Removal of Congo Red dye from Wastewater Using Orange Peel as an Adsorbent

Vidya S. Harnal, Upendra Darla and Dilip H. Lataye *

Abstract-- **Present study deals with the utilization of orange peel as an adsorbent for the removal of Congo Red (CR) dye from aqueous solution. The proximate analysis revealed that, the adsorbent has porous structure with volatile matter of about 79.14% and fixed carbon 13.08%. The adsorbent has neutral charge at the pH of 6.37. From the batch adsorption study, the optimum adsorbent dose found 40 g/L, the optimum pH was found to be 6.43 and equilibrium time was found 3 h. The increase in adsorption with temperature shows the endothermic nature of adsorption process. The maximum percent removal of CR was found to be 94.24 at the initial concentration of 100 mg/L at 30 °C. The isotherm analysis shows that the Langmuir adsorption isotherm is best-fitted for Congo Red adsorption on orange peel followed by Freundlich and Temkin isotherm equations. The values of the constants K_L and q_m are 0.053 L/mg and 11.919 mg/g respectively. Thermodynamic study shows the endothermic nature nature of the adsorption process. The heat of adsorption (ΔH) was found to be 21.475 KJ/mol and change in entropy (ΔS) was found 0.149 KJ/mol K. Gibbs free energy (ΔG) was found -24.633, -26.548 and -28.464 kJ/mol. The adsorption process is found to be feasible and spontaneous. The orange peel can be utilized as an adsorbent for the removal of other pollutants.**

Key words - Dye removal, orange peels, Congo red (CR), Isotherms, Langmuir, Freundlich, Temkin

I. INTRODUCTION

Now a days the treatment of wastewater has a major concern, because several industries discharge their effluents to the natural streams with or without treatment. Dye industry is one of the industries, the influent of which, imparts colour to the stream if discharged without treatment. Congo red (CR) dye is one the dyes used for various purposes in various industries, it is the sodium salt of benzidinediazo-bis-1-naphthylamine-4-sulfonic acid (formula: $C_{32}H_{22}N_6Na_2O_6S_2$; molecular weight: 696.66 g/mol). It is a secondary diazo dye. Congo red is water soluble, yielding a red colloidal solution; its solubility is better in organic solvents such as ethanol. It has a strong, though apparently non-covalent affinity to cellulose fibers. However, the use of congo red in the cellulose industries (cotton textile, wood pulp & paper) has long been abandoned, primarily because of its tendency to change colour when touched by sweaty fingers, to run, and because of its toxicity.

The congo red is hazardous in case of skin contact (irritant), ingestion, inhalation. It may cause adverse reproductive effects (fetotoxicity, female and male fertility) and birth defects (teratogenic-developmental abnormalities). Its affect may be genetic material (mutagenic). It affects blood (change in clotting factors, alterations of platelets and leukocytes, transient leukopenia, leukocytosis), behavior (somnolence), respiration (dyspnea, chronic pulmonary edema), and the vascular system. Thus, it is necessary to treat the wastewater containing CR and to find an effective method of wastewater treatment in order to remove colour from effluents from the dye industries.

Physical and chemical techniques such as coagulation, adsorption, ultra filtration and reverse osmosis are generally used efficiently to remove dyes from textile wastewater. However, these processes are considered as nondestructive, since they merely transfer the dye from liquid to solid wastes. Consequently, the regeneration of the adsorbent and post-treatment of solid wastes, which are expensive operations, are needed (Hamissa et al.2008, Gupta and Suhas, 2009). The adsorptive treatment is the simple and cost effective method of treatment, which is widely used for the removal of various impurities from wastewater. Several adsorbents have been used by various researchers for the removal of colour from wastewaters viz, banana peels, chitosan (Annadurai et al. 2002, 2008), polysaccharides derivatives (Delval et al. 2002), Neem leaf powder (Bhattacharyya and Sarma 2003), acid activated red mud (Tor and Cengeloglu, 2006), β-cyclodextrin and starch (Ozmen et al. 2007), bagasse fly ash (Srivastava et al. 2008), rice husk carbon in powder form (Sharma and Janveja, 2008), $CaCl_2$ modified bentoni (Lian et al. 2009). Similarly other adsorbents that have been used in past are chitosan hydrobeads (Chatterjee et al. 2009), Jute stick powder (Panda et al. 2009), biowaste materials (Kaur et al. 2013), Roots of *Eichhornia crassipes* (Wanyonyi et al. 2014), zeolitic particles (Madan et al. 2019), activated carbon prepared from coffee waste (Lafi et al. 2019), hydrothermal treated shiitake Mushroom (Yang et al., 2020).

In the present work, orange peel has been used as an adsorbent for the removal of CR from wastewater. The batch experimental adsorption study has been carried out to determine the adsorption capacity of orange peel under the temperature controlled conditions.

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II. Materials & Methodology

Adsorbent

Adsorption of congo red dye (CR) was studied using orange peel as an adsorbent. Orange peel, which is thrown as a waste material was collected from the local juice centers. The peel was dried and grounded by domestic mixer and the powder passing 0.5 mm and retaining on 0.3 mm sieve were taken. The powder was washed 4-5 times with distilled water boiled for 30 – 60 minutes. After boiling, squeezed, and sun dried for 48 hrs.

Adsorbate

The adsorbate, congo red dye was procured from Patel Scientific Company, Nagpur (India). The stock aqueous solution of 1000 mg/L (ppm) concentration was prepared by taking 1 g of dye powder in distilled water (DW). The test solutions in the range of 1-500 ppm concentration were prepared from the stock solution of congo red dye with DW.

Other chemicals

All the other chemicals used in the present study viz., acids, alkalies, KNO_3, etc., were of analytical reagent (AR) grade and were supplied by S.D. Fine Chemicals, Mumbai, India.

Batch experimental programme

The experiments were performed by batch adsorption studies. The standard solutions of 1, 10, 50 and 100 mg/L concentration were prepared from the stock solution. The standard graph of concentration versus absorbance was prepared by measuring the absorbance at 500 nm wavelength. Absorbance was found by using UV-Vis spectrophotometer (SHIMADZU, UV-2450). A temperature controlled orbital shaker (REMI Instruments, Mumbai, Model: CIS-24 BL), was used for the batch adsorption study. The temperature range for the studies was used from 20 ºC to 40 ºC. All the batch studies were performed at the rate of 150 revolutions per minute (RPM). For each experimental run, 50 ml aqueous solution of the known concentration of congo red was taken in a 250 ml conical flask containing a known mass of the adsorbent and absorbance was measured.

The amount of CR adsorbed by the adsorbent at equilibrium was calculated by:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

Where, q_e - Equilibrium solid phase concentration of CR (mg/g), C_0 - Initial concentration of CR in solution (mg/l), C_e - Equilibrium concentration (mg/L), W - Adsorbent dose (g/L), and V – Volume of sample in litre (L).

III. Result And Discussion

Characterization of orange peel

Characteristics of orange peel included proximate and particle size analysis. Due to presence of carbon content, orange peel may be treated as organic in nature. The organic nature of orange peel imparts porosity. The physico-chemical characteristics of the adsorbents are given in Table 1.

TABLE 1

Characteristics of Orange peel

Characteristics	Orange peel
Average Particle size (mm)	0.40
Moisture (%)	4.80
Ash (%)	2.98
Volatile matter (%)	79.14
Fixed Carbon (%)	13.08

Point of Zero Charge (pH_{PZC})

The point of zero charge of the adsorbent was determined by solid addition method (Balistrieri and Murray, 1981, Lataye et al. 2006). To a series (6 nos.) of 100 mL conical flasks 45 mL of KNO_3 (0.1 M) solution of known strength was taken and the pH_0 values of the solution were roughly adjusted from 2 to 12 by adding either 0.1N HCl or NaOH. The total volume of the solution in each flask was made exactly to 50 mL by adding KNO_3 solution of the same strength. The pH_0 of the solutions were then accurately noted. One gram of adsorbent was added to each flask, which were securely capped immediately. The suspensions were then manually shaken intermittently and allowed to equilibrate for 48 h. The pH_f values of the supernatant liquid were noted after 48 h. The difference between initial pH (pH_0) and final pH (pH_f) values ($\Delta pH = pH_0 - pH_f$) were plotted against pH_0. The point of intersection of the resulting curve at which $\Delta pH = 0$ gave the pH_{PZC}. The point of zero charge in the present study was found to be 6.37 as shown in Figure 1.

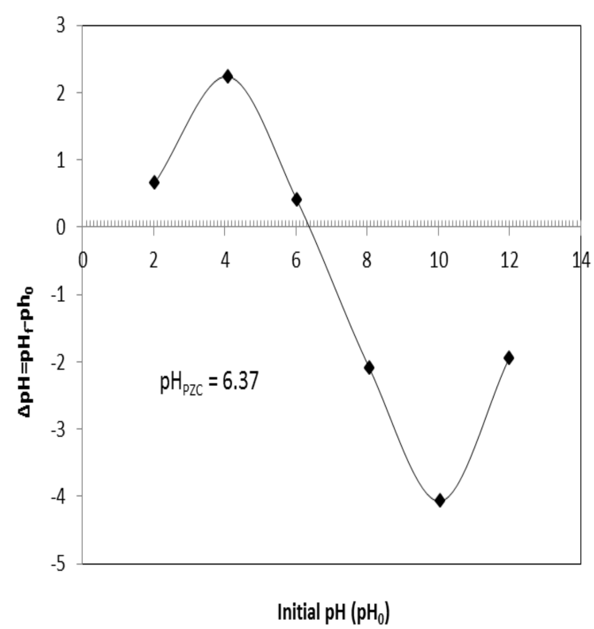

Fig. 1. Point of zero charge of adsorbent

Batch adsorption study

In order to study the effect of different parameters on adsorption of CR, the batch experiments were performed. The experiments were carried out in 250 mL stopper conical flasks for removal of CR from aqueous solution of known concentration by using orange peel. The effect of various parameters is discussed further.

Effect of adsorbent dose (m)

The effect of adsorbent dose (*m*) on the removal of CR by orange peel at initial concentration C_0 = 100 mg/L is shown in Figure 2. It can be seen that the CR removal gradually increases upto 40 g/L and after which it remains constant. Since, the removal of CR is constant after 14 g/L and there is no further change with adsorbent dosage, the equilibrium state has been considered at this dose and the dose, 40 g/L was considered as the optimum dose (*m*). An increase in the adsorption with adsorbent dose can be attributed to greater surface area and the availability of more adsorption sites (Ardejani et al. 2008, Lataye et al. 2008, 2009).

Effect of initial pH (pH0)

The pH of solution affects the surface charge of adsorbent as well as the degree of ionization of the materials present in the solution. It is a common observation that the surface adsorbs anions favorably

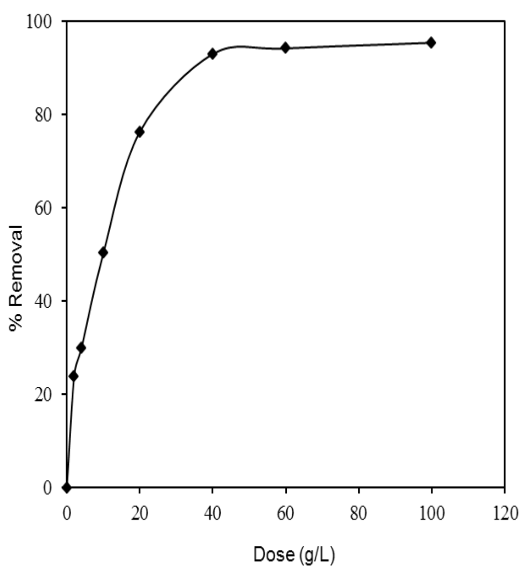

Fig 2: Effect of adsorbent dose on the adsorption of CR by orange peel (C_0 = 100 mg/L, Volume of sample = 50 mL, Temperature = 30 °C, RPM = 150, Time = 3 h and pH_0 = 6.43 (Natural).

at lower pH due to the presence of H^+ ions, whereas, the surface is active for the adsorption of cations at higher pH due to the deposition of OH^- ions. The pH of solution showed very minute decrease in the removal of CR with increasing pH. The slight decrease in removal of CR with increasing pH may be due to the increasing effect of repulsion after the point of zero, which is 6.37 in the present case, due to increasing negative charge after pH_{PZC} on the adsorbent surface. There was no much change in colour, around natural pH (pH_0 = 6.43) hence, all further experiments were conducted at natural pH 6.43. During the adsorption process, it is observed that the final pH of solution was decreased after adding the adsorbent dosage. The initial pH of the solution was 6.43 which has become <6. The pH of the solution after adding adsorbent decreases due to surface becoming negative charged by adsorbing OH^- ions from the solution (Uddin et al. 2009). The effect of pH on CR removal is shown in Figure 3.

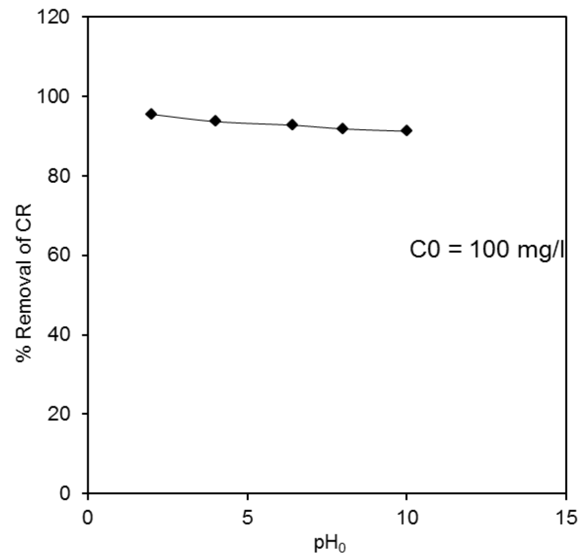

Fig 3 The effect of pH on the removal of CR by orange peels (C_0 = 100 mg/L, V = 50 mL, m = 40 g/L and t = 1 h)

Effect of initial dye concentration (C0) and temperature (T)

The effect of C_0 on the removal of CR by orange peel is shown in Figure 4. It is evident from the figure that the amount of CR adsorbed per unit mass of adsorbent (q_e) increased with the increase in C_0, although percentage CR removal decreased with the increase in C_0 (Figure 3). The adsorption capacity, q_e increases with the increase in C_0, as the resistance to the uptake of CR from the solution decreases with the increase in concentration. The rate of adsorption also increases with the increase in C_0 due to increase in the driving force. Also the figure shows that the adsorption of the dye increases with respect to temperature which indicates the endothermic process. Temperature has a pronounced effect on the adsorption capacity of the adsorbents. Figure 5 shows the plots of adsorption isotherms, for azo dye (congo red, direct red 28) orange peel systems at 20, 30 and 40 ºC. It shows that the adsorptivity of CR increases with the increase in temperature. The increase in sorption capacity of the other adsorbents with the increase in temperature has also been reported by other investigators (Prado et al., 2003, Lataye, 2006, 2011).

Effect of contact time (t)

The effect of contact time (*t*) on the removal of CR by orange peel at C_0 =100 mg/L is shown in Figure 5. There is a rapid adsorption of CR in first 30 min, thereafter, the adsorption rate decreases gradually and the adsorption reaches equilibrium in 3 h. Increase in contact time showed that the CR removal increases slightly over those obtained for optimum contact time. The adsorption curve indicates the possible mono-layer coverage of dye on the surface of orange peel (Wong and Yu, 1999; Malik, 2003).

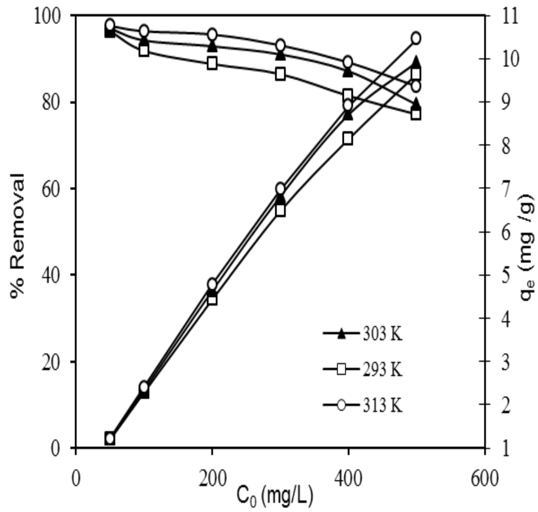

Fig. 4: Effect of initial concentration on percent removal of CR by orange peel at different temperatures (m = 40 g/L, V = 50 ml, t = 3 h, pH_0 = 6.43, RPM = 150).

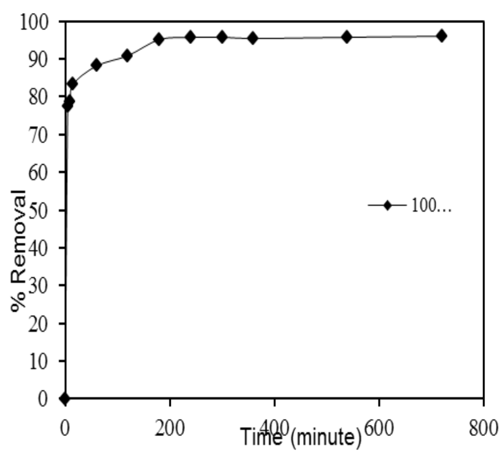

Fig. 5 Effect of contact time on percent removal of CR (C_0 = 100 mg/L, m = 40 g/L, T = 30 deg. pH_0 = 6.43, RPM = 150).

Adsorption equilibrium study

To find the adsorption capacity of adsorbent, it is important to establish the most appropriate correlation for the equilibrium isotherms. Langmuir, Freundlich and Temkin isotherm equations at 293, 303 and 313 K have been used to describe the equilibrium characteristics for adsorption of CR by orange peel.

The adsorption isotherm derived by Langmuir for the adsorption of a solute from a liquid solution is given as (Lataye et al. 2006):

$$q_e = \frac{q_m K_A C_e}{1 + K_A C_e} \qquad (2)$$

where,

q_e = Equilibrium solid phase concentration of CR (mg/g)

q_m = Amount of adsorbate adsorbed per unit wight of adsorbent required for monolayer adsorption (limiting adsorbing capacity, mg/g).

K_A = Constant related to enthalpy of adsorption.

C_e = Equilibrium concentration (mg/L)

The Langmuir isotherm equation can be rearranged to the following linear forms:

$$\frac{C_e}{q_e} = \frac{1}{K_A q_m} + \frac{C_e}{q_m} \qquad (3)$$

$$\text{or} \quad \frac{1}{q_e} = \left(\frac{1}{K_A q_m}\right)\left(\frac{1}{C_e}\right) + \left(\frac{1}{q_m}\right) \qquad (4)$$

The Langmuir constants can be determined from the plot between C_e versus C_e/q_e (Figure 6).

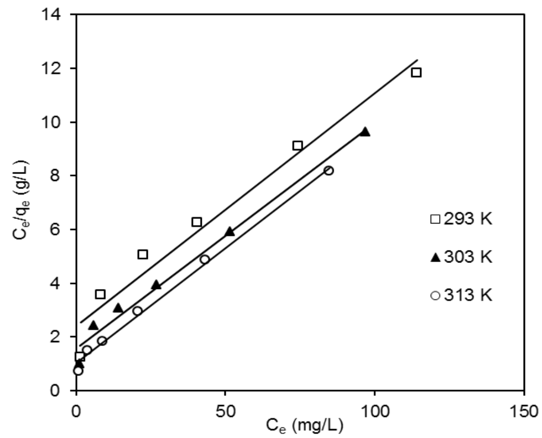

Fig. 6 Langmuir isotherm Plot (Adsorbent dose, m = 40 g/L, V = 50 mL, t = 3 h, pH0 = 6.43, RPM = 150)

The Freundlich Isotherm, given as (Lataye et al. 2006):

$$q_e = K_F C_e^{\frac{1}{n}} \qquad (4)$$

where ,

K_F and n are the constants

C_e = Equilibrium concentration (mg/L)

By taking logarithm on both sides, this equation (4) is converted into a linear form:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \qquad (5)$$

Temkin isotherm is represented by the following equation (Lataye et al. 2006):

$$q_e = \frac{RT}{b} \ln(K_T C_e) \qquad (6)$$

Equation (6) can be expressed in its linear form as:

$$q_e = B_T \ln K_T + B_T \ln C_e \qquad (7)$$

Thus a plot between $ln\ q_e$ and $ln\ C_e$ is a straight line(Figure 7).

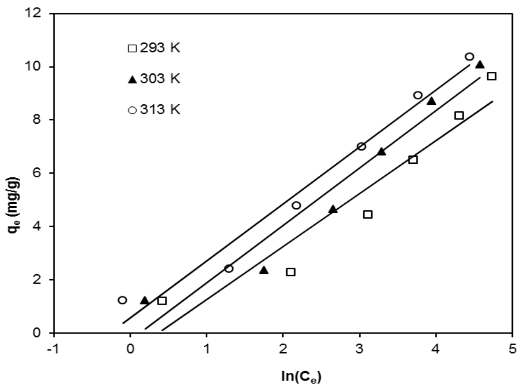

Fig. 7 Freundlich isotherm Plot (Adsorbent dose, *m* = 40 g/L, V = 50 mL, t = 3 h, pH0 = 6.43, RPM = 150)

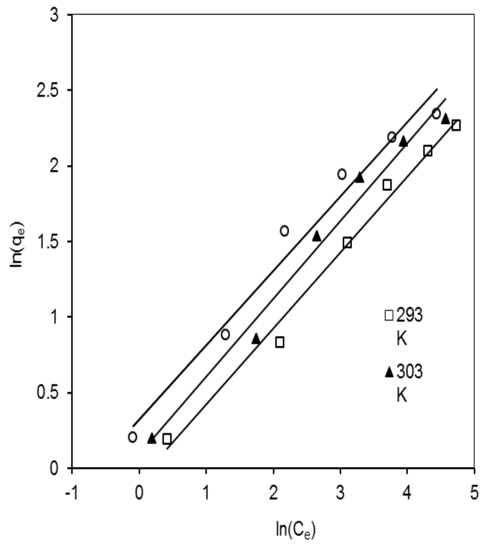

Fig. 8 Temkin isotherm Plot (Adsorbent dose, *m* = 40 g/L, V = 50 mL, t = 3 h, pH0 = 6.43, RPM = 150)

where, $B_T = \frac{RT}{b}$ and is related to the heat of adsorption. b and K_T is the equilibrium binding constant (L/mg) corresponding to the maximum binding energy. A plot of q_e versus lnC_e enables the determination of the isotherm constants K_T and B_T (Figure 8).

Figure 9 shows the comparison of fitting of these isotherm equations. The equilibrium constants calculated are shown in Table 2. From the study it is found that, Langnuir isotherm fits the data well followed by Freundlich isotherm. Temkin isotherm equation does not fit the isotherm data well. The Chi square test (χ^2) error analysis function has been used to compare the adsorption results, which is given as follows:

$$\chi^2 = \sum \frac{(q_{e,\exp} - q_{e,calc})^2}{q_{e,calc}} \tag{8}$$

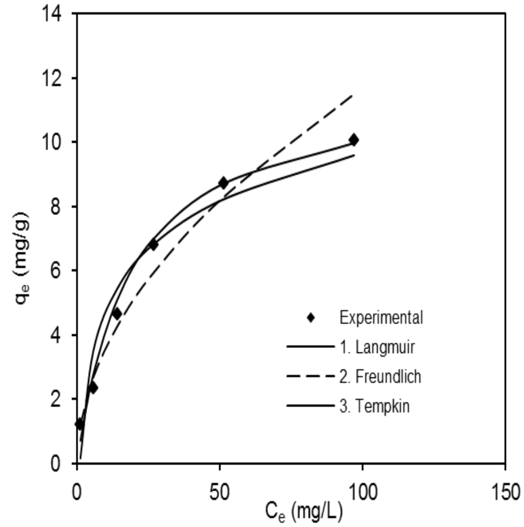

Fig. 9 Experimental and Calculated q_e Vs C_e.

Thermodynamic study

The Gibbs free energy change of the adsorption process is related to the equilibrium constant given by the classic Van't Hoff equation (Lataye et al. 2006):

$$\Delta G = -RT \ln K \tag{9}$$

According to thermodynamics, the Gibbs free energy change is also related to the entropy change and heat of adsorption at constant temperature by the following equation:

$$\Delta G = \Delta H - T\Delta S \tag{10}$$

Combining above two equations, we get

$$\ln K = \frac{-\Delta G}{RT} = \frac{\Delta S}{R} - \frac{\Delta H}{R}\frac{1}{T} \tag{11}$$

Where ΔG is the free energy change (kJ/mol), ΔH is the change in enthalpy (kJ/mol), ΔS is the entropy change (kJ/mol K), T is the absolute temperature (K) and R is the universal gas constant (8.314 J/mol K). Thus ΔH can be determined by the slope of the linear Van't Hoff plot i.e. as $\ln K$ versus $(1/T)$, using equation:

$$\Delta H = \left[R \frac{d \ln K}{d(1/T)} \right] \tag{12}$$

Van't Hoff's plot for Langmuir isotherm models for CR-orange peel systems is given in Figure 10. ΔG, ΔH and ΔS as calculated from Langmuir models are given in Table 3.

An increase in temperature induces a positive effect on the sorption process. The heat of adsorption (ΔH) was found to be 21.475 KJ/mol and change in entropy (ΔS) was found 0.149 KJ/mol K. The negative values of change in Gibbs free energy (ΔG) indicate the feasibility and spontaneity of adsorption of CR on orange peel.

TABLE 2
Isotherm constants

Isotherms	Constants	Temperatures (Kelvin, K) 293	303	313
Langmuir	K_A, L/mg	0.036	0.053	0.082
	q_m, mg/g	11.561	11.919	11.765
	R^2 (Linear)	0.962	0.985	0.994
	R^2 (Non-Linear)	0.981	0.992	0.997
	χ2	0.819	0.466	0.274
Freundlich	K_F, L/mg	0.925	1.098	1.384
	1/n	0.501	0.514	0.490
	n	1.994	1.947	2.041
	R^2 (Linear)	0.989	0.982	0.974
	R^2 (Non-Linear)	0.995	0.991	0.987
	χ2	0.118	0.393	0.570
Temkin	B_T	1.988	2.150	2.136
	K_T, L/mg	0.697	0.893	1.314
	R^2 (Linear)	0.916	0.942	0.970
	R^2 (Non-Linear)	0.957	0.970	0.985
	χ2	12.000	6.985	2.212

TABLE 3
Thermodynamic parameters for adsorption of CR by Orange peel

ΔG (KJ/mol) 20 °C	30°C	40°C	*ΔH* (KJ/mol)	*ΔS* (KJ/mol K)
-24.633	-26.548	-28.464	21.475	0.149

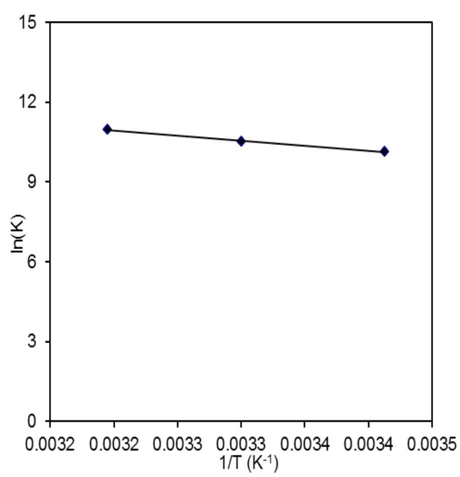

Fig. 10 Van't Hoff's plot

IV. CONCLUSION

From the characterization of orange peel, the moisture content, ash content, volatile matter, and fixed carbon were found to be 4.8 %, 2.98%, 79.14 %, and 13.08% respectively. The point of zero charge (pH_{PZC}) was found 6.37. From the adsorption study the optimum adsorbent dose was found 40 g/l. The pH study revealed that, there is very slight effect on the pH of solution and the removal found slightly reduced with increasing pH. The equilibrium time for present study was found to be 3h. The percent removal of congo red dye found decreasing with in initial concentration, however the amount of dye adsorbed per unit gram of adsorbent increases. The isotherm analysis shows that the Langmuir isotherm is best-fitted for CR adsorption on orange peel. The values of the constants K_L and q_m are 0.053 L/mg and 11.919 mg/g, with the linear and nonlinear correlation coefficients of 0.985 and 0.992 respectively at 30 ºC. Adsorption capacity of orange peel for CR removal increases with increase in temperature for the adsorbent, which shows the endothermic nature of adsorption process. The heat of adsorption (Δ*H*) was found to be 21.475 KJ/mol and change in entropy (Δ*S*) was found 0.149 KJ/mol K. Gibbs free energy (Δ*G*) was found -24.633, -26.548 and -28.464 kJ/mol. The negative values Δ*G* indicate the feasibility and spontaneity of adsorption process. Thus the orange peel can be used as an adsorbent for the removal of congo red dyes and other pollutants, which can be a good solution for the utilization solid waste remove pollutants from the waste water or water.

Abbreviations

1/n - Heterogeneity factor, dimensionless
q_e - Equilibrium solid phase concentration of CR (mg/g),
C_0 - Initial concentration of CR in solution (mg/L),
C_e - Equilibrium concentration (mg/L),
W - Adsorbent dose (g/L),
V – Volume of sample in litre (L).
q_m = Amount of adsorbate adsorbed per unit wight of adsorbent required for monolayer adsorption (limiting adsorbing capacity, mg/g).
K_A = Constant related to enthalpy of adsorption.
B_1 - constnat in Temkin isotherm equation
K_F - Constant of Freundlich isotherm, L/mg
K_T – Temkin equilibrium binding constant (L/mg)
$q_{e,cal}$ - Calculated Equilibrium solid phase concentration of CR (mg/g)
$q_{e,exp}$ - Experimental Equilibrium solid phase concentration of CR (mg/g)
R - universal gas constant (8.314 J/mol K).
T - absolute temperature, K
W - Adsorbent dose, g/L, *V* - Volume in litre
ΔG - the free energy change (kJ/mol)
ΔH - the change in enthalpy (kJ/mol)
ΔS is the entropy change (kJ/mol K)
χ = Chi error function

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