

Kinetic, thermodynamic and isotherm studies on the removal of rhodamine b dye using acid activated *Abutilon Indicum*

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Abstract

The research of the present work was to investigate the removal of Rhodamine B dye from aqueous solution using Acid Activated *Abutilon Indicum* leaves (AAAI). Generally, dyes are organic compounds used as colouring products in chemical, textile, paper, printing, leather, plastics and various food industries. There is a need for the treatment of dye contaminated waste water passed out from the industry due to its harmful effects. In this study, the activated carbon prepared from *Abutilon Indicum* leaves was studied for its potential use as an adsorbent for the removal of a cationic dye, Rhodamine B. The various factors affecting the adsorption viz., initial dye concentration, contact time, adsorbent dose and temperature were evaluated. The experimental data were fitted into the pseudo-second order kinetic model. The equilibrium of adsorption was modeled using the Langmuir and Freundlich isotherms. The objective of the present work suggests that the AAAI can be utilized as a low cost adsorbent for the removal of Rhodamine B dye from aqueous solution.

Key words: Acid Activated *Abutilon Indicum* Leaves (AAAI); Rhodamine B (Rh-B); Adsorption isotherm; Kinetics; Equilibrium models.

1. Introduction

Dyes are widely used, generally in the textiles, plastics, paper, leather, food industry to color products. In process of washing and finishing coloured products, waste water contaminated with dyes is generated. The contaminated waste water is hazardous and a great threat to the environment.¹⁻³ Dye contamination in wastewater causes problems in various ways: the presence of dyes in water, even in very low quantities, is highly visible and undesirable; color interferes with penetration of sunlight into waters; retards photosynthesis; inhibits the growth of aquatic biota and interferes with gas solubility in water bodies.⁴ These materials are the complicated organic compounds and they resist against light, washing and microbial invasions. The need for the treatment of dye contaminated waste water arose from the environmental impact. Activated carbon is one of the most popular adsorbents used for the removal of toxic substances from waste water. This could be related to their extended surface area.⁵ The major use of Acid Activated *Abutilon Indicum* leaves (AAAI)⁶ is in solution purification and for the removal of colour, odors and other unpleasant impurities from liquids, water supplies, and vegetable and animal oils.⁷⁻⁹

In recent years it has been increasingly used for the prevention of environmental pollution and antipollution laws have increased the sales of low-cost activated carbon for the control the of air and water pollutions. Various techniques like precipitation, ion exchange, chemical oxidation and adsorption have been used for the removal of toxic pollutant from wastewater. Rhodamine B (Rh-B) is selected as a model compound for evaluating the potential of AAAI to remove dye from aqueous solution.¹⁰⁻¹³

2. Materials and methods

2.1 Adsorption studies

Rhodamine -B was employed as the adsorbate in the adsorption experiment. Adsorption from the liquid phase was carried out to verify the nature the porosity and the capacities of the samples. Aqueous solutions with concentrations of 50-250 mg/L were prepared by dissolving appropriate amount of Rh-B with distilled water. Adsorption experiments were conducted by placing 0.050 g of the AAAI samples and 50 ml of the aqueous solution in a 250 ml of stoppered glass flask. The flask was then put in a constant-temperature shaker bath with a speed of 120 rpm. The isothermal adsorption experiments were performed at $30 \pm 2^\circ\text{C}$.

2.2 Preparation of adsorbent material

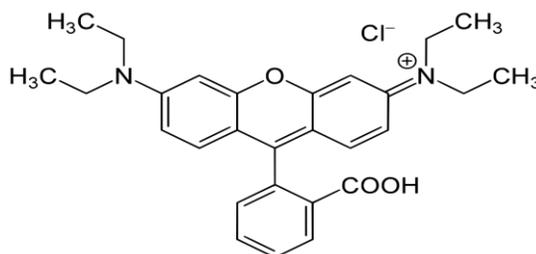


Abutilon Indicum

The *Abutilon Indicum* leaves were collected from agricultural area of Trichy district, were carbonized with concentrated sulphuric acid for 24 hours, washed with water, and activated around 400°C in a muffle furnace for 5 hours. Then it was taken out, ground well to fine powder and stored in a vacuum desiccator.

2.3 Preparation of adsorbate

A known weight of 1000 mg of Rh-B was dissolved in one litre of distilled water to get the stock solution (1000 ppm). Desired concentrations of Rh-B dye were prepared by diluting the stock solution.



Structure of Rhodamine B

2.4 Adsorption dynamic experiments

Batch equilibrium method

The adsorption experiments were carried out in a batch process at 30, 40, 50 and 60°C. A known weight of AAAI was added to 50 ml of the dye solutions with initial concentrations of 50 mg/L to 250 mg/L. The contents were shaken thoroughly using a mechanical shaker with a speed of 120 rpm. The solution was then filtered at preset time interval and the residual dye concentration was measured.

3. Result and discussions

3.1 Characteristics of the adsorbent

Acid Activated *Abutilon Indicum* is an effective adsorbent for the abatement of many pollutants. Most of the solid adsorbents possess micro porous fine structure, high adsorption capacity and high surface area. The wide usefulness of AAI is a result of its surface area, high chemical and mechanical stability. The chemical nature and pore structure usually determine the sorption activity. The prepared AAI was characterized and listed in Table 1.

Table: 1- Characteristics of the Adsorbent AAI

Properties	AAI
Particle size (mm) \leq	0.021
Density (g/cc)	0.285
Moisture content (%)	0.35
Loss on ignition (%)	0.42
Water soluble matter (%)	0.08
pH of aqueous solution	6.5
pH _{zpc}	6.75

3.2 Effect of contact time and initial dye concentration

The adsorption of Rh-B dye at various concentrations ranging from 50 to 250mg/L with contact time was investigated to study the rate of Rh-B dye removal. It was observed that equilibrium has been established at 50 minutes for all concentrations. The equilibrium data presented in table 2 reveal that the percentage of adsorption decreased with increase in initial Rh-B dye concentrations, but the actual amount of Rh-B dye adsorbed per unit mass of AAI increased with increase in Rh-B dye concentration. It means that the adsorption is highly dependent on the initial concentration of Rh-B dye. It is because of the reason that at lower concentration, the ratio of the initial number of Rh-B dye to the available surface area is low; the fractional adsorption becomes independent of initial concentration. However, at higher concentrations, the available sites of adsorption become fewer and hence the percentage removal of Rh-B dye is dependent upon initial concentration.¹⁴ There was an optimum removal of Rh-B dye observed at the initial concentration of 50mg/L of Rh-B dye concentrations. The effect of contact time on the percentage removal of Rh-B dye is given in figure 1, which reveals that the curve is single, smooth and continuous leading to saturation, suggesting the possibility of monolayer coverage of the Rh-B dye on the AAI adsorbent surface.¹⁵

Table: 2- Equilibrium Parameters for the Adsorption of Rh-B Dye onto AAI

C ₀	C _e (Mg / L)				Q _e (Mg / L)				Percentage Removal			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
50	2.64	2.65	2.54	2.52	44.70	44.68	44.90	44.95	89.41	89.37	89.81	89.91
100	5.44	5.84	5.46	4.87	89.10	88.31	89.06	90.25	89.10	88.31	89.06	90.25
150	13.83	12.89	11.84	10.65	122.3	124.20	126.3	128.6	81.55	82.80	84.20	85.79
200	26.74	25.64	11.84	28.44	146.5	148.7	176.3	143.1	73.25	74.35	88.15	71.55
250	45.84	40.33	23.84	32.98	158.3	169.3	202.3	184.0	63.32	67.72	80.92	73.61

3.3 Effect of adsorbent dosage

The adsorption of Rh-B dye on AAI was studied by varying the dosage of adsorbent (25 - 250 mg / 50ml) for 50 mg L⁻¹ of Rh-B dye concentration. The percentage of adsorption was increased with increase in AAI dose (Figure 2). This may be due to the increased AAI surface area and the availability of more adsorption sites.¹⁶ Since for an initial Rh-B dye ion concentration of 50 mg L⁻¹, the optimum Rh-B dye removal was obtained at a maximum carbon dose of 50 mg, the remaining parts of the experiments were carried out with 50 mg of adsorbent for 50 ml of adsorbate solution.

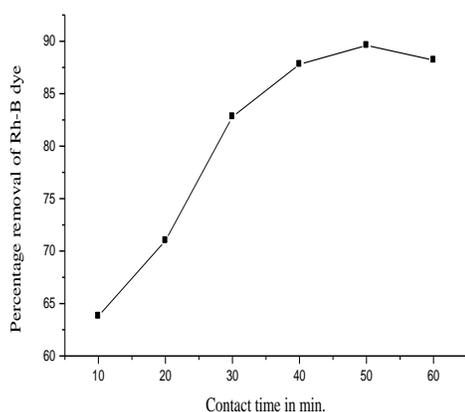


Fig.1: Effect of contact time on the removal of Rh-B dye
[Rh-B]=50mg/L; Adsorbent dose=50mg/50ml; pH=6.7; Temp.= 30°C

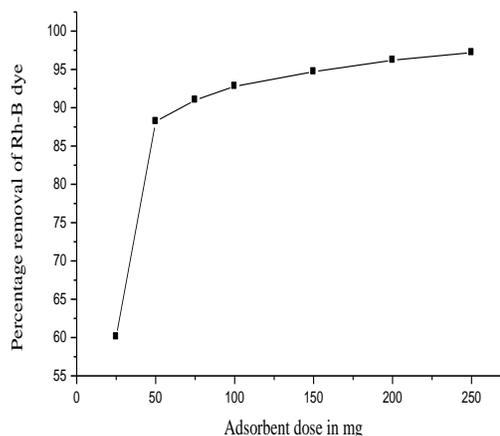


Fig.2: Effect of adsorbent dose on the removal of Rh-B dye
[Rh-B]=50mg/L; Contact time=50 min.; pH=6.7; Temp.=30°C

3.4 Effect of solution pH

One of the most important parameters controlling the adsorption process is pH. The effect of pH of the solution on the adsorption of Rh-B dye on AAI was determined. The pH of the solution was controlled by the addition of HCl or NaOH. The result is shown in figure 3. It was observed that the maximum uptake of Rh-B dye was obtained from pH 2.0 – 6.7. The maximum percentage removal of Rh-B dye occurred at pH 6.7 (closer to pH_{zpc}). However, when the pH of the solution was increased (more than 6.7), the uptake of Rh-B dye was decreased. It appears that a change in pH of the solution results in the formation of different ionic species and different AAI surface charge. At pH values lower than 6.7, the Rh-B dye can enter into the pore structure. At pH values higher than 6.7, the zwitter ionic form of Rh-B dye in water may increase the aggregation of Rh-B dye to form a bigger molecular form (dimer) and become unable to enter the pore structure of the AAI surface.^{14,16}

3.5 Effect of ionic strength

The effect of chloride ion on the adsorption of Rh-B dye on AAI in a low concentration of NaCl had a little influence on the adsorption capacity (Figure 4). At higher ionic strength, the adsorption of Rh-B dye is increased due to the partial neutralization of the positive charge on the AAI surface and a consequent compression of the electrical double layer by the chloride ion. The chloride ion can also enhance the adsorption of Rh-B dye onto AAI by pairing of their charges and hence reducing the repulsion between the Rh-B dye molecule and adsorbent surface. This initiates AAI to adsorb more amount of positive Rh-B dye.¹⁷

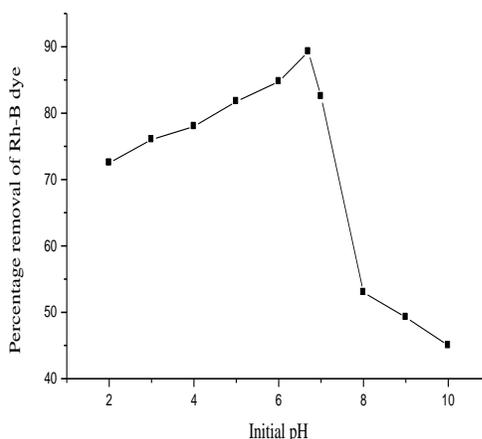


Fig.3: Effect of initial pH on the removal of Rh-B dye

[Rh-B]=50 mg/L; Contact time= 50 min.; Adsorbent dose = 50 mg/50ml; Temp.=30°C

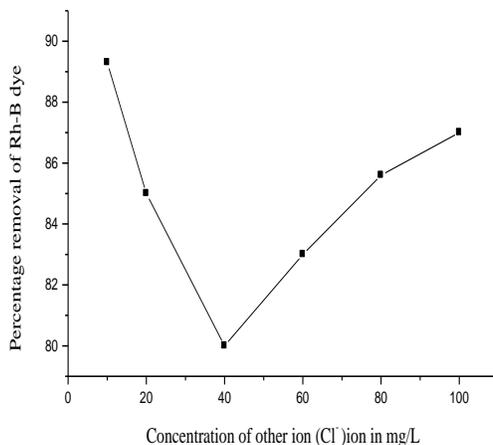


Fig. 4: Effect of ionic strength on the removal of Rh-B dye

[Rh-B]=50mg/L; Contact time=50 min; Adsorbent dose=50mg/50ml; pH=6.7; Temp.=30°C

3.6. Adsorption isotherms

3.6.1 Langmuir isotherm

The theoretical Langmuir isotherm is used for adsorption of a solute from a liquid solution as monolayer adsorption on a surface containing a finite number of identical sites. Therefore, the Langmuir isotherm model was chosen for estimation of the maximum adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface.¹⁸ The Langmuir non-linear equation is commonly expressed as follows:

$$C_{eq}/Q_{eq} = 1/Q_m b + C_{eq}/Q_m \dots\dots\dots (1)$$

Where C_{eq} is the equilibrium concentration of adsorbate in the solution (mg/L), Q_{eq} is the amount adsorbed at equilibrium (mg/g), Q_m and b are Langmuir constants related to adsorption efficiency and energy of adsorption, respectively. The linear plots of C_{eq}/Q_{eq} vs. C_{eq} suggest the applicability of the Langmuir isotherms. The values of Q_m and b were calculated from slope and intercepts of the plots are given in Table 3. From the results, it is obvious that the value of adsorption efficiency Q_m and adsorption energy b of the AAI increases on increasing the temperature. The values can conclude that the maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on adsorbent surface with endothermic nature of adsorption. To confirm the favorability of the adsorption process, the separation factor (R_L) was determined and given in Table 4. The values were established to be between 0 and 1 and confirm that the ongoing adsorption process is favorable.

3.6.2 The Freundlich isotherm

The Freundlich isotherm model is the earliest known equation describing the adsorption process. It is an empirical equation and can be used for non-ideal sorption that involves heterogeneous adsorption. The Freundlich equation was employed for the adsorption of Rh-B dye on the adsorbent.¹⁹ The Freundlich isotherm was represented by the following equation.

$$\log Q_e = \log K_f + 1/n \log C_e \dots\dots\dots(2)$$

where, Q_e is the amount of Rh-B dye adsorbed (mg/g), C_e is the equilibrium concentration of dye in solution (mg/L), and K_f and n are constants incorporating the factors affecting the adsorption capacity and intensity of adsorption, respectively. The linear plots of $\log Q_e$ vs $\log C_e$ show that the adsorption of Rh-B dye follows the Freundlich isotherm. The values of K_f and n are calculated from the intercept and slope of the plot $\log Q_e$ vs $\log C_e$ respectively and these values are tabulated in table 3. The magnitude of the exponent $1/n$ gives an indication of the favorableness of adsorption. The value of n lying in the range of 1 to 10 confirms the favorable condition for adsorption. This is further supported by Langmuir isotherm.

**Table: 3 - Langmuir and Freundlich Isotherm Parameters
for the Adsorption of Rh-B onto AAAI**

Temp. (°C)	Langmuir Parameters		Freundlich Parameters	
	Q _m	b	K _f	n
30	183.02	0.1444	4.7452	2.3763
40	204.13	0.1154	4.5714	2.1330
50	340.96	0.0626	4.1089	1.4633
60	205.77	0.1316	4.7424	2.1604

Table: 4 - Dimensionless Separation Factor (R_L) for the Adsorption of Rh-B onto AAAI

C ₀	Temperature °C			
	30°C	40°C	50°C	60°C
50	0.2168	0.2572	0.3896	0.2330
100	0.1216	0.1476	0.2419	0.1318
150	0.0845	0.1034	0.1754	0.0919
200	0.0647	0.0796	0.1376	0.0706
250	0.0524	0.0647	0.1132	0.0572

3.6 Effect of temperature

To study the effect of temperature on the adsorption of dye adsorption by AAAI, the experiments were performed at temperatures of 30, 40, 50, 60°C. It was observed that the equilibrium adsorption capacity of Rh-B onto AAAI was found to increase with increasing temperature. This indicates that the mobility of dye molecules increased with the temperature. Thermodynamic parameters such as change in free energy (ΔG°) (kJ/mol), enthalpy (ΔH°) (kJ/mol) and entropy (ΔS°) (J/K/mol) were determined using the following equations.

$$K_0 = C_{\text{solid}}/C_{\text{liquid}} \dots\dots\dots (3)$$

$$\Delta G^\circ = -RT \ln K_0 \dots\dots\dots (4)$$

$$\log K_0 = \Delta S^\circ / (2.303R) - \Delta H^\circ / (2.303RT) \dots\dots\dots (5)$$

Where K_0 is the equilibrium constant, C_{solid} is the solid phase concentration at equilibrium (mg/L), C_{liquid} is the liquid phase concentration at equilibrium (mg/L), T is the temperature in Kelvin, and R is the gas constant. The ΔH° and ΔS° values obtained from the slope and intercept of Van't Hoff plots are given in Table 5. The values of ΔH° is the range of 1 to 19 kJ/mol, indicate the

physisorption. The results show that physisorption is much feasible for the adsorption of Rh- B. The positive values of ΔH° show the endothermic nature of adsorption which governs the possibility of physical adsorption. In the case of physical adsorption, while increasing the temperature of the system, the extent of dye adsorption increases. The negative values of ΔG° (Table 5) show that the adsorption is highly favorable and spontaneous. The positive values of ΔS° (Table 5) show the increased disorder and randomness at the solid solution interface of Rh-B with AAAI adsorbent. The enhancement of adsorption capacity of the activated AAAI at higher temperatures was ascribed to the enlargement of pore size and activation of the adsorbent surface.²⁰

Table: 5- Thermodynamic Parameters for the Adsorption of Rh-B onto AAAI

C ₀	ΔG°				ΔH°	ΔS°
	30°C	40°C	50°C	60°C		
50	-5376.67	-5541.3	-5845.4	-6057.41	1.7360	23.40
100	-5292.81	-5262.43	-5631.9	-6161.48	3.6496	29.046
150	-3744.83	-4090.38	-4494.8	-4979.05	8.6780	40.896
200	-2538.41	-2770.39	-5390.4	-2554.5	6.3538	30.402
250	-1375.76	-1929.25	-3880.9	-2840.56	18.286	65.387

3.7. Adsorption kinetics

The study of adsorption dynamics describes the solute up take rate and evidently this rate controls the residence time of adsorbate uptake at the solid-solution interface .The kinetics of Rh-B dye adsorption on the AAAI were analyzed using pseudo second-order, Elovich and intra-particle diffusion kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation co- efficient (γ) and the values are close or equal to 1. A relatively high correlation coefficient (γ) value indicates that the pseudo second-order model successfully describes the kinetics of Rh-B dye adsorption.

3.7.1 The pseudo second- order equation

The pseudo second-order adsorption kinetic rate equation²¹ is expressed as

$$dq_t/dt = k_2(q_e - q_t)^2 \quad \dots\dots\dots(6)$$

where, k_2 is the rate constant of pseudo second- order adsorption (g mg/min). For the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$ the integrated form of Eq. (6) becomes

$$1/(q_e - q_t) = 1/q_e + k_2t \quad \dots\dots\dots (7)$$

This is the integrated rate law for a pseudo second-order reaction.

Equation (7) can be rearranged to obtain Eq.(8),which has a linear form:

$$t/q_t = (1/k_2 q_e^2) + ((1/q_e)t) \dots\dots\dots(8)$$

If the initial adsorption rate (h)(mg g⁻¹min⁻¹) is given as

$$h = k_2 q_e^2 \dots\dots\dots (9)$$

Equation (8) becomes

$$t / q_t = 1 / h + 1 / q_e t \dots\dots\dots(10)$$

The plot of (t/q_t) and t of Eq. (10) gives a linear relationship from which q_e and k₂ can be determined from the slope and intercept of the plot, respectively. The pseudo-second order rate constants k₂, the calculated h values, and the correlation coefficients (γ) are summarized in Table 6. At all studied initial Rh-B dye concentrations, the straight lines with extremely high correlation coefficient (>0.99) were obtained. From table 6 it is seen that the values of the rate constant k decrease with increasing initial Rh-B dye concentration for AAI adsorbent. This shows that the sorption of Rh-B dye on AAI follows pseudo- second order kinetic model.

3.7.2 The Elovich equation

The Elovich model²² equation is generally expressed as

$$dq_t / dt = \alpha \exp (-\beta q_t) \dots\dots\dots(11)$$

where, α is the initial adsorption rate (mg g⁻¹ min⁻¹) and β is the desorption constant (g/mg) during any one experiment. To simplify the Elovich equation, assumed that αβt >> t and by applying boundary conditions q_t = 0 at t= 0 and q_t = q_t at t = t Eq.(11) becomes

$$q_t = 1/\beta \ln (\alpha\beta) + 1/\beta \ln t \dots\dots\dots (12)$$

If Rh-B dye adsorption fits with the Elovich model, a plot of q_t vs. ln(t) should yield a linear relationship with a slope of (1/β) and an intercept of (1/β)ln (αβ). The Elovich model parameters α, β, and correlation coefficient (γ) are summarized in table 6. The experimental data viz., the initial adsorption rate (α) adsorption constant (β) and the correlation coefficient (γ) calculated from this model indicate that the initial adsorption (α) increases with temperature similar to that of initial adsorption rate (h) in pseudo-second-order kinetics model. This may be due to increase the pore or active site on the AAI adsorbent.

3.7.3 The intra-particle diffusion model

The intra-particle diffusion model²⁴ used here refers to the theory proposed by Weber and Morris²³ based on the following equation for the rate constant:

$$q_t = K_{id} t^{(1/2)} + C \dots \dots \dots (13)$$

where K_{id} is the intra-particle diffusion rate constant (mg/g/min) and C is the constant. The rate limiting step is intra-particle diffusion. The graph drawn between (q_t) (mg/g) verses square root of the contact time ($t^{1/2}$) yields a straight line passing through the origin. The slope gives the value of the intra-particle diffusion coefficient (K_{id}), and correlation coefficients (γ) indicate the fitness of this model. The value of C gives an idea about the thickness of the boundary layer. The values of intercept (C) indicate that the lines were not passing through origin and hence, some other processes also affect the adsorption process. But the correlation coefficient (γ) values are very high and therefore, the intra-particle diffusion takes place along with other process that may affect the adsorption. The values are given in table 6.

Table: 6- The Kinetic Parameters for the Adsorption of Rh-B onto AAI

C ₀	Temp °C	Pseudo-second order				Elovich model			Intra-particle diffusion		
		q _e	k ₂	γ	h	α	β	γ	K _{id}	γ	C
50	30	48.57	0.0022	0.994	8.6257	94.65	0.1503	0.992	1.6554	0.999	0.1684
	40	47.20	0.0018	0.995	12.089	1385	0.2206	0.991	1.7542	0.998	0.1099
	50	47.30	0.0017	0.997	12.149	2977	0.2401	0.992	1.7713	0.997	0.0998
	60	49.04	0.0022	0.999	8.172	113.84	0.1555	0.991	1.6686	0.994	0.1602
100	30	98.08	0.0024	0.998	13.464	140.44	0.0734	0.993	1.6339	0.995	0.1737
	40	94.22	0.0020	0.997	19.166	1512	0.1054	0.991	1.7311	0.997	0.1170
	50	94.06	0.0018	0.998	23.169	3348	0.1137	0.992	1.7559	0.999	0.1068
	60	95.87	0.0018	0.992	22.278	1524	0.1017	0.991	1.7426	0.998	0.1187
150	30	131.37	0.0022	0.994	25.091	1137	0.0701	0.991	1.6789	0.997	0.1277
	40	133.18	0.0022	0.991	26.075	1305	0.0702	0.994	1.6897	0.998	0.1255
	50	134.93	0.0021	0.992	27.618	1983	0.0727	0.995	1.7088	0.992	0.1186
	60	137.89	0.0021	0.992	27.756	1448	0.0682	0.997	1.7079	0.991	0.1245
200	30	159.42	0.0025	0.991	24.508	479.3	0.0507	0.999	1.5890	0.993	0.1502
	40	160.43	0.0023	0.993	28.794	939.6	0.0547	0.998	1.6250	0.991	0.1354
	50	164.56	0.0011	0.991	31.172	1066	0.0537	0.997	1.6418	0.992	0.1337
	60	149.49	0.0013	0.992	76.512	163930	0.0938	0.998	1.7270	0.991	0.0773
250	30	175.67	0.0025	0.991	23.317	406.02	0.0451	0.992	1.5155	0.991	0.1555
	40	184.03	0.002431	0.991	30.027	526.66	0.0431	0.994	1.5540	0.991	0.1530
	50	191.94	0.001599	0.991	31.296	625.97	0.0423	0.991	1.5785	0.994	0.1490
	60	199.22	0.002415	0.992	33.344	892.22	0.0427	0.992	1.6093	0.995	0.1408

3.7.4 Desorption studies:

Desorption studies help to elucidate the nature of adsorption and recycling of the spent adsorbent and the dye. If the adsorbed dye can be desorbed using neutral pH water, then the attachment of the dye molecule of the adsorbent is by weak bonds. The effect of various reagents used for desorption studies indicate that hydrochloric acid is a better reagent for desorption, because we could get more than 85% removal of adsorbed dye. The reversibility of adsorbed dyes in mineral acid is in agreement with the pH dependent results obtained. The desorption of dye molecules by mineral acids indicates that the dye was adsorbed onto the AAI through physisorption mechanism.²⁵

Conclusion

The present study has shown the effectiveness of using AAI in the removal of Rhodamine-B dye from aqueous solution. Acid Activated *Abutilon Indicum* leaves in different form have a great role in modern life to clean environment. *Abutilon Indicum* Leaves can be good precursors for producing highly porous adsorbent by simple preparative method. An adsorption test has been carried out for industrial pollutant, Rhodamine - B (Rh-B) under different experimental conditions in batch mode. The adsorption of Rh-B was dependent on adsorbent surface characteristics, adsorbent dose, Rh-B concentration, time of contact, pH and temperature. A study of the kinetic models on adsorption showed that the adsorption fits with the pseudo- second order kinetic model. The thermodynamic parameters ΔG° , ΔH° , and ΔS° values reveal the favorability of adsorption and the adsorption on AAI was a spontaneous and endothermic in nature.

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