Kinetic, Thermodynamic and Isotherm Studies on the Removal of Rhodamine B dye using Activated Carbon

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Abstract

The research of the present work was to investigate the removal of Rhodamine B dyes from aqueous solution by using Activated Acacia Nilotica Shell Carbon (ANSC). Generally, dyes are organic compounds used as colouring products in chemical, textile, paper, printing, leather, plastics and various food industries. The need for the treatment of dye contaminated waste water passed out from the industry. In this study, Acacia Nilotica Shell was studied for its potential use as an adsorbent for removal of a cationic dye Rhodamine B (Rh-B).The various factors affecting adsorption, such as initial dye concentration, contact time, adsorbent dose and effect of temperature, were evaluated. The experimental data were fitted into the pseudo-second order kinetic model. The equilibrium of adsorption was modeled by using the Langmuir and Freundlich isotherm models. The objective of the present work suggests the ANSC may be utilized as a low cost adsorbent for Rhodamine B dye removal from aqueous solution.

Key words: Activated Acacia Nilotica Shell Carbon (ANSC); Rhodamine-B (Rh-B); Adsorption isotherm; Kinetics; Equilibrium models.

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 waters are hazardous, which is a great threat to 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. These materials are the complicated organic compounds and they resist against light, washing and microbial invasions [4-7]. The need for the treatment of dye contaminated waste water arose from the environmental impact [8]. Activated minerals are 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 [9]. The major use of Activated Acacia Nilotica Shell is in solution purification and for the removal of colour, odors and other unpleasant impurities from liquids, water supplies and vegetable and animal oils.

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 minerals for control of the air and water pollution. 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 ANSC to remove Rh-B dye from aqueous solution.

Materials and methods

Adsorption studies

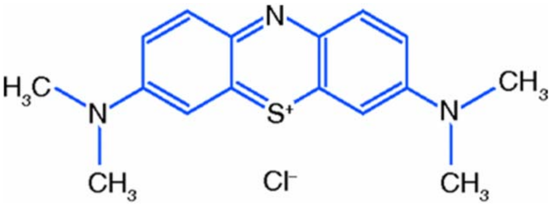
Rhodamine B (Rh-B) was employed for the adsorbate in the adsorption experiments. Adsorption from the liquid phase was carried out to verify the nature the porosity and the capacities of the samples. An aqueous solution with a concentration of 50-250 mg/L was prepared by mixing an appropriate amount of Rh-B with distilled water adsorption experiments were conducted by placing 0.025 g of the ANSC samples and 50 ml of the aqueous solution in a 250 ml of glass-stoppered flask. The flask was then put in a constant-temperature shaker bath with a shaker speed of 150 rpm. The isothermal adsorption experiments were performed at 30 ± 2°C.

Preparation of adsorbent materials

The Acacia Nilotica Shell collected from agricultural area nearby Thiruvarur districts was Carbonized with concentrated Sulphuric Acid and washed with water and activated around 600°C in a muffle furnace for 5 hrs the it was taken out, ground well to fine powder and stored in a vacuum desiccators.

Preparation of adsorbate

Rhodamine B was chosen in this work because of its strong adsorption onto solids and it recognized usefulness in characterizing adsorptive material [10, 11] Rhodamine B is employed to evaluate the adsorption characteristics of carbon. A known weight of 1000 mg of Rh-B was dissolved in about one litre of distilled water to get the stock solution.



Structure of Rhodamine B

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 ANSC was added to 50 ml of the dye solutions with an initial concentration of 50 mg/L to 250 mg/L, which is prepared from 1000 mg/L of Rhodamine B stock solution. The contents were shaken thoroughly using a mechanical shaker with a speed of 150 rpm. The solution was then filtered at present time intervals and the residual dye concentration was measured.

Result and discussions

Characteristics of the adsorbent

Activated Acacia Nilotica Shell is an effective adsorbent for the abatement of many pollutant compounds (organic, inorganic, and biological) of concern in water and wastewater treatment. Most of the solid adsorbents possess micro porous fine structure, high adsorption capacity, high surface area and high degree of surface, which consists of pores of different sizes and shapes [12]. The wide usefulness of ANSC is a result of their specific surface area, high chemical and mechanical stability. The chemical nature and pore structure usually determines the sorption activity. The physico-chemical properties of the chosen adsorbent are listed in Table 1.

Table 1-Characteristics of the adsorbent

Properties	**ANSC**
Particle size(mm)	0.0500
Density (g/cc)	0.1414
Moisture content (%)	0.1348
Loss in ignition (%)	0.0212
pH of aqueous solution	6.5000

Effect of contact time and initial dye concentration

The effect of contact time on the amount of dye adsorbed was investigated at 1000 mg/L concentration of the dye fig (1). It is observed that the percentage removal of dye increases rapidly with an increase in contact time initially, and thereafter, beyond a contact time of about 50 min, no noticeable change in the percentage removal is observed the percentage removals after 50 min were 92%. Therefore, the optimum contact time is considered to be 50 min. this is also the equilibrium time of the batch adsorption experiments, since beyond a contact time of 50 min, adsorption is not changed. The rapid removal of Rh-B dye is observed at the beginning of the contact time due to the percentage of large number of binding sites available for adsorption. The experimental results of adsorptions at different concentrations (50 to 250 mg/L) collected in Table 2 observed that percent adsorption decreased with increase in initial dye concentration, but the actual amount of Rh-B dye adsorbed per unit mass of ANSC increased leads to increase in dye concentration. This means that the adsorption is highly dependent on initial concentration of dye. At lower concentration, the ratio of the initial number of dye molecules to the available surface area is low. Subsequently, the fractional adsorption becomes independent of initial concentration. However, at high concentration the available sites of adsorption become less and hence the percentage removal of dye is dependent upon initial concentration [15, 16].

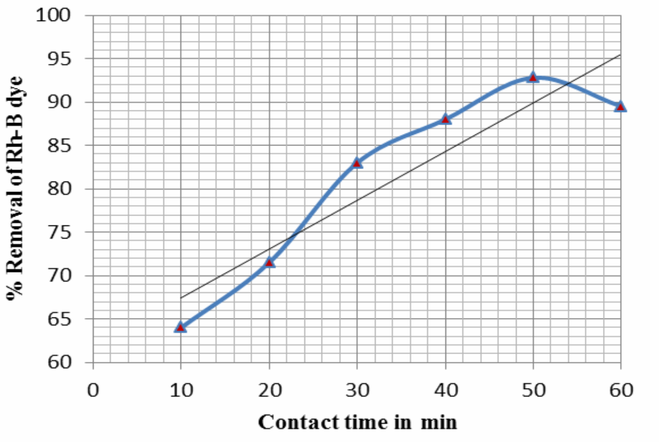


Fig. 1-Effect of contact time on the removal of Rh-B dye
[Rh-B]=50 mg/L; dose=25mg/50ml; pH=6.5; Temp.=30^{o}C

Effect of adsorbent dosage

The adsorption of the Rhodamine B dye on ANSC was studied by varying the adsorbent dose (10-250 mg/50ml) for 50 mg/L of dye concentration. The percentage of adsorption increased with increases in the ANSC concentration, which is attributed to increased carbon surface area and the availability of more adsorption sites [13, 14]. Hence, all studies were carried out with 0.025g of adsorbent/50 ml of the varying adsorbate solutions 50, 100, 150, 200 and 250. The Results obtained from this study are shown in figure (2). The amount of Rh-B adsorbed per gram reduced with increase in the dosage of ANSC. This reveals that the direct and equilibrium capacities of Rh-B are functions of the ANSC dosage.

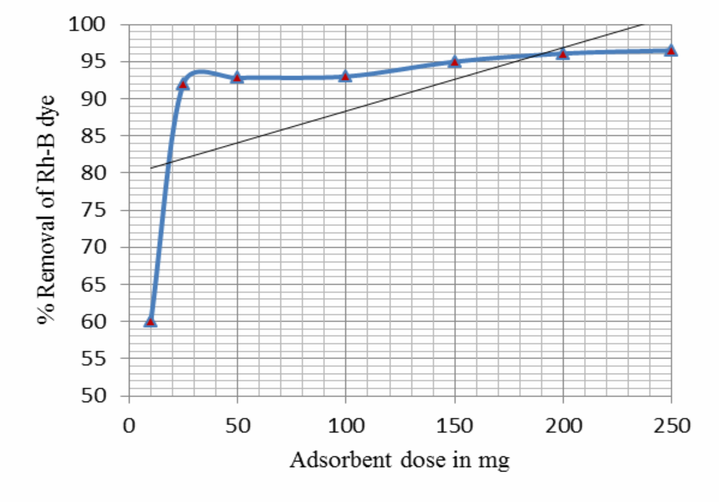


Fig. 2-Effect of adsorbent dose on the removal of Rh-B dye
[Rh-B]=50mg/L; contact time=50min; pH=6.5; Temp.=30^{o}C

Effect of solution pH

The solution pH is one of the most important factors that control the adsorption of dye on the sorbent material. The adsorption capacity can be attributed to the chemical form of dye in the solution at specific pH. In addition, due to different functional groups on the adsorbent surface, which become active sites for the dye binding at a specific pH the effect of adsorption can vary substantially. Therefore, an increase in pH may cause an increase or decrease in the adsorption, resulting different optimum pH values dependent on the type of adsorbent. To examine the effect of pH on the % removal of Rh-B dye , the solution pH were varied from 2.0 to 10.0 by adding acid and base to the stock solution

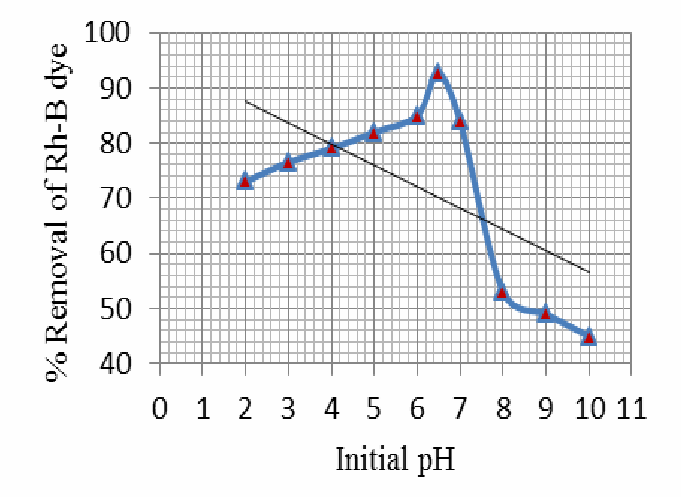


Fig. 3-Effect of initial pH on the removal of Rh-B dye
[Rh-B]=50 mg/L; contact time=50min;dose=25mg/50ml;Temp.=30^{o}C

This increases may be due to the presence of negative charge on the surface of the adsorbent ANSC that may be responds for the dye binding. However, as the pH is lowered, the hydrogen ions compete with dye for the adsorption sites in the adsorbent ANSC, the overall surface charge on the particles become positive and hinds the binding of positively charged dye. On other hand, decrease in the adsorption under pH >6.5 may be due to occupation of the adsorption sites by OH^- ions which retard the approach of such dye further toward the adsorbent ANSC surface. From the experimental results, the optimum pH range for the adsorption of the Rh-B dye is 2.0 to 6.5 shown in Figure.3.

Table: 2. Equilibrium Parameters for the Adsorption of Rh-B dye onto ANSC

	Ce (mg / L)				**Qe (mg / g)**				**Removed (%)**			
M_0	**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	3.5002	4.0124	3.2385	2.8740	92.831	91.965	93.422	94.240	92.822	91.865	93.412	94.142
100	14.575	12.878	10.655	9.2480	170.74	174.14	178.59	181.50	85.314	87.012	89.235	90.642
150	30.154	27.248	23.575	20.548	239.59	245.40	252.74	258.50	79.786	81.724	84.173	86.291
200	58.248	54.287	23.575	44.879	283.51	291.32	352.74	310.24	70.766	72.746	88.102	77.450
250	90.387	85.342	49.248	74.287	319.21	329.21	401.40	351.42	63.735	65.753	80.290	70.174

Adsorption isotherms

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. The Langmuir non-linear equation is commonly expressed as follows:

$C_{eq}/Q_{eq} = 1/Q_m b + C_{eq}/Q_m$ ……….. (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 ANSC 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 [17, 18]. 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 [19].

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 Rhodamine B dye on the adsorbent. The Freundlich isotherm was represented by the following equation.

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

Where, Q_e is the amount of Rhodamine 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. Linear plots of log Q_e Vs log C_e shows that the adsorption of Rhodamine B obeys the linear plots of log Q_e Vs log C_e shows that the adsorption of Rhodamine B dye obeys the Freundlich adsorption isotherm. The values of K_f and '*n*' are given in Table 3 shows that the increase of negative charges on the adsorbent surface makes electrostatic force like van der Waal's between the ANSC surface and dye ion. The molecular weight and size either limit or increase the possibility of the adsorption of the dye onto adsorbent. However, the values clearly show the dominance in adsorption capacity.

The intensity of adsorption is an indication of the bond energies between dye and adsorbent, and the possibility of slight chemisorptions rather than physisorption [17, 20]. However, the multilayer adsorption of Rhodamine B through the percolation process may be possible. The values of *n* are less than one, indicating the physisorption is much more favorable [21].

Table: 3. Langmuir and Freundlich Isotherm Parameter for the Adsorption of Rh-B dye onto ANSC

Temp. °C	**Langmuir Parameter**		**Freundlich parameter**	
	Q_m	**b**	**K_f**	**n**
30	351.78	0.0702	5.8616	2.5729
40	369.04	0.0687	5.7281	2.3883
50	536.75	0.0511	5.4327	1.8175
60	335.62	0.0939	6.2147	2.4770

Table: 4. Dimensionless Separation factor (R_L) for the Adsorption of Rh-B dye onto ANSC

	Temperature °C			
C_0	**30 °C**	**40 °C**	**50 °C**	**60 °C**
50	0.2181	0.2128	0.2770	0.1639
100	0.1230	0.1153	0.1308	0.0942
150	0.0855	0.0802	0.1032	0.0645
200	0.0655	0.0658	0.0774	0.0510
250	0.0531	0.0532	0.0611	0.0414

Effect of temperature

To study the effect of temperature on the adsorption of Rh-B dye adsorption by ANSC, the experiments were performed at temperatures of 30, 40, 50, 60°C. As it was observed that, the equilibrium adsorption capacity of Rh-B onto ANSC was found to increase with increasing temperature, especially in higher equilibrium concentration, or lower adsorbent dose because of high driving force of adsorption. This fact indicates that the mobility of Rh-B dye molecules increased with the temperature. The adsorbent shows the endothermic nature of adsorption. The adsorption capacity of the ANSC increased with increase of the temperature in the system from 30° to 60°C. Thermodynamic parameters such as change in free energy ($\Delta G°$) (kJ/mol), enthalpy ($\Delta H°$) (kJ/mol) and entropy ($\Delta S°$) (J/K/mol) were determined using the following equations.

$K_0 = C_{solid}/C_{liquid}$ …………………….. (3)

$\Delta G° = -RT \ln K_0$ …………….……….. (4)

$\log K_0 = \Delta S°/ (2.303R) - \Delta H°/2.303RT)$ ….(5)

Where, K_o 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 $\Delta H°$ and $\Delta S°$ values obtained from the slope and intercept of Van't Hoff plots are given in Table 5.The values of $\Delta H°$ is the range of 9 to 17 kJ/mol, indicate the physisorption. The results show that physisorption is much feasible for the adsorption of Rhodamine B (Rh-B). The positive values of $\Delta H°$ show the endothermic nature of adsorption which governs the possibility of physical adsorption [19, 22]. Because in the case of physical adsorption, while increasing the temperature of the system, the extent of dye adsorption increases, there is no possibility of chemisorption. The negative values of $\Delta G°$ (Table 5) show that the adsorption is highly favorable and spontaneous.

Table: 5. Thermodynamic Parameter for the Adsorption of Rh-B dye onto ANSC

	ΔG°					
C_0	**30° C**	**40° C**	**50° C**	**60° C**	**ΔH°**	**ΔS°**
50	-6351	-6336	-7159	-7644	7.727	46.01
100	-4354	-4875	-5610	-6222	14.64	63.16
150	-3376	-3816	-4410	-5195	13.12	54.22
200	-2140	-2469	-5304	-3333	17.75	66.80
250	-1332	-1610	-3673	-2373	13.83	51.05

The positive values of $\Delta S°$ (Table 5) show the increased disorder and randomness at the solid solution interface of Rhodamine B with ANSC adsorbent. The enhancement of adsorption capacity of the

ANSC at higher temperatures was described to the enlargement of pore size and activation of the adsorbent surface [23].

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 ANSC were analyzed using pseudo second-order[25] Elovich[26] and intra-particle diffusion[24] 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.

The pseudo second- order equation

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

$dq_t/d_t = k_2(q_e-q_t)^2$ ………..…..(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:

$1/(q_e - q_t) = 1/q_e + k_2t$ ………………. (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:

$t/q_t=(1/k_2q_e^2)+ ((1/q_e)t$ ………………. (8)

If the initial adsorption rate (h) (mg/g/min) is:

$h=k_2q_e^2$ ………………………………. (9)

Equation (8) and (9) becomes,

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

The plot of (t/q_t) and t of Eq. (10) should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot, respectively. The pseudo-second order rate constants k_2, 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 co-efficient (>0.99) were obtained. From table 6, the values of the rate constant k decrease with in increasing initial Rh-B dye concentration for ANSC powder. This is shows that the sorption of Rh-B dye on ANSC follows pseudo second order kinetic model [25].

The Elovich equation

The Elovich model equation is generally expressed as

$dq_t / d_t = \alpha \exp (-\beta q_t)$ ………(11)

Where, α is the initial adsorption rate (mg/g/min) and β is the desorption constant (g/mg) during any one experiment. To simplify the Elovich equation [26]. Assumed αβt>>t and by applying boundary conditions $q_t = 0$ at t= 0 and $q_t = q_t$ at t = t Eq.(11) becomes:

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

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 such as the initial adsorption rate (α) adsorption constant (β) and the correlation co-efficient (γ) calculated from this model indicates that the initial adsorption (α) increases with temperature similar to that of initial adsorption rate (h) in pseudo-second–order kinetics models. This may be due to increase the pore or active site on the ANSC adsorbent.

The intra particle diffusion model

The intra-particle diffusion model used here refers to the theory proposed by Weber and Morris [27] based on the following equation for the rate constant:

$q_t = k_{id}\, t^{(1/2)} + C$………………(13)

Where, k_{id} is the intra-particle diffusion rate constant (mg/g/min) and C is the constant. If the rate limiting step is intra-particle diffusion, then the graph drawn between (q_t) (mg/g) verses square root of the contact time ($t^{1/2}$) should yield a straight line passing through the origin [27]. The slope of the will give the value of the intra-particle diffusion coefficient (k_{id}) and correlation coefficient (γ) indicate the fitness of this model. The value of 'C' gives an idea about the thickness of the boundary layer. From these data the intercept value indicate that the line were not passing through origin, there are some other process affect the adsorption. But the correlation coefficient (γ) value is very high, so that intra-particle diffusion takes place along with other process that may affect the adsorption. The values are given in table 6.

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. The results indicate that hydrochloric acid is a better reagent for desorption, because we could get more than 90% removal of adsorbed dyes. The reversibility of adsorbed dyes in mineral acid or base is in agreement with the pH dependent results obtained. The desorption of dye molecules by mineral acids and alkaline medium indicates that the dye was adsorbed onto the ANSC through physisorption as well as by chemisorptions mechanisms.

Table: 6. The Kinetic Parameters for the Adsorption of Rh-B dye onto ANSC

		Pseudo Second Order				**Elovich Model**			**Intraparticle Diffusion**		
C_0	**Temp.°C**	**q_e**	**k_2**	γ	**h**	α	β	γ	**K_{id}**	γ	**C**
50	**30**	102.11	0.0016	0.9981	16.776	301.41	0.0658	0.992	1.6839	0.9982	0.1510
	40	99.078	0.0020	0.9975	19.719	672.62	0.0872	0.991	1.7149	0.9972	0.1335
	50	100.49	0.0019	0.9969	19.860	770.20	0.0898	0.993	1.7255	0.9969	0.1295
	60	101.41	0.0018	0.9973	19.420	632.47	0.0857	0.991	1.7224	0.9981	0.1345
100	**30**	186.28	0.0008	0.9989	29.816	452.02	0.0427	0.992	1.6376	0.9948	0.1571
	40	189.68	0.0008	0.9928	30.439	529.14	0.0409	0.994	1.6525	0.9994	0.1529
	50	193.69	0.0008	0.9941	33.270	715.25	0.0415	0.991	1.6763	0.9972	0.1350
	60	196.84	0.0008	0.9948	34.197	706.94	0.0405	0.992	1.6827	0.9963	0.1470
150	**30**	265.16	0.0005	0.9959	35.444	337.47	0.0258	0.992	1.5711	0.9982	0.1657
	40	269.44	0.0005	0.9981	39.554	484.78	0.0267	0.991	1.6023	0.9972	0.1657
	50	277.18	0.0005	0.9952	42.339	576.72	0.0264	0.994	1.6242	0.9959	0.1504
	60	281.83	0.0004	0.9983	35.679	753.52	0.0282	0.995	1.6373	0.9968	0.1380
200	**30**	319.30	0.0003	0.9960	36.122	217.78	0.0211	0.997	1.4843	0.9982	0.2020
	40	326.21	0.0003	0.9940	39.161	272.73	0.0214	0.999	1.5124	0.9948	0.1931
	50	336.04	0.0003	0.9946	41.623	319.85	0.0202	0.998	1.5365	0.9961	0.1768
	60	343.14	0.0004	0.9988	47.269	452.48	0.0207	0.997	1.5720	0.9987	0.1763
250	**30**	366.06	0.0002	0.9954	35.936	157.46	0.0162	0.998	1.3958	0.9967	0.2150
	40	375.62	0.0002	0.9990	38.225	179.66	0.0161	0.992	1.4195	0.9989	0.2202
	50	391.17	0.0002	0.9952	38.051	170.04	0.0153	0.994	1.4253	0.9984	0.2140
	60	397.48	0.0002	0.9983	43.032	228.25	0.0157	0.991	1.4642	0.9967	0.2100

Conclusion

The present study has shown the effectiveness of using ANSC in the removal of Rhodamine B dye from aqueous solutions. Activated Acacia Nilotica Shell in different forms has a great role in modern life to clean environment. Acacia Nilotica Shell can be good precursors for producing highly porous Activated Acacia Nilotica Shell by simple preparative methods. An adsorption test has been carried out for industrial pollutants (Rhodamine B (Rh-B)) under different experimental conditions in batch mode. The adsorption of Rhodamine B was dependent on adsorbent surface characteristics, adsorbent dose, Rhodamine B concentration, time of contact and temperature. A study of the kinetic models on sorption showed that sorption fitted the pseudo second- order kinetics model. The ΔG, ΔH, and ΔS reveal the favorability of adsorption. The thermodynamic parameters suggested that the adsorption on ANSC was a spontaneous and endothermic process.

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