrium has been reached due to the saturation of all potentially active sites [25-27]. The removal percentage of 77.4 for RBL and 89.9 for ACBL at equilibrium point (120 min) were obtained.

Effect of adsorbents dosage: Effect of both adsorbents dosage (RBL and ACBL) used to eliminate the RhB dye was carried out *via* varying adsorbents mass in the range between 5 mg to 50 mg with contact time (120 min), temperature (30 ºC), initial concentration (50 mg/L) and pH = 7 for RBL and pH = 5 for ACBL, respectively. As observed from Fig. 4c, it was found that the adsorption increased along with the dosage of the adsorbents due to an increase in the number of accessible active sites [26,28].

Effect of pH: The effect of pH on RhB dye adsorption was investigated from in the range of 1 to 10. The pH of the dye solution was kept constant by using 0.1 N NaOH and 0.1 N HCl. At time 120 min, mass 50 mg of both adsorbents (RBL and ACBL) were contacted with 50 mL of 50 mg/L RhB dye solution. The maximum % removal of RhB dye using RBL = 77.4 and ACBL = 89.9 were achieved at solution pH 7 and 5 respectively. At pH = 1 to 4, the % removal decreased due to presence of high concentration of hydrogen ions in the solution and displace on active sites (Fig. 4d). Specifically, the RhB dye cations ions would be repelled by the hydrogen ions already present in the active sites of the two adsorbents, causing a decrease in adsorption efficiency. However, it was found that upon increasing the pH solution from 1 to 7 results in the

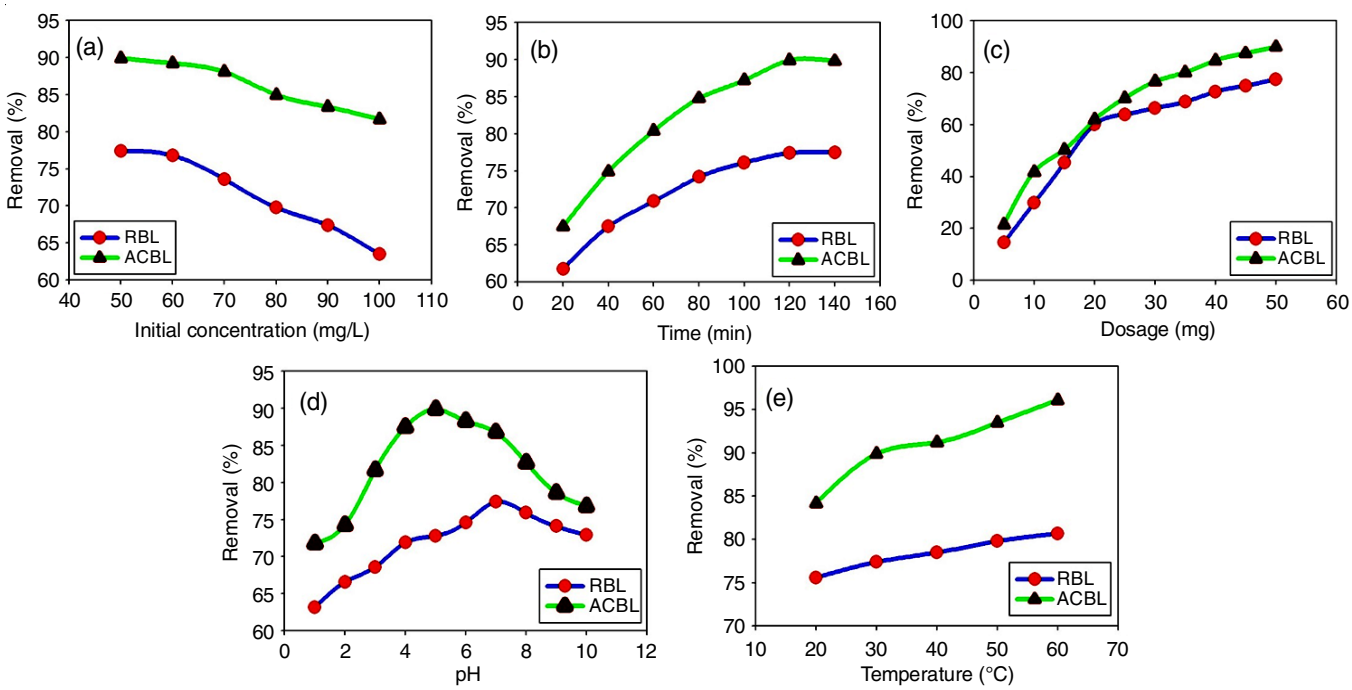

Fig. 4. Adsorption behaviour of different parameters (a) initial concentration, (b) contact time, (c) dosage of RBL and ACBL, (d) pH and (e) temperature

increased dye adsorption efficiency since the negative charges on the surface of the adsorbents were intensified, resulting in a stronger attraction of the RhB dye [29,30]. Therefore, pH = 7 for RhB dye adsorption on RBL and pH = 5 on ACBL were determined to be optimal.

Effect of temperature: Another factor which is significant in the adsorption process is the dye solution temperature, which determines how much energy is available for the process. The experiments were conducted at the different temperatures ranging from 20 ºC to 60 ºC, while keeping the other parameters *viz*. dosage of absorbent (50 mg), RhB dye concentration (50 mg/L) and time (120 min) constant. As the temperature of solutions increases, the number of available actives sites increase because of the reduction in the thickness of the boundary layer around the adsorbent (Fig. 4e) and hence these phenomena increase the ability of the adsorbents to increase the percentage removal of contaminant pollutants [16,29].

Equilibrium studies: The adsorption isotherm models describe the relation between the concentrations of dissolved dye ions and the concentration of adsorbed ions at the specified conditions. Adsorption mechanism can be better understood *via* various isotherm models such as Langmuir, Freundlich, Temkin and Dubinin Radushkevich (D-R) models. These isotherms can be represented in the form of the following equations.

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (2)$$

$$R_L = \frac{1}{1 + K_L C_o} \quad (3)$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

$$q_e = B \ln A_T + B \ln C_e \quad (5)$$

$$\ln q_e = \ln q_m - (2\beta\varepsilon^2) \quad (6)$$

$$\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right) \quad (7)$$

$$E = \frac{1}{\sqrt{-2\beta}} \quad (8)$$

where C_o = Initial RhB dye concentration (mg/L); C_e = rquilibrium RhB dye concentration (mg/L); K_L = Langmuir constant (L/mg); q_m = maximum adsorption capacity (mg/g); q_e = equilibrium adsorption capacity (mg/g); K_f = Freundlich constant; n = adsorption intensity; B = heat of the adsorption; A_T = binding energy at equilibrium (L/mol); β = D-R constant (mol^2/KJ^2); E = mean free adsorption energy; ε = Polyani potential (J/mol).

The analysis and representation of the adsorption isotherm models, including the Langmuir, Freundlich, Temkin and D-R isotherms, are shown in Fig. 5a-d, respectively. Table-1 displays the parameters and correlation coefficients for each of the four adsorption isotherms: Langmuir, Freundlich, Temkin and D-R isotherm. The correlation coefficient of the studied isotherms was found to be decreasing order of 0.99403 > 0.990473 > 0.978241 > 0.96259 (Langmuir > Temkin > Freundlich > D-R isotherms model) for adsorption of RhB on ACBL, while 0.99544 > 0.98307 > 0.9821 > 0.96171 (Langmuir > Temkin > D-R > Freundlich isotherms model) in case of RhB adsorption on RBL. The highest correlation coefficients of Langmuir isotherm were observed in both the adsorbents, hence Langmuir model best fitted for adsorption on both the adsorbents [23]. In present study, the calculated separation factors of 0.2622 and 0.13081 were found for RBL and ACBL respectively, in the range 0-1, which favours the adsorption in both cases, while the monolayer maximum adsorption capacities of 113.82 and 86.89 mg/g for adsorption of RhB on amorphous surface of ACBL and RBL, respectively were found. Moreover, the value of n > 1 favours adsorption of RhB dye for both adsorbents (eqn. 4). The numerical value of B for ACBL = 27.1579; for RBL = 20.5482 and A_T for ACBL = 1.07900; for RBL = 0.6331 were examined using slope and intercept from eqn. 5. These calculated results confirmed the electrostatics interaction between both adsorbents and RhB dye molecules and pore heterogeneity of adsorbents [31]. The D-R constant (K_{id}) ACBL = 1.47994×10^{-6} and RBL = 1.15149×10^{-5} and mean free energy were (E) ACBL = 0.581258 and RBL = 0.29649 were calculated using slope of eqns. 6 and 8. The numerical value of mean free adsorption energy (E) was less than 8 kJ/mol for adsorption of RhB dye on ACBL and RBL, which indicated the physical adsorption [32].

TABLE-1
CALCULATED PARAMETERS OF DIFFERENT ISOTHERMS FOR ADSORPTION OF RhB DYE ON RBL AND ACBL

Isotherms	Parameters	Adsorbents	
		RBL	ACBL
Langmuir isotherm	q_m (mg/g)	86.89	113.82
	K_L (l/mg)	0.07627	0.13289
	R_L	0.2622	0.13081
	R^2	0.99544	0.99403
Freundlich isotherm	n	2.4728	2.28196
	K_f	3.2710	3.70594
	R^2	0.96171	0.978241
Temkin isotherm	A_T (L/mg)	0.6331	1.07900
	b_T		
	B	20.5482	27.1579
	R^2	0.98307	0.990473
D-R isotherm	q_s (mg/g)	11.3455	11.9297
	K_{id} (mol^2/kJ^2)	1.15149×10^{-5}	1.47994×10^{-6}
	E (kJ/mol)	0.29649	0.581258
	R^2	0.9821	0.96259

Adsorption kinetics models: The mechanism and adsorption kinetics were commonly investigated based on these kinetics models such as pseudo-first order, pseudo-second order and intraparticle diffusion model. These kinetics models can be represented in the form of eqns. 9 and 10. Moreover, Weber and Morris proposed the linear form of intraparticle diffusion models by eqn. 11:

$$\ln(q_e - q_t) = \ln q_e - \frac{K_1}{2.303} t \quad (9)$$

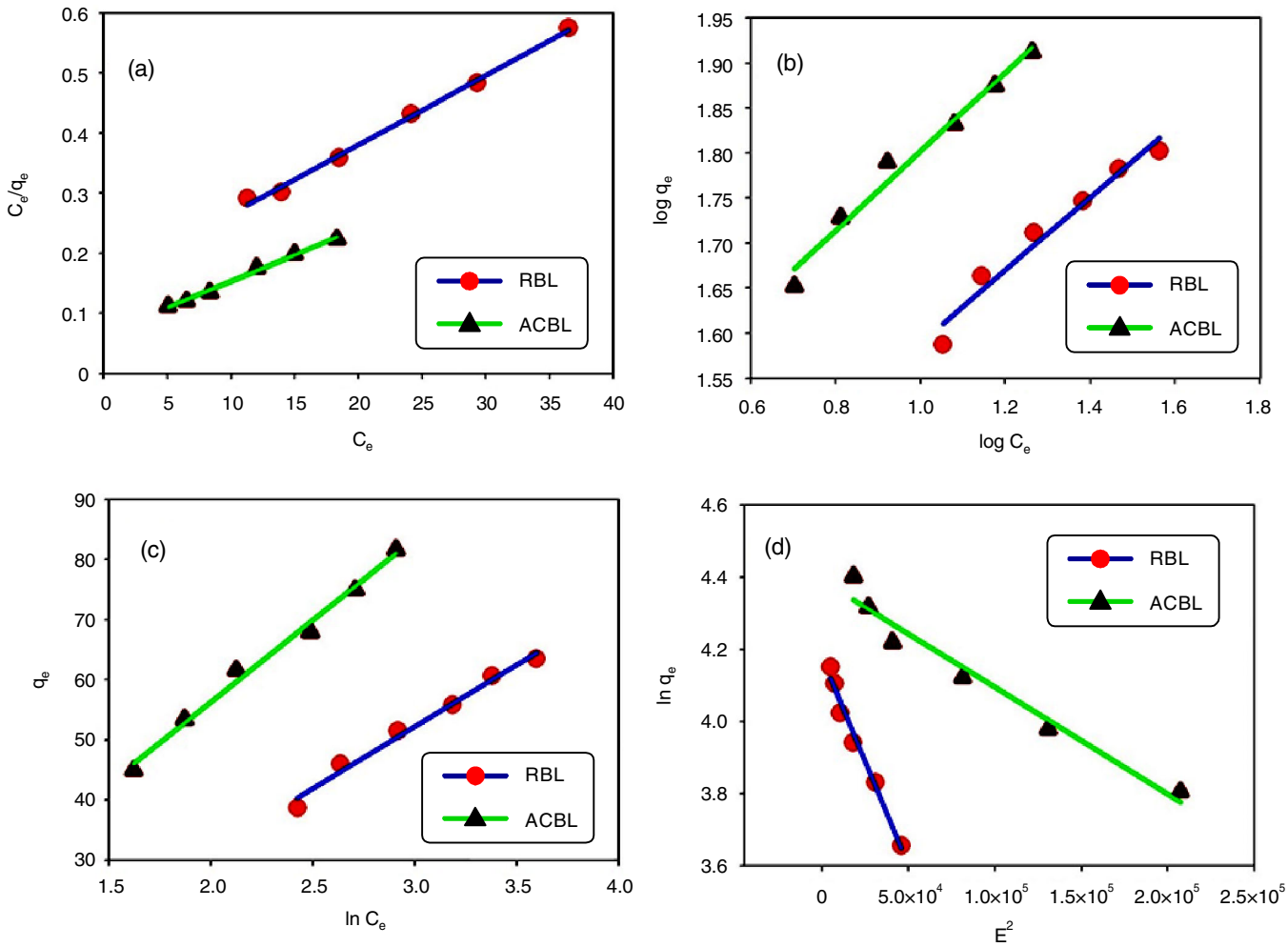

Fig. 5. Isotherms models of RhB dye adsorption on RBL and ACBL adsorbents (a) Langmuir isotherm (b) Freundlich isotherm (c) Temkin isotherm and (d) D-R isotherm

$$\frac{1}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (10)$$

$$q_e = K_{id} t^{0.5} + C \quad (11)$$

where K_1 = pseudo-first order rate constant (min^{-1}); q_e = equilibrium adsorption capacity (mg/g); q_t = adsorption capacity at given time (mg/g); K_2 = pseudo-second order rate constant (g mg^{-1} $min^{-0.5}$); t = given time, min; and C = intercept.

In present study, the adsorption kinetics for adsorption of RhB dye on RBL and ACBL adsorbents were examined and obtained results data were plotted *via* pseudo-first order (Fig. 6a), pseudo-second order (Fig. 6b) and intraparticle diffusion (Fig. 6c). The K_1 (0.080512 = RBL, 0.061149 = ACBL) and q_e (RBL = 6.60455, ACBL = 8.34673) were obtained by slope and intercept from eqn. 9. The numerical value of pseudo-second order rate constant was found to be 0.0076647 for adsorption of RhB in case RBL adsorbent and 0.004857 in case of ACBL adsorbent. The value of adsorption capacity at given time for pseudo-second order were 16.39378 = RBL and 19.3429 = ACBL adsorbent. In this study, the value of correlation coefficient of pseudo-second order is highest than other two kinetics models for adsorption of RhB on RBL and ACBL as shown in Table-2. As a result, the best kinetic model fit in the present investigation was pseudo-second order, revealing that chemical adsorption governed RhB adsorption on RBL and ACBL (sharing of electrons between RhB dye-RBL and RhB dye-ACBL) [33,34]. Furthermore, if the adsorption favour mechanism of intraparticle models, the plots between q_t *versus* $t^{0.5}$ arise straight line with passes through origin, then the rate limiting step only the reason of intraparticle diffusion, but it not occurred then other mechanism with intraparticle diffusion involved [35,36].

Thermodynamics study: Based on temperature study, the adsorption process can exist either endothermic or exothermic in nature. Basically, it depends on the nature of adsorbent and adsorbates molecules. The thermodynamics properties such as standard Gibb's free energy change (ΔG^o), standard change in enthalpy (ΔH^o) and standard change in entropy (ΔS^o) for RhB adsorption on RBL and ACBL adsorbents were investigated (293 K to 333 K) using eqns. 12-13:

$$\Delta G^o = RT \ln K_c \quad (12)$$

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (13)$$

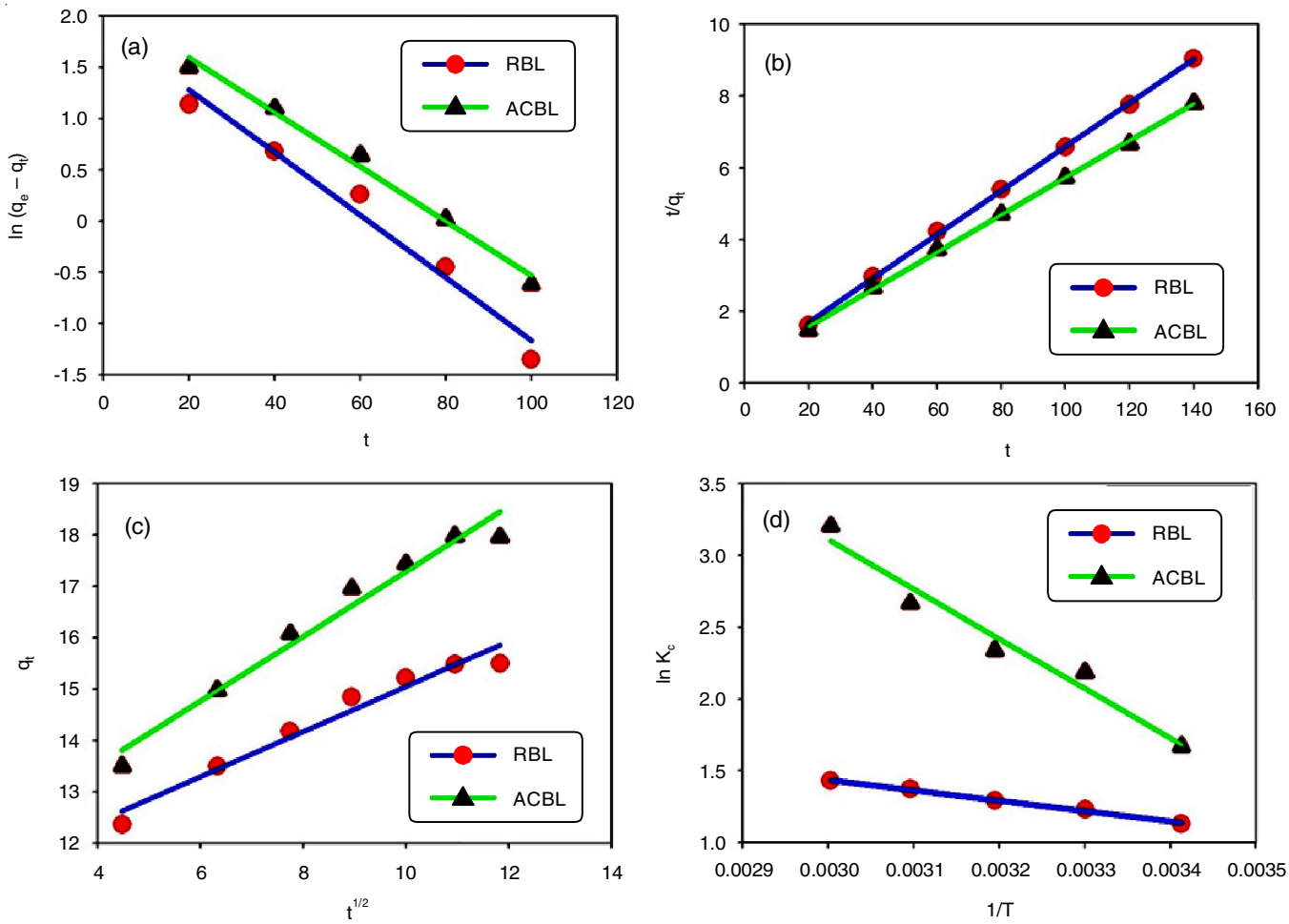

Fig. 6. (a) Pseudo-first order kinetic model, (b) Pseudo-second order model, (c) intraparticle diffusion model and (d) van't Hoff for RhB dye adsorption on RBL and ACBL adsorbents

TABLE-2
SUMMARY OF CALCULATED PARAMETERS OF KINETICS FOR ADSORPTION OF RhB ON RBL AND ACBL

Kinetics	Parameters	Adsorbents	
		RBL	ACBL
Pseudo-first order	q_e (mg/g)	6.60455	8.34673
	K_1 (min^{-1})	0.080512	0.061149
	R^2	0.972024	0.989484
Pseudo-second order	q_e (mg/g)	16.39378	19.3429
	K_2 (min^{-1})	0.0076647	0.004857
	R^2	0.999516	0.999095
Intraparticle diffusion	K_{id} (g/mg $min^{0.5}$)	0.440123	0.631125
	C (mg/g)	10.6503	10.98
	R^2	0.962868	0.969394

TABLE-3
PARAMETERS OF THERMODYNAMIC PROPERTIES

Parameters	Adsorbents	
	RBL	ACBL
ΔH° (kJ/mol)	6.0355	28.6595
ΔS° (J/mol)	30.0664	111.888
R^2	0.99509	0.96424
−ΔG° (kJ/mol)		
293 K	2.7548	4.0759
303 K	3.1011	5.5073
313 K	3.3701	6.0849
323 K	3.6893	7.1598
333 K	3.9608	8.8716

where K_c = Langmuir constant (L/mg), R = Universal gas constant; 8.314 J/mol K, T = temperature (K).

The negative value of change in enthalpy and entropy were found to be 28.6595 kJ/mol and 111.888 J/mol for ACBL and 6.0355 kJ/mol and 30.0664 kJ/mol for RBL, respectively (Table-3). The negative value of standard change Gibb's free energy revealed the adsorption were spontaneous process for both cases (adsorption of RhB on RBL and ACBL). During adsorption, the unbalanced residual forces on the surface of the adsorbents are reduced so that energy is released [37]. Hence, the adsorption process is exothermic and change in enthalpy is negative. As the RhB dye solution getting trapped at the surface of the adsorbents, the disorder of the system decreases, consequently the change in entropy is also negative [38].

Comparative studies: The adsorption efficiency of the powdered raw bael leaves and its activated carbon towards the Rhodamine-B dye were also compared with the adsorption effeciency of other reported adsorbents and are presented in Table-4. The absorbent capacity of the prepared activated

TABLE-4
COMPARISON ADSORPTION DATA OF RHODAMINE-B (RhB) DYE WITH DIFFERENT REPORTED ADSORBENTS

Adsorbents	Maximum adsorbent capacities (q_m) (mg/g)	Ref.
Fly ash	1.8706	[39]
Activated carbon of mango leaf powder	3.31	[40]
Chemically treated *Acacia nilotica* leaf	22.37	[41]
Microwave treated *Acacia nilotica* leaf	24.39	[41]
Cation-exchange resin-Duolite C-20	28.57	[42]
Casuarina equisetifolia cone powder	49.5	[43]
Animal bone meal	62.1	[44]
Perlite	63.7	[45]
Azolla pinnata	72.2	[46]
Activated carbon from white sugar	123.46	[47]
RBL	86.89	This study
ACBL	113.82	This study

carbon from bael leaves (ACBL) were found to be far efficient as compare to others.

Conclusion

Raw bael leaves (RBL) and activated carbon of bael leaves (ACBL) have revealed to have better potential as adsorbents for remediation of Rhodamine-B (RhB) dye from wastewater. The RBL is used to prepare activated carbon with H_2SO_4 as a chemical agent. The processes have significantly fast kinetics which reaches equilibrium at 120 min for both the adsorbents. Out of four adsorption isotherms models, the Langmuir isotherm model favours with numerical values of maximum adsorption capacities 86.89 for RBL and 113.82 mg/g of ACBL. In this investigation, the correlation coefficients of the pseudo-second order model for RhB adsorption on both adsorbents were greater than those of other kinetics models. Therefore, the kinetic model for RhB adsorption on RBL and ACBL that best suited the data was the pseudo-second order model. The ΔG° values revealed the adsorption process is spontaneous process. The negative values of ΔH° and ΔS° showed adsorption is exothermic and decrease in randomness. These interpretations suggest that RBL and ACBL are better adsorbents for removing RhB dye from wastewater.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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