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Research Article

Influence of Ultrasound on Adsorption of Direct Red 81 Dye onto Low Cost Activated Carbon: Kinetic and Thermodynamic Studies

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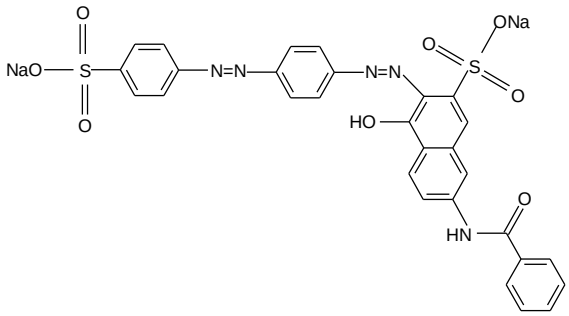
Abstract In this study, the potential of low cost activated carbon obtained from *Hibiscus Sabdariffa* stem as biosorbent was investigated for the adsorption of Direct red 81 (DR 81) from aqueous solution. The effect of parameters such as temperature, pH, adsorbent dose, contact time, initial dye concentration and ultrasonic irradiation were evaluated. Also, experimental equilibrium and kinetic data were fitted by Langmuir and Freundlich isotherms and pseudo-first-order and pseudo-second-order kinetic models, respectively. The results obtained from kinetic experiments showed that the adsorption of DR onto activated carbon can be described with pseudo second-order model and the equilibrium isotherm results were described by the Langmuir model. The negative values of thermodynamic parameters of ΔH^o and ΔG^o showed that the adsorption process is an endothermic and occurs spontaneously. The overall results showed that the synthesized activated carbon can be an efficient adsorbent due to its low cost and high efficiency.

Keywords Biosorption, low cost activated carbon, Thermodynamic, Kinetic models, Ultrasonic irradiation

Introduction

Textile wastewater contains many types of dye molecules that are toxic to microorganisms and human beings [1]. Therefore, the removal of effluents containing dyes has been a challenging problem in the environmental technologies and it has always been necessary to discover efficient dye-removal methods [2,3]. Several methods have been reported for efficient removal of dyes from industrial wastewater, including chemical precipitation [4], adsorption [5], electro-coagulation [6], biosorption [7], electro-deposition [8] and membrane systems [9,10].The conventional methods for treatment of the wastewater usually suffer from some disadvantages, such as complicated procedures, formation of by -products, expensive and energy-intensive and limited versatility [11]. Recently, there has been a special interest to low -cost, easily obtainable, highly efficient, and environmentally benign alternatives to the current methods [12,13]. In this regard, biosorption may be used as one of the efficient method for removal of substances from solution by biological organic or inorganic material [14,15]. The major advantages of biosorption system are good removal performance, less investment in terms of initial cost, flexibility, simplicity, easiness of operation and insensitivity to toxic pollutants as compared with the conventional treatment process [16,17]. Recently, various natural materials have been used for the treatment of wastewaters such as banana pith [18], rice husk [19], papaya seeds [20], orange peel, banana peel [21], *Caulerpa racemosa* var. cylindracea [22], *Caulerpa lentillifera* [23], *Posidonia oceanica* [24], Camel Thorn Plant [25] and Chamomilla Plant [26].The present study showed the adsorptive removal of Direct red 81 (DR 81) (Scheme.1) by activated carbon prepared from *Hibiscus Sabdariffa* stem by as natural source. Also, to obtain a better understanding of the biosorption mechanisms, the kinetic, isotherm and thermodynamic of dye removal were studied. Furthermore, the effect of parameters such as temperature, pH, initial dye concentration, biosorbent dosage, contact time and ultrasonic irradiation were investigated on dye removal efficiency. Direct red was selected in this study as an azo based dye model, because it has many applications in various industries.



Scheme 1: The chemical structure of Direct Red 81

Materials and Instruments

All the analytical chemicals and DR dye were purchased from Merck and used without further purification. The UV-Vis absorption spectra were recorded using a Shimadzu UV-2550 spectrophotometer. The DR concentration was determined by using UV-Vis spectrophotometer at maximum wavelength of DR (λ_{max} = 510 nm).

Preparation of biosorbent

Hibiscus Sabdariffa plant was collected from local area of Sistani and Baluchistan in Iran. In this procedure, *Hibiscus Sabdariffa plant* was carbonized with concentrated sulphuric acid at 120 °C for 1 hour. Then the resultant carbon was washed with distilled water and dried at 110 °C and was soaked in 5% sodium bicarbonate solution to remove any residual acid. In order to eliminate surface groups by thermal activation, the carbonized material was treated at 1100 °C for 5 hours in furnace. The physicochemical characteristics of the adsorbent were determined using standard procedure and reported in Table.1.

Table 1: Physicochemical characteristics of adsorbent

Analysis	Value
Specific gravity	0.93
Moisture content (%)	0.07
Bulk density (g/cm^3)	0.186
Particle density (g/cm^3)	0.25
Conductivity (μs/cm)	36.2
Surface area (m^2/g)	20.2
Na^+ (mg/L)	95.2
K^+ (mg/L)	420.3
pH_{zpc}	4.5

Adsorption Experiment

The adsorption behavior of the activated carbon was evaluated by removal of DR dye solution. The adsorption experiments were carried out under the same conditions (85 mgL^{-1} concentration of DR dye solution, 1.5 g adsorbent dosage and t=25 °C). After stirring, the solution was centrifuged and the dye concentration in the supernatant was calculated by measuring absorbance at λ_{max} = 510 nm. The disappearance of peak at λ = 510 nm was chosen for monitoring of DR adsorption. The adsorption capacity q_e (mg/g) and percentage adsorbed were calculated from equation (1) and (2), respectively:

$q_e = [(C_\circ - C_e)/V/m]$ (1)

$\% R = [(C_\circ - C_e)/C_\circ] \times 100$ (2)

Where $C_\circ$ and $C_e$are the initial concentration and the concentration of dye at time t, respectively. V volume of the solution and m the mass of adsorbent used.

Results and Discussion

The effect of contact time

The effect of contact time on the amount of DR dye was studied at 50 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 and beyond

contact time of 40 min no obvious change in the percentage removal is observed. Fig.1 showed the percentage removal after 40 min was %98. So, the optimum contact time is considered to be 40 min. Also, the rapid removal of DR dye is observed at the beginning of the contact time due to the large number of binding sites available for adsorption.

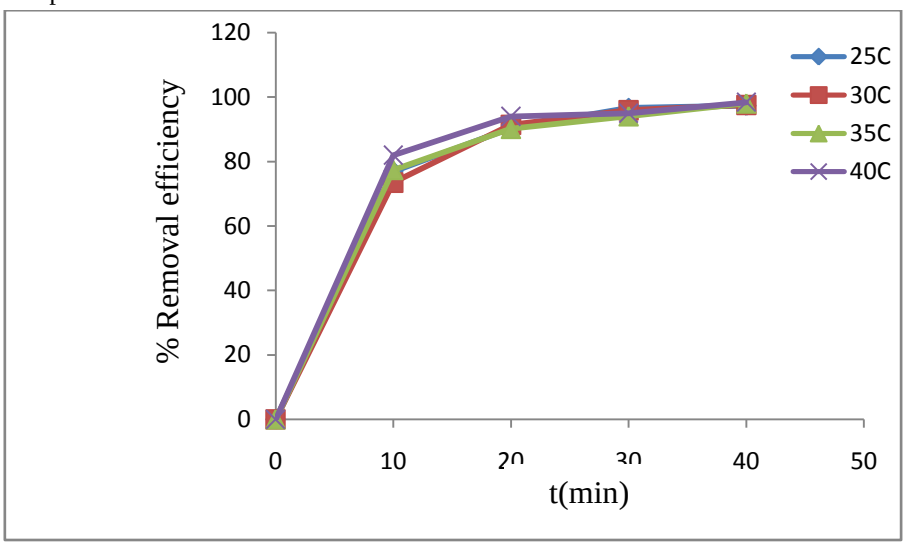


Figure 1: Effect of contact time on the adsorption of DR onto activated carbon

The effect of initial solution pH

The solution pH is an important factor that controls the adsorption of dye. The adsorption capacity can be attributed to the chemical form of dye in the solution at specific pH. Therefore, an increase in pH may cause an increase or decrease in the adsorption. In order to examine the effect of pH on the removal of DR dye, the solution pH were varied from 2.0 to 9.0 by addition of acid and base to the stock solution. The obtained results are shown in Fig.2.

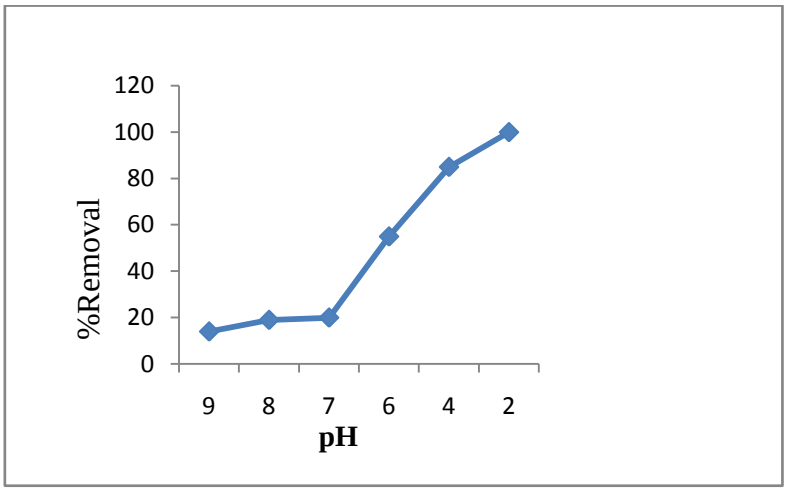


Figure 2: Effect of initial pH on the adsorption of DR onto activated carbon

As the of pH is lowered the overall surface charge on the activated carbon become positive that favors uptake of anionic dye due to increased electrostatic force of attraction. But, decrease in the adsorption at high pH may be due to occupation of the adsorption sites by OH ions and hence prevented the binding of negatively charge dye such as DR onto the negatively charged activated carbon.

The effect of temperature

The effect of temperature on the removal efficiency percent was studied in the temperature range of 25-40 °C at various initial dye concentration (25, 50, 85 and 100 mg/L) (Fig.3). As shown in Fig. 3, the optimum adsorption temperature was obtained to be 25 °C and the biosorption of DR dye decreased with increasing of temperature.

The decrease in biosorption capacity above 25 °C may be attributed to the deactivation of biosorbent surface or the destruction of some active sites on the biosorbent surface.

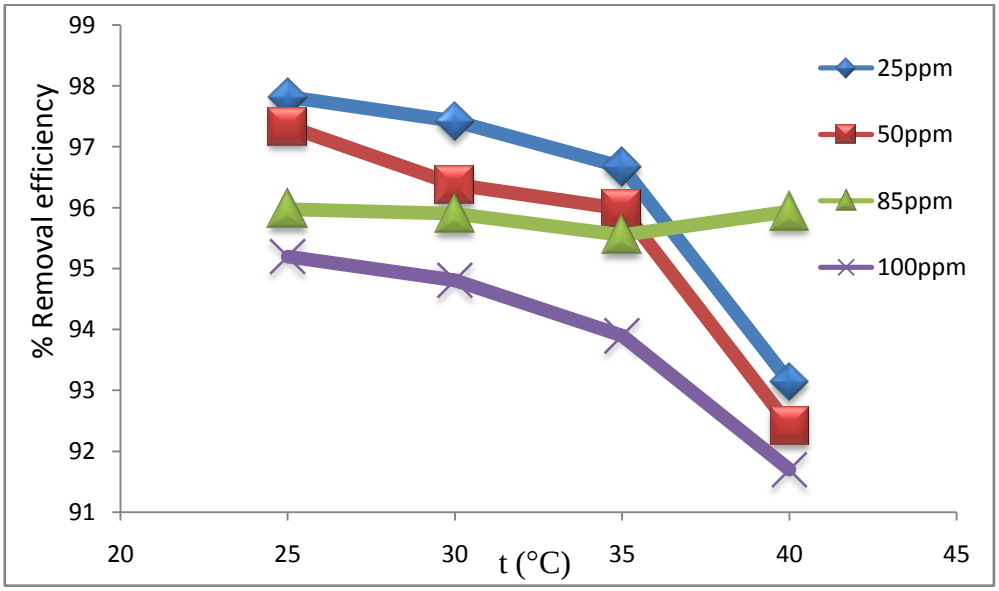


Figure 3: Effect of temperature on the adsorption of DR onto activated carbon

The Effect of Biosorbent Amount

The adsorption of DR dye on activated carbon was investigated by varying the absorbent dose (0.5-1.5 g) for 85 mg/L of dye concentration. As can be seen in this Fig.4, dye removal efficiency increased rapidly with increasing of biosorbent, which can be explained by the increased surface area of the biosorbent and availability of more binding sites for dye molecules [27]. On the other hand, further increase in the biosorbent dosage up to 2.0 g did not significantly change the biosorption yield. This was due to the binding of almost all dye to biosorbent surface and the maintenance of equilibrium between the dye molecules on the biosorbent and those present in solution.

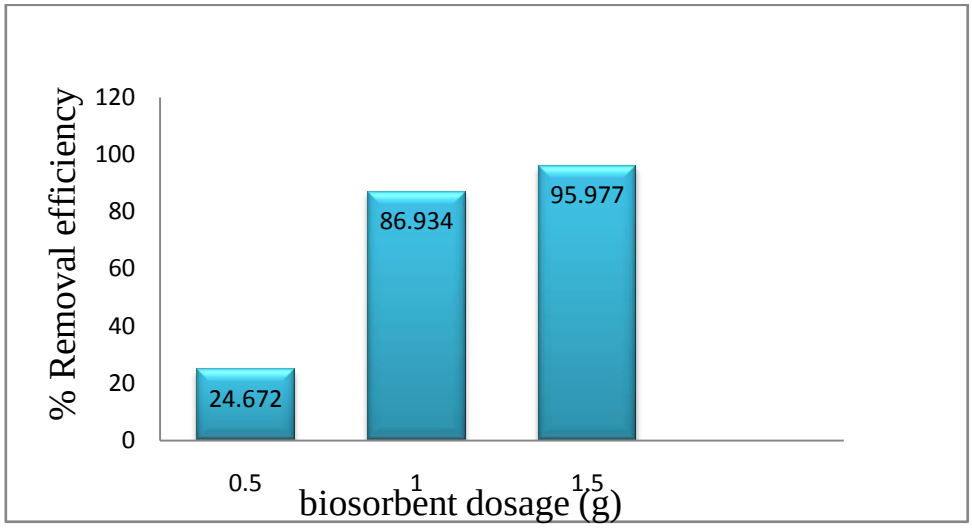


Figure 4: Effect of adsorption dosage on the adsorption of DR onto activated carbon

Effect of initial dye concentration

The experimental results of adsorption at different concentrations (20-100 mg/L) collected in Table.2 showed that the percent adsorption decreased with increase in initial dye concentration, but the actual amount of dye adsorbed per unit mass of activated carbon increased which 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 becomes less and hence the percentage removal of dye is dependent on initial concentration [28].

Table 2: The effect of initial DR 81 dye concentration on the removal efficiency

T (°C)	C_0	C_e	q_e	C_e/q_e	% R
25	23	0.5	4.5	0.111111	97.82609
	52	1.38	10.124	0.136310	97.34615
	87	3.5	16.7	0.209581	95.97701
	98	4.7	18.66	0.251876	95.20408

The Effect of Ultrasonic Irradiations

The effect of ultrasonic irradiation on biosorption was studied by equipment operating at 40 kHz. This biosorption experiment was performed by adding1 g of biosorbent into a beaker containing 50 mL of initial concentrations 85 ppm of dye solution. Fig.5 indicates the effect of ultrasonic irradiations on the removal efficiency of DR onto biosorbent.

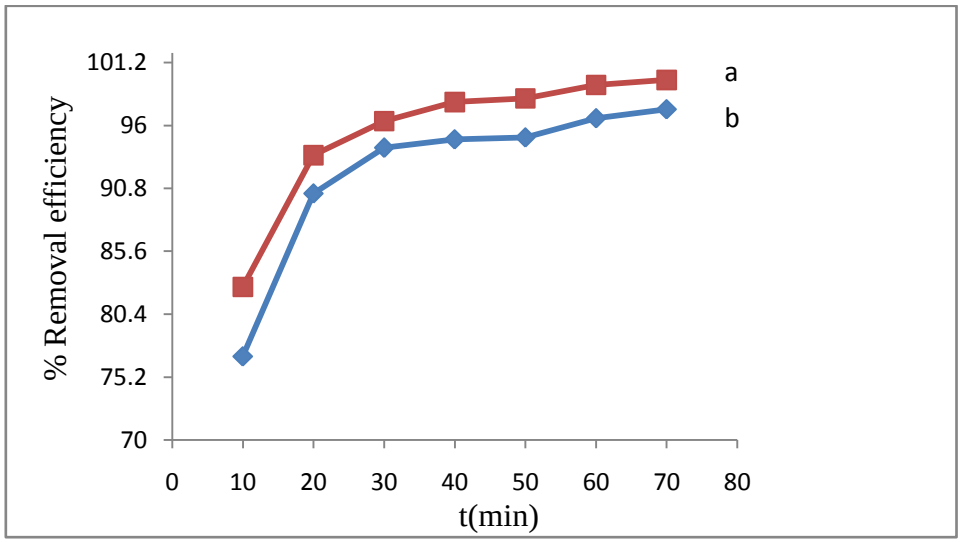


Figure 5: Effect of ultrasonic waves on the adsorption of DR onto activated carbon

As can be seen, the removal efficiency increased with using the ultrasonic irradiations from 0 to 70 minutes. Moreover, the results show that removal efficiency was increased from 76.92 to 82.65 by using ultrasonic irradiation at the initial minutes of contact time. This could be justified regarding the fact that at the presence of ultrasonic irradiations, there are more bubbles and also the bubbles collapse more violently. This behavior can lead to more considerable effects on the biosorption process.

Adsorption isotherms

The results obtained from adsorption of DR dye on the activated carbon derived from *Hibiscus Sabdariffa* stem by were analyzed by Langmuir and Freundlich models.

Langmuir isotherm

The Langmuir isotherm assumes a monolayer adsorption onto a solid surface with a definite number of identical sites. It can be considered as Eq. 3 [29]:

$$C_e/q_e = 1/q_{max} K_L + C_e /q_{max} \quad (3)$$

Where q_e (mg/g) and C_e (mg/ L) are the amount of adsorbed dye per unit mass of biosorbent and unadsorbed dye concentration in solution at equilibrium, respectively. q_{max} is the maximum amount of the biosorbed dye per unit mass of biosorbent to form a complete monolayer on the surface bound at high C_e (mg/g), and K_L is adsorption equilibrium constant (L/mg) that is related to the apparent energy of sorption. The linear plots of C_e/q_e versus C_e suggest the applicability of the Langmuir isotherms (Fig.6). The values of q_{max} and K_L were determined from slope and intercepts of the plots and are presented in Table 3.

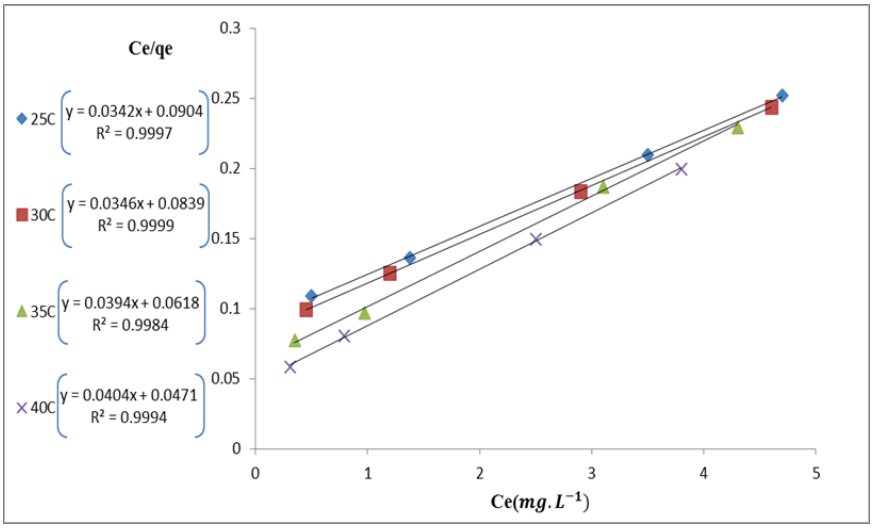


Figure 6: Linear plot of the model of Langmuir for the adsorption of DR onto activated carbon

The isotherm of activated carbon was found to be linear over the whole concentration range studies and the correlation coefficients were extremely high as shown in Table 3. The values of q_m increses with increase in temperature, thereby confirming that the process is endothermic [30].

Table 3: The Langmuir and Freundlich isotherm parameter at different temperature

T(K)	Langmuir isotherm			Freundlich isotherm		
	K_L	q_m	R^2	K_F	$1/n_F$	R^2
298	0.342	30.86	0.9996	7.421	0.635	0.985
303	0.262	33.67	1	6.818	0.661	0.990
308	0.182	36.9	0.9977	5.802	0.693	0.981
313	0.042	69.44	0.9994	2.903	0.871	1

Freundlich Isotherm Model

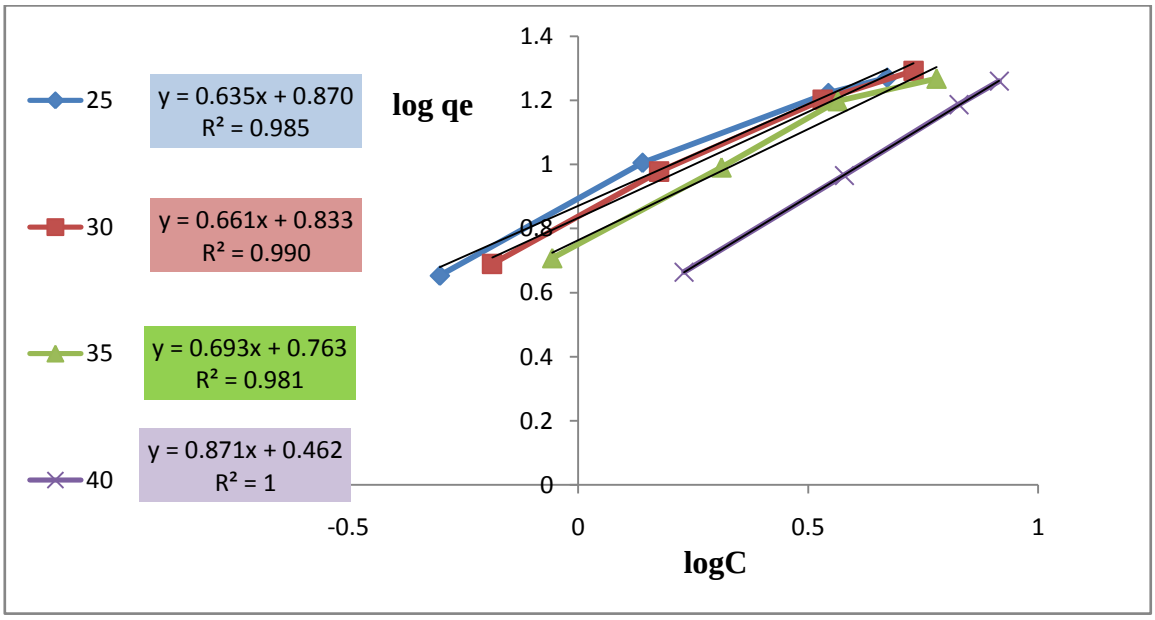


Figure 7: Linear plot of the model of Freundlich for the adsorption of DR onto activated carbon

The Freundlich's adsorption isotherm model, can be applied for a multilayer heterogenous adsorption and expressed as Eq. 4 [31]:

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

Where K_F (L/g) is the Freundlich isotherm constants that related to maximum biosorption capacity and n is the intensity of biosorption. Fig.7shows the linear plot of (log q_e versus log C_e) of Freundlich isotherm. The plot of log q_e versus log C_e was employed to generate the intercept value of K_F and the slope of $1/n_F$ (Table 3).

The Langmuir and Freundlich constants and regression coefficients for the DR biosorption are collected in Table 3. Based on the R values the Langmuir isotherm is better in predicting the DR biosorption than the Freundlich isotherm (Table 3). It is noteworthy that the Freundlich constant 1/n smaller than unity indicated that the biosorption process was favorable [32].

Adsorption Kinetics Study

In order to investigate the process of adsorption rates two kinetic moleds, (i) the Lagregren's pseudo first-order model (Eq.5) and HO's pseudo second-order models (Eq.6) were used to analyze the experimental data.

(i) The linearized form of pseudo first-order kinetic model can be written as [33]:

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

Where q_e and q_t are the amounts of DR adsorbed at equilibrium and at time t (mg/g) respectively, k_1 is the rate constant of pseudo first-order adsorption (min^{-1}). The values of k_1 and q_e were calculated from the slopes and intercepts of the plots ln (q_e- q_t) versus t (Fig.8).

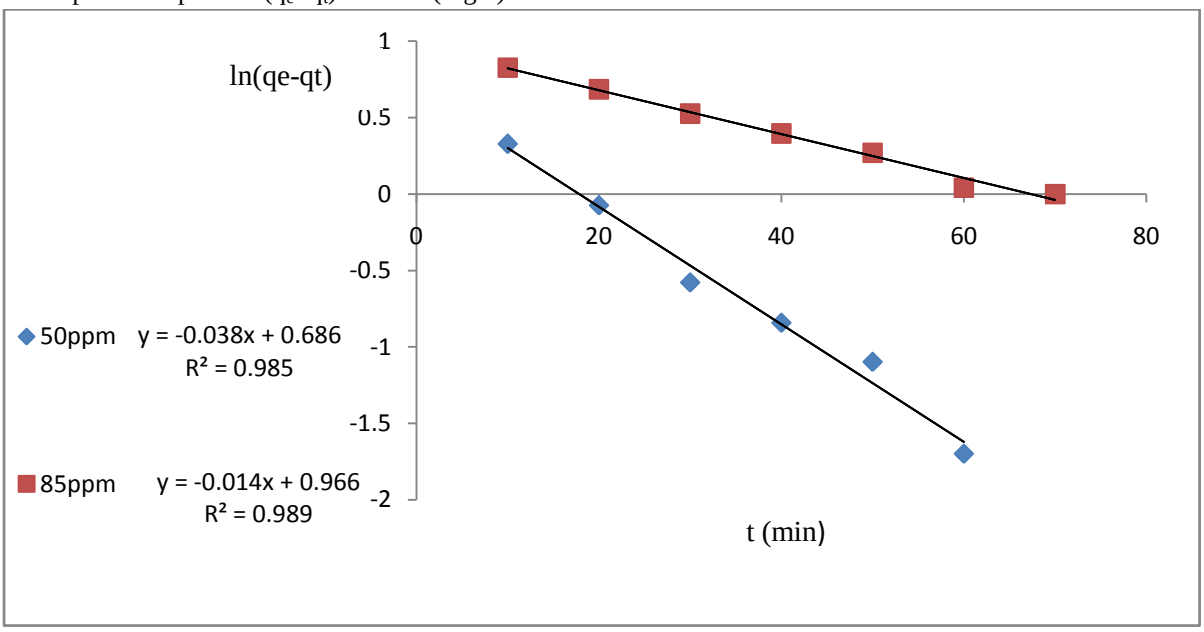


Figure 8: Pseudo-first order kinetic model for the adsorption of DR onto activated carbon

(ii) The pseudo second-order adsorption kinetics can be written as [34]:

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad (6)$$

Where k_2 is the equilibrium rate constant for pseudo second-order adsorption (g mg^{-1}.min^{-1}). The slope and intercept of plot of (t/q_t versus t) were used to calculate the pseudo second-order rate constant k_2 and q_e (Fig.9).

The results are shown in Table 4. The correlation coefficients of pseudo-second order models are closer to unity and the calculated q_e values computed from pseudo-second order equation, show good agreement with experimental values indicating the applicability of pseudo-second order kinetic model for adsorption of DR dye on activated carbon.

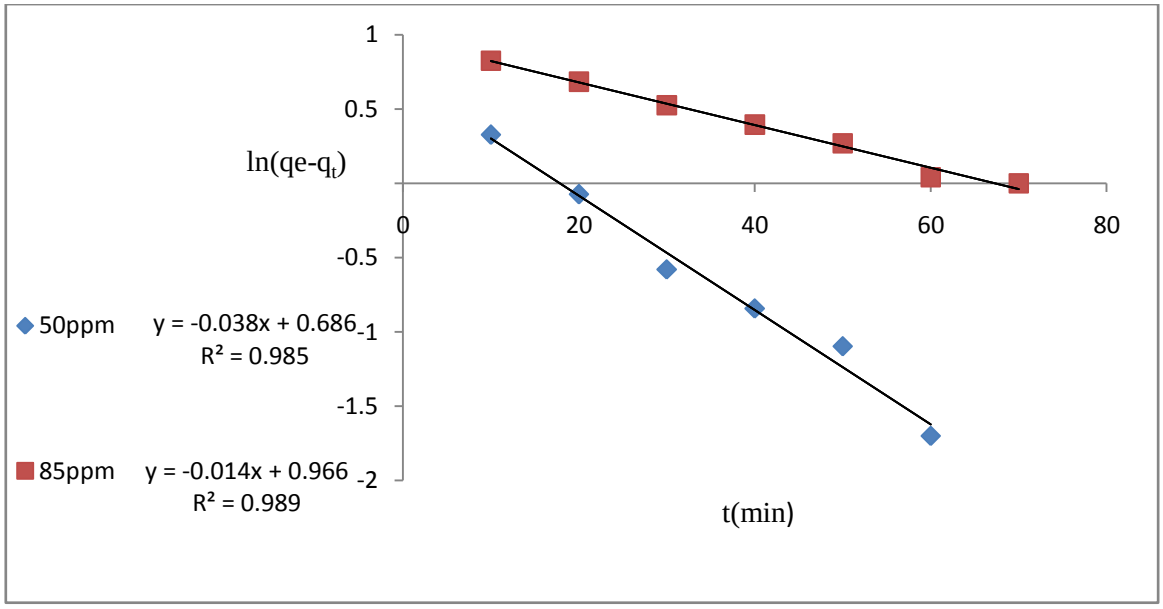


Figure 9: Pseudo-second order kinetic model for the adsorption of DR onto activated carbon

Table 4: Kinetic parameters calculated by using pseudo first-order and pseudo second-order rates models for biosorption of DR

Concentration of DR (mg L^{-1})	Pseudo first-order kinetic model		Pseudo second-order kinetic model	
	k_1	R^2	k_2	R^2
85	0.033	0.989	6.193	0.998
50	0.088	0.985	0.035	0.999

Thermodynamic Study

In order to describe the thermodynamic behavior of the biosorption of DR onto activated carbon, thermodynamic parameters were calculated including the change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) using the following thermodynamic equations (Table 5) [35]:

$\Delta G° = -RT \ln K_c$ (7)

$\text{Ln } K_c = \Delta S°/R - \Delta H°/RT$ (8)

Where R is the universal gas constant (8.314 J K^{-1} mol $^{-1}$), T is the absolute solution temperature (K) and K_c represents the equilibrium biosorption constant.

Table 5: The thermodynamic parameters for biosorption of Direct red 81 at different temperature

T (K)	ΔG° (kJ.mol^{-1})	ΔH° (kJ.mol^{-1})	ΔS° (kJ mol^{-1})
298	-4.937	-55.352	-168.167
303	-4.649		
308	-4.003		
313	-2.319		

The plots of lnK_c*vs.* 1/T gives the straight line from which ΔH° and ΔS° are calculated from the slope and intercept. The value of ΔG° is -4.937, -4.649,-4.003 and -2.319 kJ/mol at 298, 303, 308 and 313 K respectively. The negative values of ΔG° indicate that the DR biosorption onto activated carbon was spontaneous and feasible at all the studied temperatures. Moreover, the negative value of ΔH° (−55.32 kJ mol) indicates that biosorption followed an exothermic process. The negative value of ΔS° (-168.167 kJ/mol) also suggests decreased randomness at the solid-solution interface during the DR biosorption onto activated carbon.

Conclusion

In this study, carbon obtained from *Hibiscus Sabdariffa Stemby* as biosorbent was used to adsorb DR dye from aqueous solution. The effect of different parameters such as temperature, pH, adsorbentdose, contact time, initial dye concentration and ultrasonic irradiation were evaluated on removal efficiency. It was observed that the modified silk maze had maximum monolayer adsorption capacity, mg/g. The Langmuir and Freundlich isotherms were well fitted with the experimental values. Based on the R values the Langmuir isotherm is better in predicting the DR adsorption than the Freundlich isotherms. The thermodynamic studies showed that the biosorption process was spontaneous and exothermic by the negative ΔG° value and negative ΔH° value. Also the adsorption of DR onto activated carbon well agreed with the pseudo -second -order kinetic model. Also the effect of ultrasonic irradiations was studied on biosorption. The results showed removal efficiency increased in the presence of ultrasonic irradiations.

Acknowledgements

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