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RESEARCH ARTICLE

ADSORPTION OF RHODAMINE B FROM AN AQUEOUS SOLUTION: KINETIC AND THERMODYNAMIC STUDIES

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Abstract

The Acid Activated Vitex Negundo Stem was used as a potential low cost and available adsorbent for the removal of dyes from waste water. Adsorption studies were carried out in a batch process with initial dye concentration, contact time, adsorbent dose and effect of temperature. The equilibrium data satisfied both Langmuir and Freundlich isotherm models. The experimental data were analyzed using the pseudo –second order kinetic model. The changes in standard free energy(ΔG^o), standard entropy(ΔS^o), standard enthalpy (ΔH^o) were calculated. The thermodynamic study has showed that the dye adsorption phenomenon onto AAVNS was favourable, endothermic and spontaneous.
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Keywords: Acid Activated Vitex Negundo Stem (AAVNS), Rhodamine B, Adsorption isotherm, kinetics, Equilibrium models.

Introduction

Synthetic dyes are used extensively in many industries including dyehouses, paper printers and textile dyers. A significant proportion of synthetic dyes are lost annually to waste streams during textile processing, which eventually enters the environment [1]. Some of dyes are toxic and carcinogenic and require separation and advanced treatment of textile effluents before discharge into conventional systems [2]. Due to biodegradability of dyes, a conventional biological wastewater treatment process is not very efficient in treating dye wastewater. The removal of colour from wastewater can be accomplished by flotation, chemical coagulation, chemical oxidation and adsorption [3]. Hence, investigations have been conducted on physicochemical methods of removing colour from textile effluents [4].

These studies include chemical oxidation [5], membrane filtration [6]and adsorption techniques [7]. In these techniques, adsorption has been found to be an efficient process to remove dye. Activated carbon [8] and natural adsorbents such as banana and orange peel [9], apple pomace and wheat straw [10], waste mud [11] and wood materials [12] have been extensively used as adsorbents. Activated carbon adsorption has been found to be an effective means of waste water treatment.

Adsorption hold promise in the treatment of wastewater, as it is inexpensive, simply designed, easy to handle. The aim of this study was to use the dried acid activated vitex negundo stem (*AAVNS*) to remove one important reactive dye, Rhodamine-B (Rh-B) from aqueous solution.

Experimental procedure

Preparation of absorbent

Dried vitex negundo stem was carbonized with concentrated sulphuric acid in the weight ratio of 1:1 (w/v). Heating for 12 h in a furnace at 500^o C completed the carbonized and activation. The resulting carbon was washed with distilled water until a constant pH of the slurry was reached. Then the carbon was activated around 500^0 C in a muffle furnace for 5hrs. The dried material was ground well to a fine powder and stored in vacuum desiccators.

Adsorption experiment

The adsorption experiments were out in a batch process at 30, 40, 50, and 60^oc. A know weight of adsorbent was added to 50mL of dye solutions with an initial concentration of 50-250mg/L. The contents were shaken thoroughly using a mechanical shaken rotating at 150rpm. The solution was then filtered at preset time intervals and the residual dye concentration was measured.

Effect of adsorbent dosage

Various doses of the adsorbent were mixed with dye solution and the mixture was agitated in a mechanical shaker. The adsorption capacities for different doses were determined at define time intervals by keeping all other factors constant.

Effect of initial concentration of dye

In order to determine the rate of adsorption, experiments were conducted with different initial concentration of dyes ranging from 50-250mg/L. All other factors were kept constant.

Effect of contact time

The Effect of period of contact on the removal of the dye on adsorbent in a single cycle was determined by keeping particle size, initial concentration, dosage, *p*H and concentration of other ions constant.

Effect of *p*H

Adsorption experiments were carried out at *p*H 5-10. The acidic and alkaline *p*H of the media was maintained by adding the required amount of dilute HCL and NaOH. All other factors were kept constant while carrying out the experiment. The *p*H of the sample was determined using a portable *p*H meter.

Effect of temperature

The adsorption experiments were performed at four different temperatures viz., 30, 40, 50, and 60^oC in a thermostat attached with a shaker. The constancy of the temperature was maintained with an accuracy of $\pm 0.5^OC$.

Zero point charge

The *p*H at the potential of zero charge of the carbon (pH_{zpc}) was measured using the *p*H drift method. The *p*H of the solution was adjusted by using 0.01 M NaOH or HCL. Nitrogen was bubbled through the solution at 25^oC to remove the dissolved carbon dioxide. 50 mg of the activated carbon was added to 50 ml of the solution. After equilibration, the final *p*H was recorded. The graphs of final *p*H versus initial *p*H were used to determine the zero point charge of the activated carbon.

Desorption studies

Desorption studies help to elucidate the nature of adsorption and recycling of the spent adsorbent and the dye. The effectiveness of various reagent used for desorption were studied.

Results and Discussion

Effect of contact time and initial dye concentration

The results of variation of adsorption of RDB with contact time are show in fig .1. The equilibrium data collected in table 1 reveals that, percent adsorption decreased with increase in initial dye concentration, but the actual amount of dye adsorbed per unit mass of carbon increase in dye concentration. It means that the adsorption is highly dependent on initial concentration of dye. It is because of the fact that 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 becomes fewer and hence the percentage removal of dye dependents upon initial concentration [13] , Equilibrium have established at 60 min for all concentration. Figure 1 reveals that the curves are single, smooth, and continuous, leading to saturation, suggesting the possible monolayer coverage of the dyes on the carbon surface.

Effect of adsorbent dosage

The adsorption of the dyes on carbon was studied by varying the carbon concentration (50mg/250mL) for 50mg/L of dye concentration. The percent adsorption increased with increase in the carbon concentration Fig. 2. This was attributed to increased carbon surface area and availability of more adsorption sites [14]. Hence the remaining parts of the experiments were carried out with the adsorbent dose of 25mg/50mL.

Effect of 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 5.0 to 10.0 by adding acid and base to the stock solution This increases may be due to the presence of negative charge on the surface of the adsorbent Acid Activated Vitex Negundo Stem 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 Acid Activated Vitex Negundo Stem, 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 >6.8 may be due to occupation of the adsorption sites by OH^- ions which retard the approach of such dye further toward the adsorbent Acid Activated Vitex Negundo Stem surface. From the experimental results, the optimum pH range for the adsorption of the Rh-B dye is 5.0 to 6.7 shown in Figure.3.

Adsorption isotherm

The experimental dada were analyzed according to the liner from of the Langmuir and freundlich isotherms [15,16]. The Langmuir isotherm is represented by the following equation

$$C_e/Q_e = 1/ Q_m b + C_e/Q_m$$

Where C_e is the equilibrium concentration (mg/L), Q_e is the amount adsorbed equilibrium (mg/g) and Q_m and b are Langmuir constants related to adsorption efficiency and energy of adsorption, respectively. The liner plots of C_e/Q_e versus C_e suggest the applicability of the Langmuir isotherms. The values of Q_m and *b* were determined from slope and intercepts of the plots and are presented in table 2. From the results, it is clear that the value of adsorption efficiency Q_m and adsorption energy *b* of the carbon increases on increasing the temperature. From the values it is concluded that the maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on adsorbent surface with constant energy and no transmission of adsorbate in the plane of the adsorbent surface. The observed *b* value shows that the adsorbent prefers to bind acidic ions and that speciation predominates on sorbent charecterstics, when ion exchange as the predominant mechanism takes place in the adsorption of RDB; it confirms the endothermic nature of the process involved in the system [17,18]. To confirm the favorability of the adsorption process, the separation factor (RL) was calculated Table 3 the values were found to be between 0 and 1 which that the ongoing adsorption process is favorable[15].

Freundlich equation was employed for the adsorption of Rhodamine B dye on the adsorbent. The freundlich isotherm was represented by

$$\text{Log } Qe = \log Kf + 1/n \log Ce$$

Where Qe is the amount of Rhodamine B dye adsorbed (mg/g), Ce is the equilibrium concentration of dye in solution (mg/L) and Kf and n are constants incorporating the factors affecting the adsorption capacity and intensity of adsorption, respectively. Linear plots of log Qe versus log Ce show that the adsorption Rhodamine B dye obeys the Freundlich adsorption isotherm. The values of Kf and n given in table 2 show the increase in negative charges on the adsorbent surface that makes electrostatic force like Vanderwaal's in to play between the carbon surface and dye ion, which increases the adsorption of RDB. The molecular weight, size and radii either limit or increase the possibility of the adsorption of the dye onto adsorbent. However, the values clearly show the dominance in adsorption capacity of adsorbent to adsorb RDB on the surface. The intensity of adsorption is an indication of the bond energies between dye and adsorbent, the values show that the possibility of chemisorptions is rather less than physisorption[19]. The values of n are greater than one indicating the adsorption is much more favourable[20].

Effect of Temperature

The adsorption capacity of the carbon increased with increase in the temperature of the system from 30-60 ^{0}C Thermodynamic parameters such as change in free energy (ΔG^0) (Kj/mol), enthalpy (ΔH^0) (k j/mol) and entropy (ΔS^0) (K j/mol) were determined using the following equations.

$$K_o = C_{solid} / C_{liquid}$$
$$\Delta G^0 = -RT \ln K_o$$
$$\log K_o = \Delta S^o / (2.303RT) - \Delta H^o / (2.303RT)$$

Where Ko 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^o and ΔS^o values obtained from the slope and intercept of Van't Hoff plots are presented in Table 4. From the results it is clear that physisorption is much more favourable for the adsorption of RDB. The positive value of ΔH^o show the endothermic nature of adsorption and it governs the possibility of physical adsorption. Since in case of physical adsorption, while increasing the temperature of the system, the extent of dye adsorption increases, this rules out the possibility of chemisorptions [21]. The low of ΔH^o value suggest that the dye is physisorbed onto adsorbent TPC.
The negative values of ΔG^o Table 4 show that the adsorption is highly favorable and spontaneous. The positive values of ΔS^o Table 4 show the increased disorder and randomness at the solid solution interface of RDB with TPC adsorbent that bring out some structural changes in the dye and the adsorbent. The enhancement of adsorption capacity of the activated carbon at higher temperatures was attributed to the enlargement of pore size and activation of the adsorbent surface[20-23].

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 Acid Activated Vitex Negundo Stem were analyzed using pseudo second-order [23] Elovich[24] and intra-particle diffusion[19] 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

$$dq_t/d_t = k_2(q_e-q_t)^2 \ldots\ldots\ldots\ldots.(6)$$

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:

$$1/(q_e - q_t) = 1/q_e + K_2t \ldots\ldots\ldots\ldots\ldots\ldots (7)$$

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:

$$t/q_t=(1/k_2q_e^2)+ ((1/q_e)t \ldots\ldots\ldots\ldots\ldots\ldots(8)$$

If the initial adsorption rate (h)(mg $g^{-1}min^{-1}$) is :

$$h=k_2q_e^2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

Equation (8) and (9) becomes,

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

The plot of (t/q_t) and t of Eq. (10) gives 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 (5).At all studied initial Rh-B dye concentrations, the straight lines with extremely high correlation co-efficient (>0.99) were obtained. From table 5, the values of the rate constant k decrease with in increasing initial Rh-B dye concentration for Acid Activated Vitex Negundo Stem powder. This one shows that the sorption of Rh-B dye on Acid

Activated Vitex Negundo Stem follows pseudo second order kinetic model.

The Elovich equation

The Elovich model equation is generally expressed as

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

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 [24]. assumed $\alpha\beta t >> t$ and by applying boundary conditions $q_t = 0$ at t= 0 and $q_t = q_t$ at t = t Eq.(11) becomes:

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

If Rh-B dye adsorption fits with the Elovich model, a plot of q_t vs. ln(t) yields 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 5. 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 Acid Activated Vitex Negundo Stem adsorbent.

The intra particle diffusion model

The intra-particle diffusion model used here refers to the theory proposed by Weber and Morris [25] 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. The rate limiting step is intra-particle diffusion, since 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 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 the intra-particle diffusion takes place along with other process that may affect the adsorption. The values are given in table 5.

TABLE: 1. EQUILIBRIUM PARAMETERS FOR THE ADSORPTION OF Rh **B**

M_0	**Ce (Mg / L)**				**Qe (Mg / L)**				**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	3.80	3.63	3.73	3.00	92.39	92.72	92.53	93.98	92.39	92.72	92.53	93.98
100	14.26	12.36	10.73	9.08	171.47	175.27	178.53	181.83	85.73	87.63	89.26	90.91
150	30.36	27.64	23.09	20.68	239.26	244.70	253.80	258.62	79.75	81.56	84.60	86.20
200	58.62	54.09	23.09	44.70	282.74	291.81	353.80	310.58	70.68	72.95	88.45	77.64
250	90.67	85.68	49.06	74.68	318.65	328.62	401.87	350.63	63.73	65.72	80.37	70.12

TABLE: 2. LANGMUIR AND FREUNDLICH ISOTHERM PARAMETER FOR THE ADSORPTION OF Rh B

Temp. (°C)	**Langmuir Parameters**		**Freundlich Parameters**	
	Q_m	B	**K_f**	n
30°C	361.73	0.0703	1.7603	2.5411
40°C	373.39	0.0753	1.7743	2.4949
50°C	574.08	0.0464	1.6440	1.6932
60°C	395.68	0.0940	1.8278	2.4673

TABLE: 3. DIMENSIONLESS SEPERATION FACTOR (R_L) FOR THE ADSORPTION

(C_i)	**Temperature îC**			
	30^{o}C	**40^{o}C**	**50^{o}C**	**60^{o}C**
50	0.2214	0.2096	0.3011	0.1753
100	0.1244	0.1171	0.1772	0.0960
150	0.0865	0.0812	0.1256	0.0661
200	0.0663	0.0621	0.0972	0.0504
250	0.0538	0.0503	0.0793	0.0407

TABLE: 4. THERMODYNAMIC PARAMETER FOR THE ADSORPTION OF Rh B

(C_0)	**ΔG^o**				a**H**î	a**S**î
	30^{o}C	**40^{o}C**	**50^{o}C**	**60^{o}C**		
50	-6289.62	-6623.7	-6759.89	-7612.5	6.029	40.41
100	-4518.22	-5096.11	-5689.3	-6377.31	14.15	61.56
150	-3453.76	-3870.54	-4575.28	-5074.15	13.42	55.54
200	-2217.31	-2582.12	-5467.31	-3447.26	18.39	68.61
250	-1420.07	-1694.32	-3786.12	-2362.47	13.97	51.23

TABLE: 5. THE KINETIC PARAMETERS FOR ADSORPTION FOR THE ADSORPTION OF RhB

C_0	**Temp îC**	**Pseudo second order**				**Elovich model**			**Intraparticle diffusion**		
		q_e	**K_2**	**¥**	**H**	**~**		**¥**	**K_{id}**	**¥**	**C**
50	**30**	102.15	0.002182	0.9952	14.01	134.90	0.0694	0.9959	1.6461	0.994	0.1771
	40	100.41	0.001934	0.9983	16.28	427.67	0.0846	0.9968	1.7065	0.995	0.1416
	50	99.85	0.001898	0.9960	17.75	744.47	0.0913	0.9982	1.7284	0.997	0.1298
	60	101.59	0.001782	0.9940	17.50	401.61	0.0818	0.9948	1.7105	0.999	0.1446
100	**30**	188.47	0.002592	0.9946	26.66	297.98	0.0387	0.9961	1.6238	0.998	0.1705
	40	192.25	0.002474	0.9988	27.23	353.65	0.0391	0.9987	1.6417	0.997	0.1647
	50	195.11	0.002330	0.9954	29.74	442.24	0.0395	0.9967	1.6622	0.994	0.1590
	60	197.29	0.002122	0.9990	32.32	712.14	0.0418	0.9989	1.6921	0.995	0.1462
150	**30**	265.76	0.002869	0.9987	33.59	302.95	0.0263	0.9984	1.5714	0.997	0.1812
	40	269.83	0.002734	0.9967	38.25	412.92	0.0268	0.9967	1.6016	0.999	0.1718
	50	278.59	0.002604	0.9961	40.42	500.05	0.0267	0.9983	1.6260	0.998	0.1662
	60	281.20	0.002472	0.9981	34.63	673.53	0.0288	0.9943	1.6406	0.997	0.1520
200	**30**	319.81	0.003118	0.9975	34.36	200.22	0.0197	0.9982	1.4709	0.998	0.2079
	40	326.80	0.002998	0.9969	38.74	273.80	0.0202	0.9972	1.5100	0.992	0.1946
	50	338.38	0.001427	0.9973	39.64	267.73	0.0193	0.9969	1.5198	0.994	0.1973
	60	345.49	0.002899	0.9989	44.37	382.72	0.0200	0.9981	1.5588	0.991	0.1830
250	**30**	364.90	0.003149	0.9928	35.93	168.18	0.0163	0.9948	1.3953	0.992	0.2255
	40	377.59	0.003236	0.9941	36.36	164.50	0.0156	0.9994	1.4028	0.991	0.2288
	50	391.93	0.002028	0.9948	37.38	174.85	0.0152	0.9972	1.4219	0.992	0.2264
	60	397.03	0.003140	0.9959	42.08	228.70	0.0156	0.9963	1.4589	0.991	0.2129

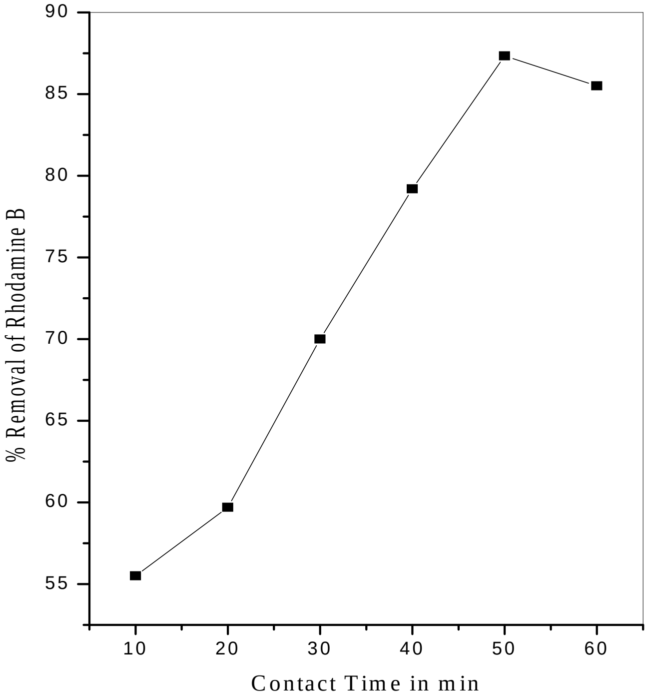
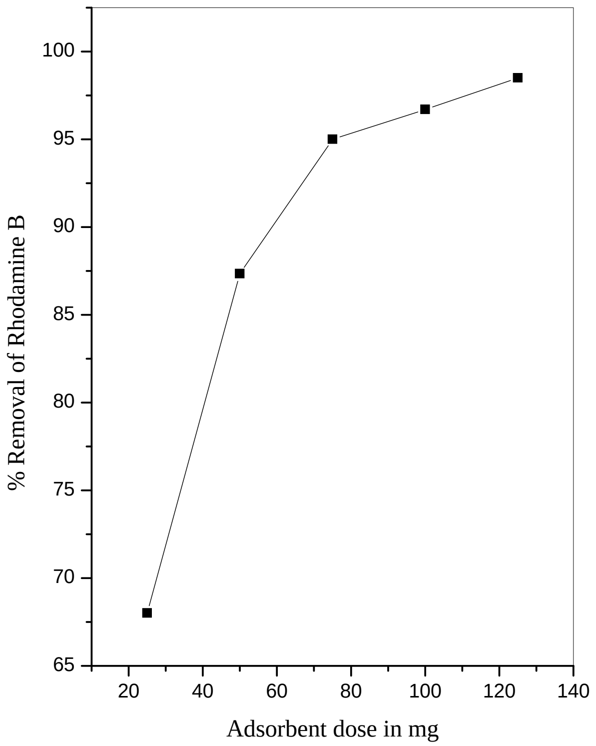
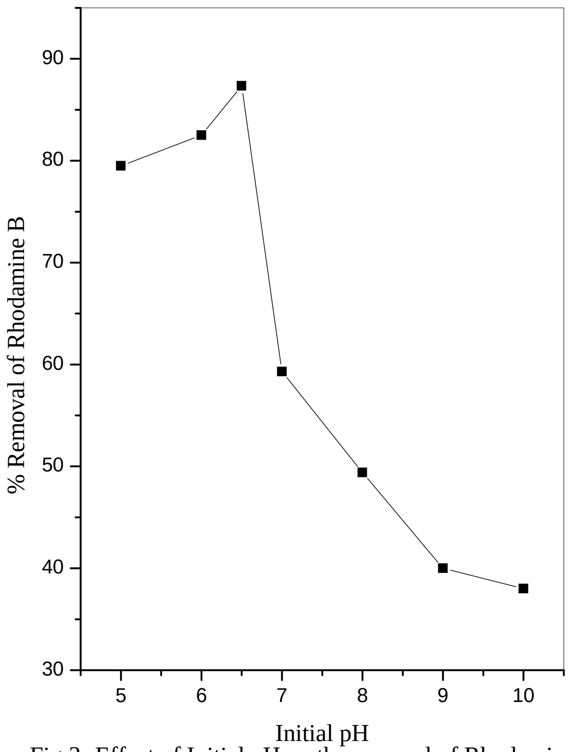


Fig:1- Effect of Contact Time on the Removal of Rhodamine B
[M]=50 mg/L;Temprature 30^{o}C;Adsorbent dose=25mg/50ml



Fig;2- Effect of Adsorbent dose on the removal ofRhodamine B
[M]=50mg/L;Contact Time 60min;Temprature 30^{0}C



Fig;3- Effect of Initial pH on the removal of Rhodamine B
[M]=50 mg/L;Temprature 30^{o}C;Adsorbent dose=25mg/50ml

Desorption studies

Desorption studies help to elucidate the nature of adsorption and recycling of the spent adsorbent and the dye. If the adsorbed dyes can be adsorbed using neutral pH water, then the attachment of the dye of the adsorbent is by weak bonds. If sulphuric acid or alkaline water desorp the dye then the adsorption is by ion exchange. If organic acids, like acetic acid can desorp the dye, then the dye has been held by the adsorbent through chemisorptions. The effect of various reagents used for desorption studies indicate that hydrochloric acid is a better reagent for desorption, because more than 90% removal of adsorbed dye is achieved. The reversibility of adsorbed dye in mineral acid or base is in agreement with the pH dependant results obtained. The desorption of dye by mineral acids and alkaline medium indicates that the dye is adsorbed onto the activated carbon through by physisorption mechanisms[20,26].

Conclusions

The experimental data correlated reasonably well by the Langmuir and freundlich adsorption isotherms and the isotherm parameters were calculated. The low as well as high pH value plays the way to the optimum amount of adsorption of the dye. The amount of Rhodamine B adsorbed increased with increasing ionic strength, and increasing temperature. The dimensionless separation factor (R_L) showed that the activated carbon could be used for the removal of Rhodamine B from aqueous solution of AAVNS. The values of ΔH^0, ΔS^0 and ΔG^0 show that the carbon employed has a considerable potential as an adsorbent for the removal of Rhodamine B

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