

Adsorption of Congo Red Dye from Aqueous Solution: Equilibrium and Its Kinetics

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Abstract - Congo red (CR), one of the toxic dyes, which is extensively used for dyestuffs, textile, paper and plastic industries. CR does not easily biodegrade in aqueous medium and show harmful effect on aquatic as well as human life. In the present work adsorption studies of CR onto Sarp Gandha (*Rauwolfia serpentina*) leaves powder (SLP) adsorbent was examined in aqueous solution at 27.5°C with the effects of initial concentration, adsorbent dose, temperature and contact time. Highest 70.98% adsorption efficiency recorded for 20 mg/L solution concentration onto 2.5g of SLP. The applicability of Langmuir and Freundlich isotherm model was investigated, and the Freundlich adsorption isotherm model exhibited the best fit than Langmuir isotherm model with the experimental data. The kinetic data was fitted to pseudo-second-order kinetic model. The adsorption technique was found to be SLP is very useful and cost effective for a better removal of hazardous CR dye.

Keywords: Congo red, Isotherm, Kinetics, Sarp Gandha leaves powder, Thermodynamics.

I. INTRODUCTION

Nowadays, dyes are widely used in chemical Industries, which may enter ecosystems (e.g. aquatic, soils) during dyes production and subsequent application processes¹. Dyes are natural or synthetic coloured organic compounds having the property of imparting their colours to other substances. Although there are many natural dyes available, the ingrain coloured by these are neither bright nor durable. Hence synthetic dyes are generally used which are cheap and readily available. Synthetic dyes are widely used in industries such as textiles, plastics, paper rubber, tanning, cosmetics, pharmaceutical and food stuff. In industrial effluents dyes are one of the most hazardous chemical compound found and need to be treated, since their presence in water bodies reduces light penetration, producing the photosynthesis of aqueous flora². Among various dye species, Congo red (CR) is a typical benzidine-based azo dye and mainly exists in the effluent of textile, paper, printing industries, etc. After entering natural environment, CR can be metabolized to benzidine, a well-known human carcinogen, which may be a cause for human allergic diseases. Due to its high chemical stability and low

biodegradability, conventional biological process was found to be ineffective to decolorize and degrade CR wastewater treatment. Over the past few decades, several processes have been used for the removal of dyes from wastewater such as biological, chemical precipitation, coagulation or flocculation, solvent extraction, membrane filtration, ion exchange, ozonation, electrochemical destruction and adsorption³⁻⁴. Adsorption process has simplicity of design, more efficient, easy to operate, insensitivity to toxic substances, environmental friendliness, non-toxicity, availability of a wide range and cost effective, hence, it has been suggested as a potential alternative to the existing physical / chemical /biological methods for the removal of dyes from industrial effluents or waste water.

Natural adsorbents such as agricultural waste, waste food or low-cost inorganic material have been most popular for wastewater treatment due to availability and low-cost adsorbent.

Various low-cost adsorbents that have been successfully used for the adsorption of dyes such as, peanut hull⁵, gram seed husk⁶, watermelon rind⁷, rice husk⁸, bentonite⁹, *Azadirachta indica* leaf¹⁰, Jujuba seeds¹¹, green gram seed husk¹² and ball-milled sugarcane bagasse¹³ etc.. Today, more attention is being given to the use of low-cost adsorbents.

For the present work low-cost agricultural adsorbent was selected from *Rauwolfia serpentina* (Sarp Gandha) shrub is used to adsorption of Congo red organic dye. Its leaves when freshly ground and used as adsorbent for removal of CR. The objective of the present study was to investigate the potential of Sarp Gandha leaves powder (SLP) as an alternative adsorbent for the removal of Congo red dye from aqueous solution.

Kinetics of adsorption has also been studied to explore the equilibrium as well as the rate of adsorption of Congo red dye on SLP adsorbent. The equilibrium study was investigated to observe the effects of various adsorption parameters such as contact time, adsorbent dose and initial dye concentration on the process. Adsorption data were analysed using Langmuir and Freundlich adsorption isotherm models. The adsorption technique was found to be very useful and cost effective for a better removal of hazardous CR dye.

II. MATERIALS AND METHODS

a) Preparation of adsorbent

The mature and fresh Sarpagandha leaves were collected from local area and washed thoroughly by using distilled water to clean them from dirt and impurities. After that, the Sarpagandha leaves are dried in shadow region. After drying the leaves was ground by grinder to constant size of 65 μm fine powders of Sarpagandha leaves. The dried fine Sarpagandha leaves powder (SLP) adsorbent was kept in an air tight glass bottle ready for further experiments.

b) Preparation of adsorbate

Congo Red (CR) (CI: 22120, MW: 696.66 g.) supplied by Loba Chemicals Pvt. Ltd. Mumbai (India) were used as adsorbate without purification.

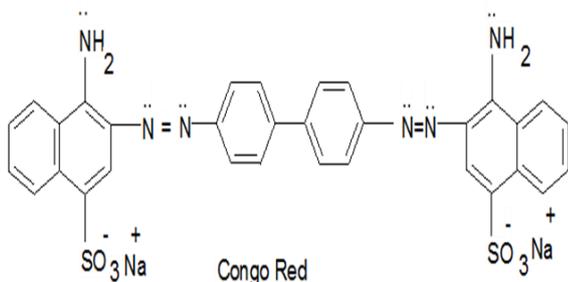


Figure 1: Chemical structure of Congo Red

The stock solution of 1000 mg/L CR dye was prepared by dissolving the desired amount of Congo red in double distilled water and suitable diluted to require initial concentrations. The structure of this dye is shown in Fig.1.

III. BATCH ADSORPTION EXPERIMENTS

Adsorption experiment was carried out by batch adsorption techniques at room temperature. The effect of pH on CR removal were studied by shaking 50 ml, 20 mg/L. of CR dye solution concentration with 0.5 g. adsorbent dose in conical flasks. The effect of contact time and initial concentration were studied by shaking 100 ml 20 mg/L CR solutions concentration with 1.0 g. adsorbent in a 250 ml conical flask. After definite time intervals, a sample were withdrawn from the flask, the supernatant solution was analyzed for residual dye concentration. Adsorbent dose effect was studied using 20 mg/L CR solution concentration. The optical density was analyzed using a UV-Visible single beam Spectrophotometer (BioEra: Cal No.BI/CI/SP/SB-S-03), at $\lambda_{\text{max}} = 510 \text{ nm}$. The pH of the CR solution was adjusted by adding 0.1 M HCl or 0.1 M NaOH solution and measurement was done by digital pH-meter (Elico: LI 615). The amount of dye adsorbed per unite weight of husk adsorbent at time, 't',

q_t (mg/L) and percentage dye adsorption capacity was calculated as

$$q_t = \frac{V(C_0 - C_t)}{M} \quad (1)$$

$$\% \text{ adsorption capacity} = \frac{(C_0 - C_t)}{C_0} * 100 \quad (2)$$

Where, C_0 is the initial CR dye concentration (mg/L), C_t is the concentration of CR dye at any time t , 'V' is the volume of solution (mL) and M is the mass of SLP (g).

IV. RESULTS AND DISCUSSION

a) Effect of contact time and initial concentration

The effect of contact time is an important parameter; the dose of adsorbent was kept constant in all bottles. The effect of contact time was studied at different initial concentration of CR dye with time. The time is varies in parameter for the adsorption of CR dye on SLP is shown in Fig.2. The experimental results of adsorptions of CR dye on SLP investigates that the percentage adsorption capacity increased with increase in contact time due to availability of more number of active sites on the surface of the SLP adsorbent. As increase the initial concentration of CR dye, decrease the percentage adsorption capacity due to at low concentration CR present in adsorption medium could interact with the binding sites on the surface of adsorbent so higher adsorption yields were obtained. At higher concentrations, lower adsorption yields were observed because of the saturation of the adsorption sites.

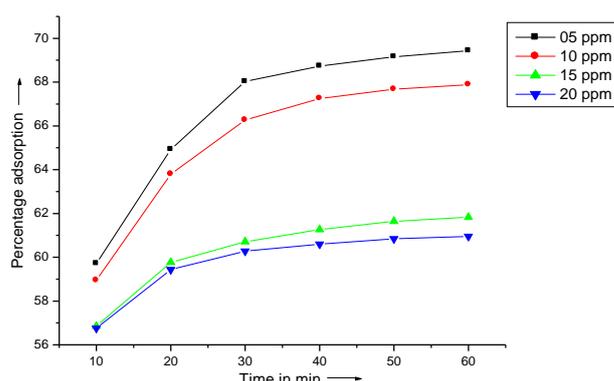


Fig.2: Effect of initial concentration on percentage adsorption of congo red dye. [Adsorbent dose=1.0 g, Volum of adsorbate=100 mL, Temp=300.5 k, pH=6.4]

b) Effect of adsorbent dose

It is an important parameter that strongly influences the adsorption technique by affecting adsorption capacity of the adsorbent. The experiments were carried out following general procedure for adsorption studies at the various contact time for each adsorbent. The effect of dose of SLP adsorbent the initial

concentration of the dye solution in all the bottles were kept constant and the dose of adsorbent of fixed particle size was varied. The plots of percentage adsorption of CR dye versus contact time of various doses of adsorbent. The results are shown in Fig. 3.

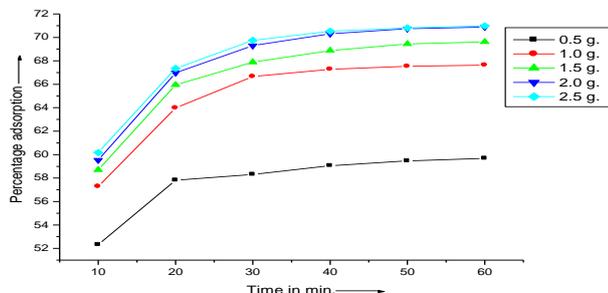


Fig.3: Effect of adsorbent dose on percentage adsorption of congo red dye. [Initial conc.=20 ppm, Volume of adsorbate=100 mL, Temp=300.5 K, pH=6.4]

The influence of adsorbent dose on CR adsorption by SLP was investigated in the range of 0.5–2.5 g. The adsorption efficiency increased from 59.68 to 70.98 % as the adsorbent dose increased from 0.5 to 2.5 g. The increase in the percentage adsorption of the CR dye adsorption with adsorbent dose could be attributed to an increase in adsorbent surface area augmenting the large number of adsorbent sites available for adsorption as already reported¹⁴.

c) Effect of temperature

It is one of the important parameter affecting separation in most of the adsorption processes. In order to examine the effect of temperature on CR dye adsorption five different temperatures were selected. In the present work percentage removal of CR dye decreases from 59.93 to 56.30 % by increase in temperature from 305.5 to 325.5 K. The percentage adsorption of CR dye was found to decrease with increase in temperature as shown in Fig.4.

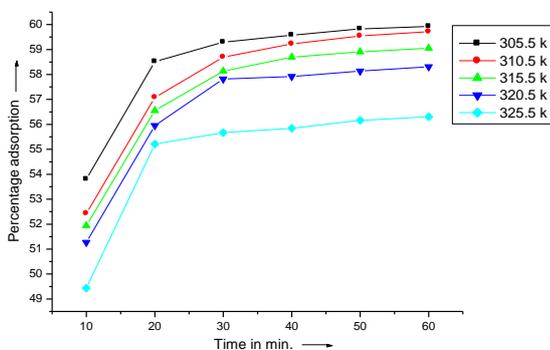


Fig.4: Effect of temperature on percentage adsorption of congo red dye. [Initial conc.=20 ppm, Adsorbent dose=1.0 g, Volume of adsorbate=100 mL, pH=6.4]

It reveals that the adsorbate-adsorbent system is exothermic in nature for which the evaluation of thermodynamic parameter was carried out. Thus the removal

of CR dye is leading to a decrease in the residual forces on the surface of the SLP adsorbent and hence causing a decrease in the surface energy of the adsorbent¹⁵.

Thermodynamic study was performed to find the nature of adsorption process. Thermodynamic parameters such as Gibb's free energy change ΔG , enthalpy change ΔH and entropy change ΔS were calculated by using Van't Hoff's equation. The calculated thermodynamic parameter values are shown in Table: 1.

TABLE 1

Thermodynamic parameter values of SLP adsorbent with CR solution at different temperatures

Temp (K)	Free energy change (ΔG°), ΔH° and ΔS° values of CR solution at different temperatures		
	ΔG° KJ/mole.	ΔH° KJ/mole	ΔS° J/mole
305.5	-1.493	- 6.962	- 17.888
310.5	-1.404		
315.5	-1.314		
320.5	-1.225		
325.5	-1.135		

In present research work, the ΔG^0 values at various temperature obtained are < -10 KJ/mole; it indicates that physical adsorption was the predominant mechanism in the adsorption process. The Gibb's free energy indicates the degree of spontaneity of the adsorption process, where more negative value reflects a more energetically favorable adsorption process. The negative value of ΔG^0 indicates that the adsorption is favourable and spontaneous¹⁶⁻¹⁷. The negative value of ΔS and ΔH suggests that the decreased disorder and randomness at the solid solution interface with exothermic adsorption¹⁸⁻¹⁹.

V. ADSORPTION ISOTHERM

Adsorption isotherms are important for the description of how molecules of adsorbate interact with adsorbent surface. Hence Langmuir and Freundlich isotherms were selected in the present study.

a) Langmuir isotherm

Langmuir adsorption isotherm describes quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent and after that no further adsorption takes place. The Langmuir isotherm is valid for monolayer adsorption onto the surface containing a finite number of identical sites. The linear form of the equation is given by,

$$\frac{1}{q_e} = \left(\frac{1}{Q_0}\right) + \frac{1}{bQ_0C_e} \quad (3)$$

Where, C_e (mg/L) is the equilibrium concentration of the adsorbate, q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent, at equilibrium, Q_0 (mg/g) and b (L/mg) are Langmuir constants related to maximum monolayer adsorption capacity and energy of adsorption respectively. The values of Q_0 and b are calculated from the slope and intercept of plot of $\frac{1}{q_e}$ against $\frac{1}{C_e}$ respectively. The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter (R_L). Equilibrium parameter (R_L) is a dimensionless constant referred to as separation factor.

$$R_L = \frac{1}{1+bC_0} \tag{4}$$

Where, C_0 is initial concentration in (mg/L) and b is Langmuir constant related to the energy of adsorption. R_L Value indicates the adsorption nature to be either unfavourable if $R_L > 1$, linear if $R_L = 1$, favorable if $0 < R_L < 1$ and irreversible if, $R_L = 0^{20}$.

b) Freundlich isotherm

Freundlich presented an empirical adsorption isotherm for non-ideal sorption on heterogeneous surface as well as multilayer sorption and is also expressed as

$$\frac{x}{m} = K_f C_e^{1/n} \tag{5}$$

Where, x is the quantity adsorbed, m is the mass of the adsorbent, C_e is the equilibrium concentration of adsorbate (mg/L), The constants K_f and n can be obtained by taking \log on both sides of equation (5) as follows,

$$\log \frac{x}{m} = \frac{1}{n} \log C_e + \log K_f \tag{6}$$

The constant K_f is an approximate indicator of adsorption capacity, while $\frac{1}{n}$ is a function of the strength of adsorption in the adsorption process. If $n = 1$ then the partition between the two phases are independent of the concentration.

If value of $\frac{1}{n}$ is below one, it indicates a normal adsorption, on the other hand $\frac{1}{n}$ being above one indicates co-operative adsorption. A plot of $\log \frac{x}{m}$ against $\log C_e$ gives a straight line with an intercept on the ordinate axis.

The value of n and K_f can be obtained from the slope and the intercept of the linear plot. The Langmuir isotherm parameter values are given in Table 2(a).

TABLE 2(a)

Langmuir isotherm parameter values of SLP with CR dye solution

Conc. of CR (mg/L)	Langmuir constants			
	Q ₀ (mg/g)	B (L/g)	R _L	R ²
20	4795.3	0.124	0.287	0.995

The Freundlich isotherm parameter values are given in Table 2(b).

TABLE 2(b)

Freundlich isotherm parameter values

Conc. of CR (mg/L)	Freundlich constants		
	$\frac{1}{n}$	K _f (mg/g(L/g)) ^{1/n}	R ²
20	1.458	5.89	0.997

The R_L value was found to be between 0 and 1 for CR studies, it is confirm that the on-going adsorption of CR is favourable. The data reveals that the Freundlich model yields better fit than the Langmuir model. The value of n suggests that deviation from linearity, if $n = 1$ the adsorption is homogenous and there is no interaction between adsorbed species.

The value of n is greater than unity, ($1 < n < 10$), that means favorable adsorption²¹. If value of $\frac{1}{n} > 1$ indicates the adsorption is favored and new adsorption sites are generated²²⁻²⁵. The value of $\frac{1}{n}$ presented in Table: 2 (b) was found to be greater than one, indicates the adsorption is favoured and new adsorption sites are generated.

VI. KINETIC MODEL OF ADSORPTION

Kinetic studies are significant for any kind of adsorption process. Lagergren pseudo-first and pseudo-second order kinetic models can be suggested for an adsorption. Pseudo-first order kinetics is present to describe the rate of adsorption process in liquid-solid phase. The Lagergren pseudo-first order rate equation is given as,

$$\frac{dq}{dt} = K_1(q_e - q_t) \tag{7}$$

After definite integration by applications of the conditions $t = 0$ to $t = t$ and $q = 0$ to $q = q_e$ Equation (7) becomes,

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \tag{8}$$

Where, q_e (mg/g) is the amount of adsorption at equilibrium, q_t (mg/g) denotes the amount of adsorption at time t (min.) and K_1 (min⁻¹) is the rate constant of the pseudo-

first order model. Based on experimental results, linear graphs were plotted between $\log(q_e - q_t)$ versus t , to calculate K_1 , q_e and R^2 . Adsorption rate was calculated from the slop and intercept the calculated rate s given in Table.3. (a)

TABLE 3(a)

Pseudo first order kinetic parameter values of SLP adsorbent with CR

Adsorbent	Pseudo-First order		
	K_1 (min^{-1})	q_e (mg/g)	R^2
SLP	1.241×10^{-2}	387.035	0.753

The pseudo-second order equation can be written as

$$\frac{dq}{dt} = K_2(q_e - q_t)^2 \quad (9)$$

Where, K_2 ($\text{g.mg}^{-1}\text{min}^{-1}$) is the rate constant of the pseudo-second order.

The linear form of equation is

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (10)$$

K_2 and q_e can be obtained from the intercept and slope of plotting t/q_t against t . Adsorption rate was calculated from the slop and intercept the calculated rate is given in Table 3(b).

Table 3(b)

Pseudo second order kinetic parameter values of SLP adsorbent with CR

Adsorbent	Pseudo Second order		
	K_2 (g./mg.min)	q_e (mg/g)	R^2
SLP	3.747×10^{-3}	1405.97	0.998

The value of R^2 with pseudo-first order kinetics was 0.753, while for second order is 0.998 for SLP adsorbent. The best correlation for the system provided by the pseudo second order kinetic model suggests that chemical adsorption involving valence forces through sharing or exchange of electrons between adsorbent and adsorbate might be significant²⁶. It is clear that the adsorption of CR on SLP adsorbent was better represented by pseudo second order kinetics.

VII. CONCLUSION

The conclusions can be drawn based on the investigation of CR dye adsorption by SLP adsorbents. The percentage adsorption of CR dye increased with increase in adsorption dose of SLP adsorbent, while percentage adsorption of CR dye increased with decrease in initial concentration of CR dye solution. Higher percentage adsorption capacity of CR dye on SLP was observed at lower temperature. The negative value of

ΔG^0 confirms that the feasibility of the reaction and spontaneous nature of the adsorption. The negative value of ΔS and ΔH suggests that the decreased disorder and randomness at the solid solution interface with exothermic adsorption. The experimental data for the adsorption of CR dye on SLP fits well for the Freundlich adsorption isotherm model than Langmuir isotherm model. The investigation showed that SLP adsorbent was agricultural waste, abundant, cheap, readily available and eco-friendly effective adsorbent, which could be used as potential adsorbent for removal of CR dye from aqueous solution and polluted water.

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