

Removal of Rhodamine B from Aqueous Solution by Using Pine Cone Activated with HNO₃

Meryem Gören^{1,*}, Hatice Begüm Murathan¹, Nihan Kaya², Atilla Mirati Murathan¹

¹Gazi University, Faculty of Engineering, Chemical Engineering Department, Ankara, Turkey; ²Hitit University, Faculty of Engineering, Chemical Engineering Department, Çorum, Turkey

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Abstract: In the experimental study, optimum conditions were determined for the adsorption of Rhodamine B dye, which is frequently used in the textile industry, on modified pine cone adsorbent. It was observed that the adsorption duration, solution pH, temperature and initial dye concentration affected the adsorption capacity of HNO₃ activated pine cone. The maximum dye removal efficiency was found to be 97% on pine cone pretreated with HNO₃ at T = 55 °C, pH = 3, C₀=10⁻⁴ M dye concentration. Experimental data were applied to pseudo first order and pseudo second order kinetic models and Langmuir and Freundlich adsorption isotherms for investigating rate and kinetics of adsorption process. Kinetic analyzes demonstrated that the experimental data were highly compatible with the pseudo second order kinetic model. Besides it was seen that the equilibrium values were represented by the Langmuir isotherm equivalence for the whole process. From the calculations made as a result of thermodynamic studies, (ΔH°) and (ΔS°) were found to be positive, while (ΔG°) was found to be negative. As a result, in this study, the applicability of pine cones, which is a cheap and natural material that can be used in the removal of dyestuffs from aqueous media, has been demonstrated.

Keywords: Adsorption, cationic dye, pine cone, Rhodamine B

Introduction

Dyes are widely utilized in many industries due to its bright colors, excellent stability and easy applicability. However, since the dyes remaining in the waste water after dyeing processes create water pollution, these substances must be removed from aqueous medium. But it is very difficult to remove the dyes with traditional treatment methods. Today, dyestuffs are largely removed by physical and chemical processes. Adsorption technique has much attracted attention in recent years due to its efficiency in removing over-stable pollutants. Activated carbon is a quite good adsorbent, but it is not economically viable (Lacerda *et al.*, 2015; Haddad *et al.*, 2012). Therefore, in recent years, researchs on the use of more economical adsorbents in the removal of dyestuffs from wastewater has been come to the fore (Murathan and Bütün, 2006; Orman *et al.*, 2015; Bilgin *et al.*, 2014).

Rhodamine B is one of the most main water-soluble organic dyes, widely used as a colorant in the textile and food industries and in biomedical laboratories. This dyestuff has a toxic character and carries a carcinogenic risk for health. It causes cough, sore throat and chest pain symptoms and respiratory disorders if inhaled. For this reason, it is of great importance of discharge the waste waters containing dyes in industrial applications to nature after being subjected to inexpensive and practical appropriate treatment processes (Zamouche and Hamdaoui, 2012a; Khan *et al.*, 2011; Jain *et al.*, 2007).

In this study, the efficiency of pine cone, a low-priced and natural adsorbent, in the removal of a basic dye, Rhodamine B (RhB), was investigated. Before starting the adsorption experiments, pine cone was activated with HNO₃. Initial dyestuff concentration, contact time, pH value and temperature are the factors affecting the adsorption process and the effects of each of them were investigated separately. Isotherm, kinetic and thermodynamic studies have been carried out in order to get an idea about the sorption mechanism of the sorbent.

Materials and Method

Materials

Rhodamine B dye used in this study was supplied from Sigma-Aldrich company. The molecular structure of Rhodamine B is shown in Figure 1 (Zamouche and Hamdaoui, 2012a). Pine cones which

* Corresponding: E-Mail: meryem.goren@gazi.edu.tr; Tel: +90 0312 582 3530; Fax: +90 0312 230 8434;

were obtained from Yaprak region in Ankara were used as adsorbent in experimental studies. The pine cones used in the study were washed with purified water before the adsorption process in order to remove the waste materials they contain, and after they were kept in an oven at 110 °C for 1 hour. They were dried and sieved. In order to improve the adsorption capacity of the pine cones, pine cones were activated with HNO₃ and used in adsorption experiments (Zamouche *et al.*, 2014).

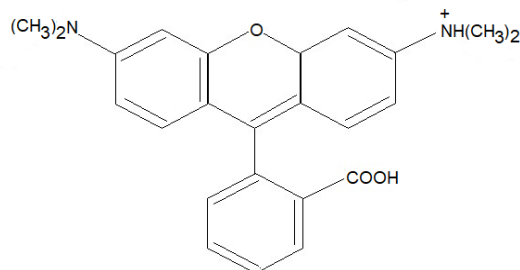


Figure 1. The molecular structure of Rhodamine B

Adsorption Studies

In adsorption experiments, dye solutions with initial concentrations of 10⁻⁵-10⁻⁴ M were prepared and 1 g adsorbent were used in 100 ml dye solution. Adsorption studies were performed using a batch technique in a shaking water bath in 250 mL conical flasks in the temperature range 25-55 °C and solution pH range 2-8. Samples were taken at 15-minute intervals and were filtered through filter paper. Their absorbance was measured with a UV-vis spectrometer at 554 nm wavelength. Their concentration was determined by using calibration graph. The adsorption capacity of activated pine cone was calculated as follows (Equation 1):

$$q_t = \frac{(C_o - C_t) \cdot V_t}{W} \quad (1)$$

where q_t is the adsorption capacity (mg dye/g solid) at time t , C_o is the initial dye concentration (mg/L), C_t are the final dye concentration (mg/L) at time t , V_t is the volume of solution (L) at time t , and W is the sorbent weight (g). In addition, the percent adsorption efficiencies were calculated with the help of the experimental data obtained.

Results and Discussions

Effect of pH

The pH of the solution where the adsorption takes place affects the adsorption process. It not only controls the surface charge of the adsorbent, but also affects the degree of ionization of the pollutant. In this study, dilute HCl and NaOH solutions were used to adjust the pH of the solutions. The effect of pH on the removal of Rhodamine B dye from aqueous solution by using activated pine cone is shown in Figure 2.

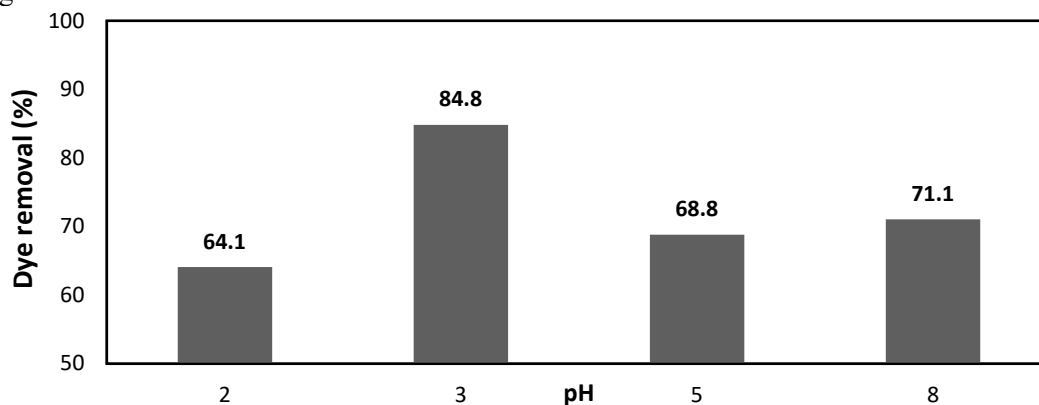


Figure 2. Effect of pH on the adsorption of Rhodamine B dye (dye concentration 1x10⁻⁴ M, adsorbent dosage 1 g, temperature 25 °C, volume of dye solution 100 ml)

The maximum percentage of dye removal was reached at pH 3 (84.8%). This result was attributed to the change in the molecular form of Rhodamine B dye at high pH values. In cases where the solution pH is greater than 3.0, considering that Rhodamine B dye has a greater molecular form, dye molecules becomes difficult to disperse into the pores on the adsorbent surface and thus the adsorption efficiency decreases (Zamouche and Hamdaoui, 2012a; Thakur and Kaur, 2016).

Effect of Initial Dye Concentration

Figure 3 and Table 1 shows the adsorption of Rhodamine B at different initial dye concentrations (10^{-5} - 10^{-4} M) on pine cone pretreated with HNO_3 .

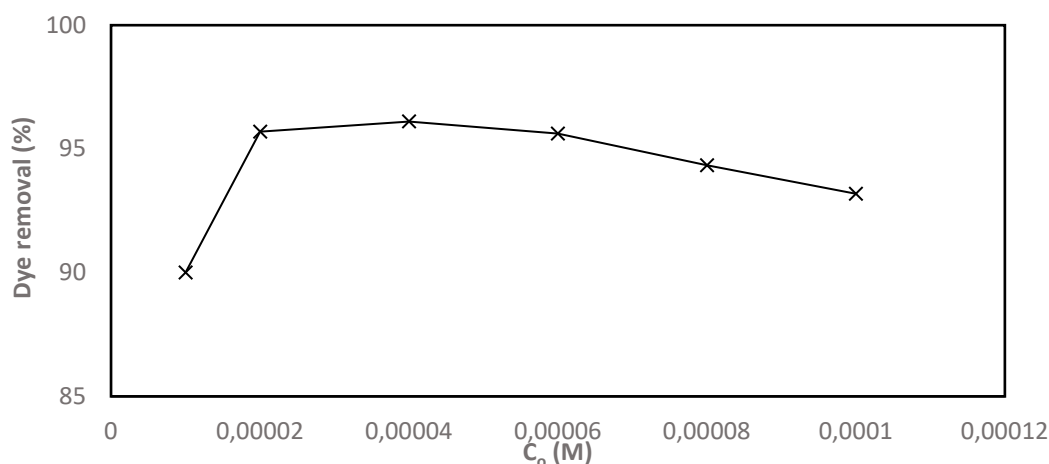


Figure 3. Effect of initial dye concentration on adsorption of Rhodamine B dye (adsorbent dosage 1 g, pH = 3, temperature 25 °C, volume of dye solution 100 ml)

Table 1. Effect of initial concentration on dye removal efficiency

C_0 (M)	Dye removal (%)
1×10^{-5}	90.0
2×10^{-5}	95.7
4×10^{-5}	96.1
6×10^{-5}	95.6
8×10^{-5}	94.3
1×10^{-4}	93.2

As seen in Figure 3, the dye removal efficiency increased from 90% to 93.2% with the increase of dye concentration from 10^{-5} M to 10^{-4} M (Table 1). The increase in percent dye uptake can be associated with the increase in driving force depending on initial dye concentration (Zamouche and Hamdaoui, 2012b). The reason why the removal efficiency decreases slightly after concentration values of 4×10^{-5} M is due to the decrease interaction of the dye molecules with the limited number of empty active sites on the adsorbent surface (Khan *et al.*, 2011).

Effect of Contact Time

One of the most important factors affecting the rate and adsorbed amount in adsorption studies is the contact time. The effect of contact time on the adsorption of Rhodamine B was determined using 10^{-4} M dye solution at 25 °C and pH 3. Dye removal increased with increasing contact time (Figure 4), and after a while, the dye concentration remaining in the aqueous solution reached a constant value where a dynamic equilibrium was established. The optimal time for Rhodamine B adsorption was 60 minutes. Considering the relationship between adsorption and contact time, there was an increase in the adsorbed amount as expected as a result of the high active sites available at the beginning. As the time progresses, the adsorption rate started to decrease due to the filling of active pores. Upon reaching the saturation value, adsorption only took place inside the pores of the adsorbent. However, there was no significant increase in adsorption due to the smaller inner surface area of pores (Zamouche and Hamdaoui, 2012a; Khan *et al.*, 2011).

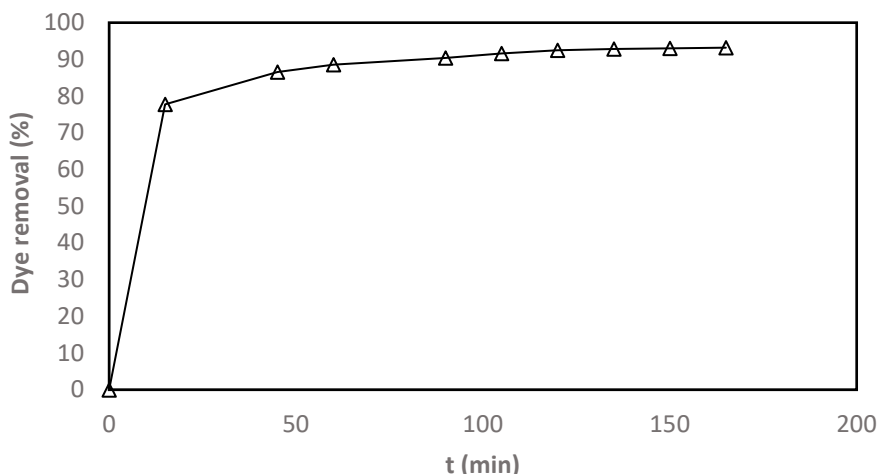


Figure 4. Effect of contact time on adsorption of Rhodamine B dye (dye concentration 1×10^{-4} M, adsorbent dosage 1 g, pH = 3, temperature 25 °C, volume of dye solution 100 ml)

Effect of Temperature

In order to determine the effect of temperature on the adsorption of Rhodamine B on the pine cone activated with HNO_3 , four different temperatures (25 °C-55 °C) were studied and the obtained removal efficiencies are shown in Figure 5. It is clearly seen from the experimental results that the dye removal increased as the temperature increases. The higher temperature facilitated the adsorption of Rhodamine B on activated pine cone. This was probably due to the increased in the mobility of dye molecules with the increase in temperature (Vijayakumar, 2012).

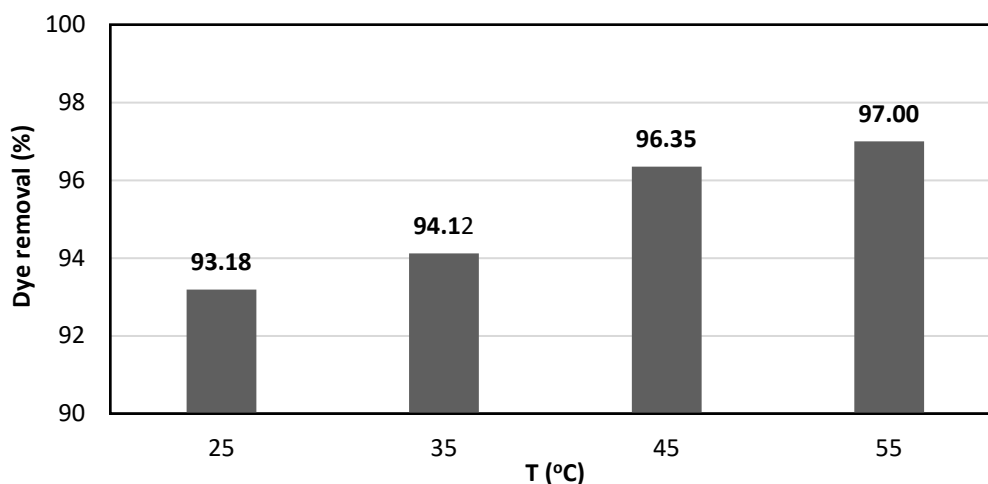


Figure 5. Dye removal efficiencies at different temperatures (dye concentration 1×10^{-4} M, adsorbent dosage 1 g, pH = 3, volume of dye solution 100 ml)

Adsorption Isotherms

Adsorption is an equilibrium phenomenon. Adsorption isotherms are very important for the design of adsorption systems. In general, adsorption isotherms describe how the adsorbent interacts with the substance to be adsorbed and are therefore necessary for the optimization of adsorbents. These isotherms are plotted using experimental data of adsorption at different temperatures. Langmuir, Freundlich, Temkin and Dubinin-Radushkevich were often used to evaluate experimental isotherm data in literature (Motahari *et al.*, 2015; Santhi *et al.*, 2014).

Langmuir isotherm

Langmuir isotherm theory assumes a single layer adsorbate coverage on a homogeneous adsorbent surface and the isotherm values are calculated from Equation 2.

$$\frac{1}{q} = \frac{1}{q_0} + \left(\frac{1}{K_L q_0}\right)\left(\frac{1}{C}\right) \quad (2)$$

Here; q_0 is maximum monolayer adsorption capacity of adsorbent, K_L is the Langmuir isotherm constant. When $1/q$ versus $1/C$ values are plotted (Figure 6a), q_0 and K_L are found from the slope and intercept values of the resulting graph (Zamouche and Hamdaoui, 2012b; Aksu *et al.*, 2011).

Freundlich isotherm

Freundlich isotherm describes multilayer adsorption on multiple surfaces and the linearized Freundlich isotherm equation can be shown as follows (Equation 3):

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (3)$$

Here; C_e is the dye concentration remaining unadsorbed in solution at equilibrium (mg/L), q_e is the amount of dye adsorbed on the unit adsorbent at equilibrium (mg/g), K_f is the constant showing the strength of the relationship between adsorbed and adsorbent (L/g), n is an expression of the adsorption intensity.

By plotting the variation of $\ln q_e$ versus $\ln C_e$ (Figure 6b), the point where the line obtained from the graph intersects the y-axis gives $\ln K_f$ and its slope gives $1/n$. The value of $1/n$ varies between 0 and 1, and the value of $1/n$ close to 0 indicates that the heterogeneity of the surface is higher. The accuracy of this isotherm is better than Langmuir isotherm in heterogeneous adsorption systems (Khan *et al.*, 2011).

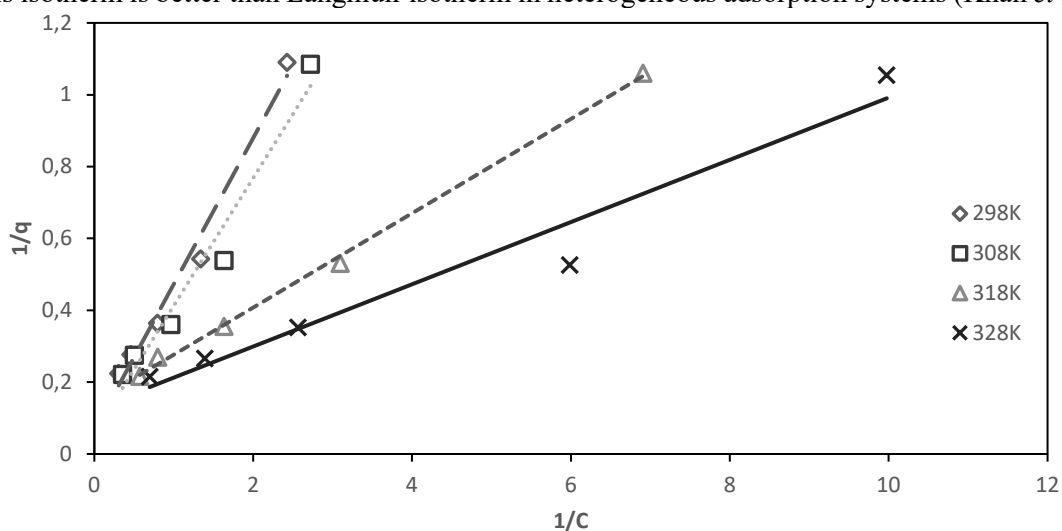


Figure 6a. Langmuir adsorption isotherms for Rhodamine B removal

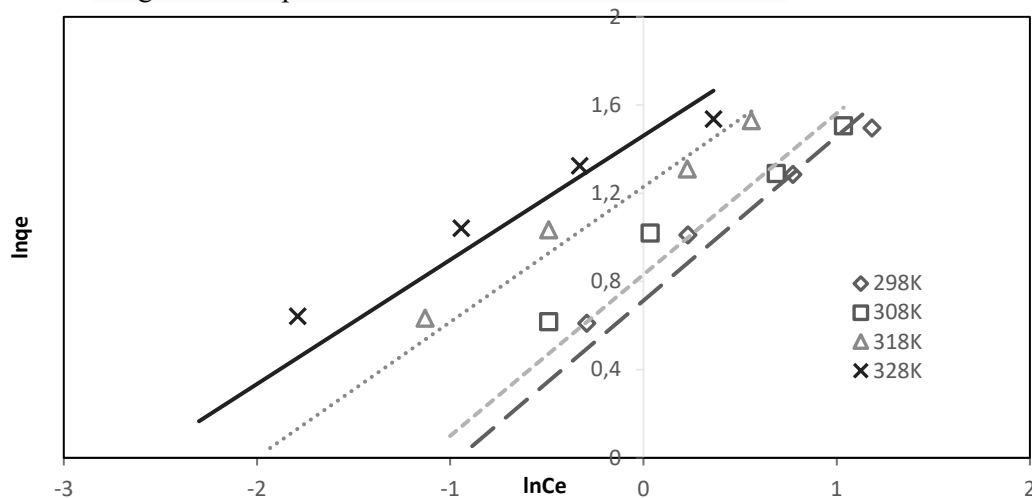


Figure 6b. Freundlich adsorption isotherms for Rhodamine B removal

When the data in Table 2 are examined, it is seen that the adsorption process was more suitable for the Langmuir isotherm model according to the correlation coefficients (R^2) values. R^2 value close to 1 indicated the compatibility of the process with the isotherm.

Table 2. Isotherm constants

T (K)	Langmuir isotherm			Freundlich isotherm		
	q_o (mg/g)	K_L (L/mg)	R^2	n	K_F (mg/g) (L/mg) ^{1/n}	R^2
298	15.0	0.16	0.983	1.34	5.2	0.962
308	17.4	0.16	0.963	1.37	6.7	0.943
318	6.8	1.12	0.998	1.63	17.0	0.977
328	7.9	1.46	0.958	1.77	29.0	0.927

Adsorption Kinetics

Kinetics is an important step in understanding the adsorption steps that affect the rate of the adsorption process. In order to design an effective and useful adsorption model, it is necessary to evaluate the adsorption process from a kinetic point of view. For the kinetic calculations of the liquid phase Rhodamine B dye on adsorbent prepared from pine cones, the concentration ranges of $10^{-5}M$ - $10^{-4}M$ were studied under the appropriate conditions. Rhodamine B dye adsorption kinetics on activated pine cone has been studied using two different models. The pseudo first order and pseudo second order kinetic models are the most widely used among the various kinetic models proposed to determine what kind of mechanism plays a role during the adsorption of the adsorbate onto the adsorbent surface.

Pseudo first order kinetic model

This model equation was first proposed by Lagergren, developed according to the basic approach of interface kinetics, and the differential form of the equation is shown in Equation 4.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (4)$$

Here, q_e is the equilibrium adsorption capacity, q_t is adsorption capacity at time t , k_1 is the adsorption rate constant (min^{-1}), t is the contact time. The rate constant k_1 can be obtained from the slope of the plot of $\log (q_e - q_t)$ versus t . The theoretical q_e value is calculated from the intercept. The first order rate kinetics of the activated pine cone is given in Figure 7a (Bozkan, 2012).

Pseudo second order kinetic model

This model rate equation is given by Ho as in Equation 5.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

Here, q_e is the equilibrium adsorption capacity (mg/g), q_t is adsorption capacity at time t (mg/g), k_2 is the rate constant ($\text{g/mg}\cdot\text{min}$). The values q_e and k_2 are determined from the slope and the intercept of the plot t/q_t against t . The graphs drawn with the help of this model are given in Figure 7b (Bozkan, 2012).

When the results of the relevant calculations and the correlation coefficients in Table 3 were evaluated, it was accepted that the kinetic model was compatible with the pseudo second order kinetic model for the adsorption of Rhodamine B dye on activated pine cone.

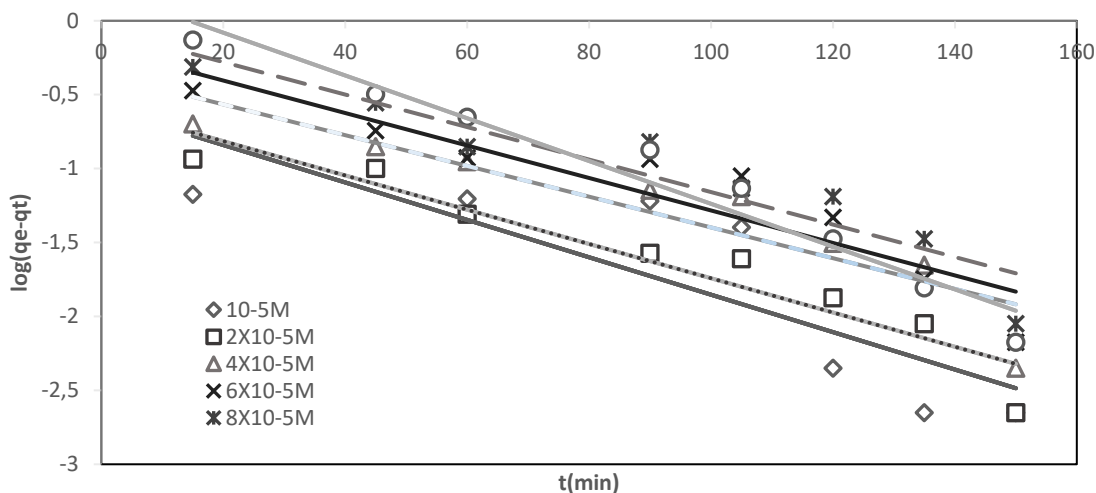


Figure 7a. Pseudo first order kinetics curves obtained at varied concentration values

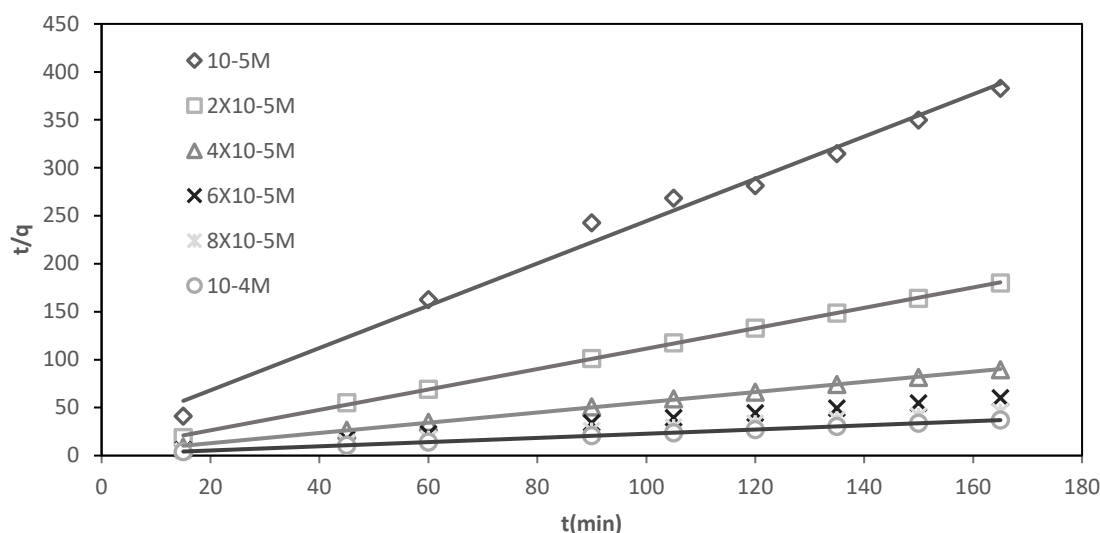


Figure 7b. Pseudo second order kinetics curves obtained at varied concentration values

Table 3. Kinetic values for Rhodamine B adsorption on activated pine cone

T (K)	Pseudo first order kinetic model		Pseudo second order kinetic model	
	k_1 (1/min)	R^2	k_2 (g/mg.min)	R^2
298	0.033	0.955	0.050	0.999
308	0.020	0.983	0.048	0.999
318	0.026	0.933	0.065	0.999
328	0.019	0.987	0.070	0.999

Adsorption Thermodynamics

Adsorption thermodynamics has been studied to get an idea on adsorption behavior. For the adsorption process, ΔH° (enthalpy change), ΔS° (entropy change) and ΔG° (Standard free energy change) values, which are important thermodynamic quantities, were calculated with the help of adsorption data (Ding *et al.*, 2014). These parameters were calculated using the Equation 6, 7 and 8:

$$\Delta G^o = -RT \ln K_C \quad (6)$$

$$K_C = \frac{C_a}{C_e} \quad (7)$$

$$\ln K_C = \frac{-\Delta H^o}{R T} + \frac{\Delta S^o}{R} \quad (8)$$

When plotted $\ln K_c$ against $1/T$, the slope of the line and the point where it intersects the y-axis give $-\Delta H^o/R$ and $\Delta S^o/R$, respectively. Here, K_C is the equilibrium constant, C_a is the adsorbed dye concentration in equilibrium (mol/L); C_e is the remained dye concentration in solution in equilibrium (mol/L) (Ding *et al.*, 2014).

Table 4. Thermodynamic values calculated for Rhodamine B adsorption on activated pine cone

C_o (mol/L)	T (K)	K_c	ΔG^o ($\frac{kJ}{mol}$)	ΔH^o ($\frac{kJ}{mol}$)	ΔS^o ($\frac{J}{mol K}$)
1x10 ⁻⁵	298	2.19	-5.42	49.5	182.2
	308	2.30	-5.89		
	318	2.78	-7.35		
	328	4.09	-11.15		
2x10 ⁻⁵	298	3.10	-7.68	42.9	168.5
	308	3.22	-8.24		
	318	4.17	-11.02		
	328	4.55	-12.40		
4x10 ⁻⁵	298	3.20	-7.93	42.7	168.7
	308	3.40	-8.71		
	318	4.10	-10.84		
	328	4.73	-12.89		
6x10 ⁻⁵	298	3.08	-7.63	33.4	137.2
	308	3.28	-8.39		
	318	3.82	-10.10		
	328	4.28	-11.67		
8x10 ⁻⁵	298	2.81	-6.96	31.4	127.6
	308	2.90	-7.42		
	318	3.38	-8.93		
	328	3.95	-10.70		
1x10 ⁻⁴	298	2.62	-6.49	24.98	105
	308	2.77	-7.09		
	318	3.27	-8.64		
	328	3.48	-9.48		

Thermodynamic studies were performed at the specified temperatures and concentrations for Rhodamine B adsorption on pine cone activated with HNO₃ and the results are given in Table 4. At all temperatures, Gibbs free energy (ΔG^o) values gave a negative value, confirming that the adsorption of Rhodamine B dye was spontaneous and thermodynamically favorable. The positive values of ΔH^o

indicated the endothermic nature of its adsorption. Initial concentrations vary from 10^{-5} M to 10^{-4} M, while ΔH° values were between 49.5-24.98 kJ/mol. The positive ΔS° values (182.2-105 J/mol.K) indicated increased irregularity (Zamouche and Hamdaoui, 2012b; Tasmakıran, 2010).

Conclusions

This experimental study was accomplished to investigate the use of pine cones, which is a natural waste, as a potential adsorbent in the removal of Rhodamine B dye from aqueous solution. The maximum dye removal was found to be 97% on activated pine cone with a size of 355-710 μm at $T = 55^\circ\text{C}$, $\text{pH} = 3$, 10^{-4} M initial dye concentration. According to the R^2 correlation coefficient values calculated by applying the obtained results to the kinetic models, the pseudo second order kinetic model was determined as the kinetic model that best explains adsorption. It was found that the adsorption process was best explained with the Langmuir isotherm model based on the graphs obtained by applying all the experimentally obtained data to the isotherm equations. Gibbs free energy change (ΔG°) was negative. The fact that the Gibbs free energy was negative indicated that the adsorption process can be occurred spontaneously under normal conditions. Positive ΔH° values indicated that the adsorption process was endothermic.

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