Utilization of Hen Feathers for the Adsorption of Indigo Carmine from Simulated Effluents

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Abstract

The aim of this study is to exploit the hen feathers as adsorbent for the removal of a hazardous dye, Indigo Carmine from simulated aqueous solutions of the dye. Effects of various experimental parameters like pH of the solution, initial concentration of the dye, amount of adsorbent, temperature and contact time have been investigated using a batch adsorption technique to obtain information on treating simulated effluent of the dye. The sorption data was then correlated with Langmuir, Freundlich and D-R adsorption isotherm models. The kinetics of Indigo Carmine adsorption on hen feathers has also been studied by fitting the data in Lagergren's first order and Ho-McKay's pseudo second order kinetics hypothesis. It has been observed that the removal of Indigo Carmine over hen feathers undergoes via first order process and at all the concentration of the dye film diffusion mechanism operates.

Keywords: Indigo Carmine, Hen Feathers, Adsorption, Dye, Kinetics, Adsorption Isotherm.

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Introduction

Colored materials and dyes constitute the focus of many environmental concerns because of their non-biodegradable and polluting nature [1, 2]. Thus prior to their discharge into receiving waters, there is a considerable need to treat colored effluents efficiently [3 – 5]. There exist several physicochemical processes for removal and recovery of colored materials and dyes from effluents, out of which the adsorption is one of the most effective one [6 – 8]. The important aspect of adsorption processes is easy regenerability and less operational cost. Adsorption techniques have proven successful in removing colored organic species and choice of adsorbent is one of the key factors determining the effectiveness of any adsorption process. In recent years, activated carbon has been found most widely used adsorbent, as it possesses high capacity for the adsorption of organic materials [9 – 11]. However, due to high cost of activated carbon scope of many adsorption studies has been focused to derive cheaper adsorbents from the waste materials. Thus cheap and effective adsorbents have been developed from various waste materials such as chitin and chitosan [12], peat [13], rice husk [14], clay [15], bottom ash and de-oiled soya [16 – 18], coal ash [19], crushed bricks [20], sugar beet pulp [21], tea waste [22], feathers [23, 24] etc. Gupta and coworkers [25 – 30] have made substantial contributions in this area. Literature survey also reveals that amongst waste material adsorbents used for the removal of dyes, metals and other pollutants from the industrial effluents, hen feather possesses better potentiality [31 – 33]. Feathers represent four to six percentage of the total weight of hen and are generated in huge quantities as a waste by-product at commercial poultry processing plants. Presently, between two to four billion pounds of feathers are produced annually by the poultry processing industries. These feathers pose a disposal problem and are usually converted to animal feed via hydrolyzation [34]. Unlike activated carbons, feather is a relatively cheap material and available easily. Moreover, it is easier to handle and exhibits excellent adsorbing ability [35].

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In the present research, hen feathers have been employed as an adsorbent for the removal of a toxic, indigoid class of cationic dye - Indigo Carmine. Indigo Carmine is widely used in food, drugs and cosmetic industries. It is commonly administered to facilitate diagnosis of Barrett's esophagus [36] and helps to target biopsies [37]. Indigo Carmine is also used for staining purpose and contributes to intra-vital staining for contrasting and accentuating changed mucosal processes [38]. Despite several uses of Indigo Carmine, it is also toxic to the human being. In contact with eyes it can cause permanent injury to cornea and conjunctiva. Life-threatening anaphylactoid reaction to this dye has also been reported, which may be either due to drug allergy or intrinsic serotinergic properties [39]. It causes strong clastogenic activity on bone marrow chromosomes of mice [40] and also found to cause acute hemodynamic effects [41].

Thus the removal of Indigo Carmine from wastewater needs considerable attention. In the present study, hen feathers have been utilized as adsorbent for the removal of Indigo Carmine from simulated aqueous samples. During the present study, various effects have been observed on the adsorption of the Indigo Carmine over Hen Feathers, these include – effects of initial concentration of the dye, pH of the dye solution, amount of adsorbent, temperature etc. The sorption data has also been correlated with adsorption isotherms and kinetics of adsorption has been studied to determine the efficiency of adsorption process. A suitable mechanism operating at different concentrations of the dye has also been suggested for the on-going adsorption.

Materials & Methods

Indigo Carmine (Figure 1), which is also commonly known as Acid Blue 74 or Food Blue 1 or FD & C Blue 2 (CASRN 860-22-0), is a dark blue, water-soluble powder. The molecular formula of the dye is $C_{16}H_8O_8N_2S_2Na_2$ and molecular weight is 466.36. For the present studies Indigo Carmine is procured from M/s Merck. Adsorbent – Hen Feather was collected from the local poultry.

A stock of solution of 1×10^{-2} M concentration of Indigo Carmine was prepared in double distilled water and all working solutions of desired concentrations were prepared by the stock solution. The microprocessor based pH meter; model number HI 8424 (M/s Henna Instruments, Italy) was used to measure the pH of the solutions. Concentrations of the aqueous solutions of dye were monitored on UV/Visible spectrophotometer, Model 117 (M/s Systronics, Ahmedabad, India) over a wavelength range of 610 nm.

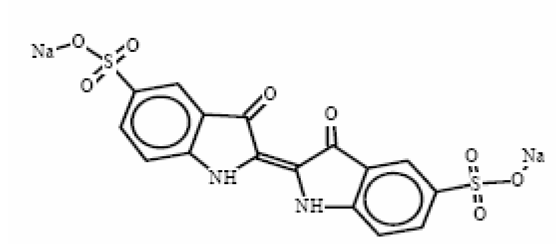

Figure 1. Chemical Structure of Indigo Carmine.

Preparation of adsorbant

Before using hen feathers as adsorbent, these were subjected to thorough washing with detergent and then rinsed with distilled water, several times. With the help of scissors, small size barbs of each feather were carefully cut, excluding the rachis. The barbs were then treated with hydrogen peroxide (30% w/v) for about 24 hours to remove adhering organic materials. The obtained mass was once again washed in the pool of distilled water and dried in the oven, overnight. The dried material thus obtained was then stored in desiccators for subsequent studies.

Feather has protein contents of around 84 percent along with the inorganic constituents like Calcium, Magnesium, Selenium, Zinc, etc. Nevertheless, raw feathers are relatively insoluble and have a very low digestibility of five percent due to the high keratin contents and the strong disulphide bonding of the amino acids. The constituents of Feather have been clearly portrayed in Table 1.

Table 1. Chemical Constituents of Hen Feathers

Constituents	*Percent by weight*
Dry matter	90
Crude protein	82
Fat	6
Ash	4
Crude fibre	0.6
Available lysine	1.8
Methionine + cysteine	4.9

Adsorption studies

To study the effect of important parameters like initial concentration, pH, amount of adsorbent, temperature and time for the adsorptive removal of Indigo Carmine, batch experiments were conducted. For each experimental run, 25 ml of the dye solution of known initial concentration, initial pH and amount of feathers were taken in 100 mL stoppered measuring flask and was kept in temperature controlled water bath. The mixture was intermittently shaken and then kept for 24 hours for saturation. Thereafter supernatant liquid was filtered through Whattmann Filter Paper No.42 and the amount of dye adsorbed was determined spectrophotometrically at the Indigo Carmine λ_{max} 610 nm. The amount of the dye adsorbed at equilibrium (q_e) was calculated by following equation:

$$q_e = \frac{(C_o - C_e)\,V}{W} \qquad (1)$$

Where C_o and C_e (g/L) are the liquid-phase concentrations of the dye respectively at initial time (t = 0) and equilibrium, V is the volume of the solution in liter and W is the mass of the adsorbent used (g).

Kinetic studies

To carry out kinetic measurements, in different measuring flasks 25 mL of dye solution of known concentration was taken with a definite amount of adsorbent at temperatures 30, 40 and 50 ^oC and constant pH. Each flask was agitated for a fixed duration and solutions were then filtered using Whattmann Filter Paper No. 42. The filtered solution was evaluated spectrophotometrically to observe the amount of adsorbed Indigo Carmine.

Results & Discussion

Adsorption studies

Adsorption characteristics of Indigo Carmine by Feathers were studied at varying pH range from 2 to 6. The profile (Figure 2) regarding pH shows that in the observed pH range the adsorption capacity was much pronounced at lower pH 2, while as the pH increases adsorption decreases. Thus all the successive studies were performed at pH 2.0. Higher adsorption at lower pH may be due to increased protonation by the neutralization of the negative charges at the surface of the adsorbent, which facilitates diffusion process and provides more active sites of the adsorbent. While with increase in the pH of the solution protonation reduces, which thereby retards diffusion and adsorption.

The adsorption of Indigo Carmine was also recorded in the concentration range from 1×10^{-4} to 10×10^{-4} M, at a fixed pH of 2 and temperatures 30, 40 and 50 ºC, respectively (Figure 3). It was observed that the initial removal of dye is faster and with the rise in concentration the percentage uptake gradually decreases. Figure 3 also exhibits that the adsorption of Indigo Carmine by Feathers increases with the increase in temperature, indicating thereby the process to be endothermic in nature.

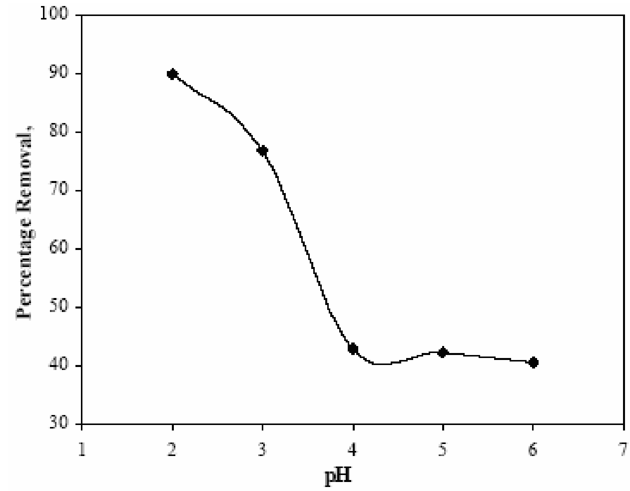

Figure 2. Effect of pH on Uptake of Indigo Carmine by Hen Feathers at 50 ^oC.

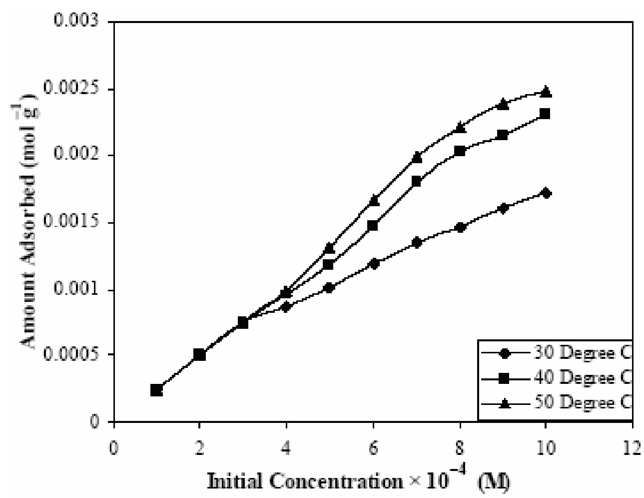

Figure 3. Effect of Concentration for the Uptake of Indigo Carmine on Hen Feathers at Different Temperatures.

The adsorption profiles obtained at different temperatures were then allied with the Freundlich, Langmuir and Dubinin-Radushkevich (D-R) adsorption isotherm models. Linear plots Figures 4 (Freundlich isotherms), 5 (Langmuir isotherms) and 6 (DR isotherm) were obtained at different temperatures, which reveal the applicability of the isotherms on the on-going adsorption process. These plots were then used to evaluate the values of Freundlich, Langmuir and D-R constants using equations (2), (3) and (4), respectively.

$$\log q_e = \log K_F + 1/n \log C \quad (2)$$

$$1/q_e = 1/Q_o + 1/(bQ_oC) \quad (3)$$

$$\ln q_e = \ln X_m - \beta.\varepsilon^2 \quad (4)$$

where, C is the molar concentration in bulk-fluid phase, q_e is the number of moles of solute adsorbed per unit weight at concentration C. In equation (2) K_F and n are the Freundlich constants. The Freundlich equation is basically empirical but is often useful as a means for the data description. In equation (3), b and Q_o are the Langmuir constants. While in equation (4), X_m is the maximum sorption capacity, β *is* the activity coefficient related to mean sorption energy, and ε *is* the Polanyi potential, which is equal to:

$$\varepsilon = RT. \ln (1 + q_e^{-1}) \quad (5)$$

where R is the Gas constant and T is temperature in Kelvin.

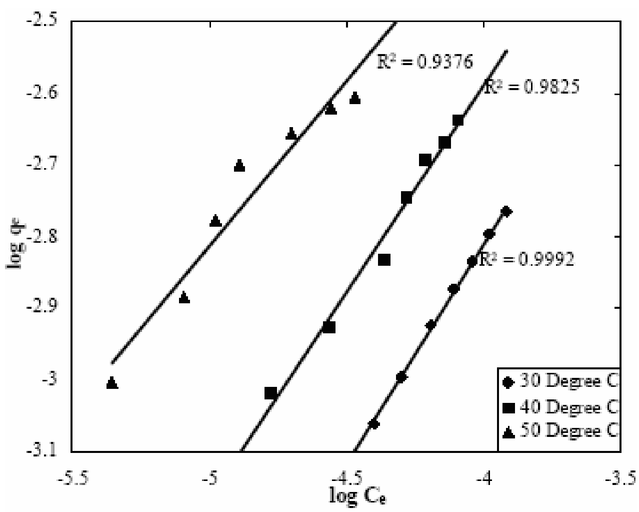

Figure 4. Freundlich Adsorption Isotherm for Indigo Carmine – Hen Feather System.

Values of Freundlich, Langmuir and D-R isotherm constants are presented in Table 2. The adsorption capacity was found to increase with rising temperature, which is further confirmed by the decline in Q^o values with decreasing temperature. This, once again reveals the endothermic nature of the ongoing process. To examine the progression of adsorption dimensionless constant, separation factor 'r' [42] was calculated by following equation:

$$r = \frac{1}{1 + bC_o} \quad (6)$$

where, b and C_o values were derived from Langmuir Isotherm.

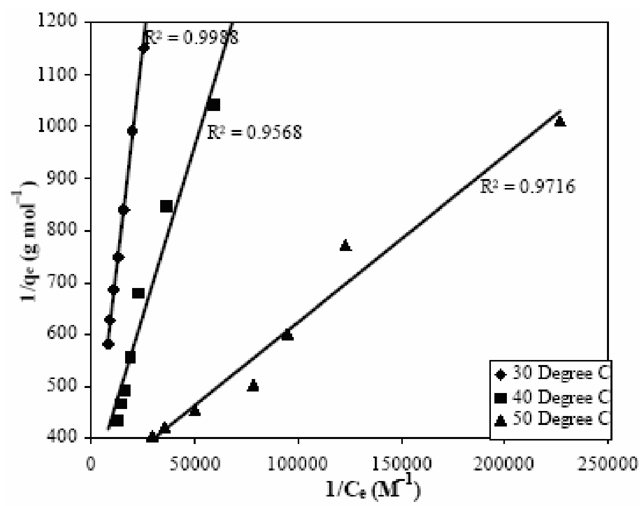

Figure 5. Langmuir Adsorption Isotherm for Indigo Carmine – Hen Feather System.

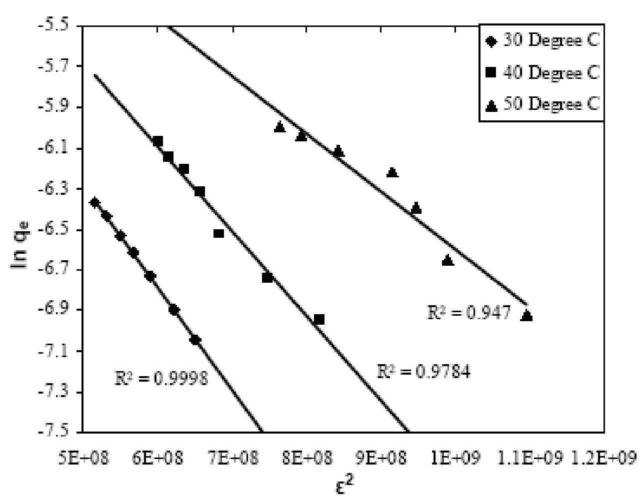

Figure 6. D-R Adsorption Isotherms for Indigo Carmine – Hen Feather System.

Table 2. Langmuir, Freundlich, and D-R Constants of Indigo Carmine Adsorption by Hen Feathers at Different Temperatures.

Langmuir constants			
Temperature	30°C	40°C	50°C
$Q_o \times 10^{-3}$ (Mol/g)	3.205	3.250	3.333
$b \times 10^{2}$ (L/Mol)	92.875	236.700	939.594
Freundlich constants			
Temperature	30°C	40°C	50°C
n	2.272	1.837	1.266
K_F	0.226	0.363	2.734
D-R constants			
Temperature	30°C	40°C	50°C
β	3×10^{-9}	4×10^{-9}	5×10^{-9}

Values of separation factor were calculated by equation (6) and found less than unity at all the temperatures, which indicates a favourable adsorption process in each. It has also been observed that 'r' values decrease with rising temperatures. This further indicates that the on-going adsorption process is more favorable at higher temperature.

The thermodynamic data were evaluated from Langmuir isotherms using following equations:

$$\Delta G^o = -R\,T \ln b \qquad (7)$$

$$\Delta H^o = R \frac{T_2\,T_1}{T_2 - T_1} \ln \frac{b_2}{b_1} \qquad (8)$$

$$\Delta S^o = \frac{\Delta H^o - \Delta G^o}{T} \qquad (9)$$

where, b, b_1 and b_2 are the equilibrium constants at the temperature 30, 40 and 50 °C respectively.

Evaluated thermodynamic parameters, change in free energy (ΔG^o), change in enthalpy (ΔH^o) and change in entropy (ΔS^o) are presented in Table 3. Negative values of ΔG^o establish the feasibility of adsorption process. Further, the decrease in the values of ΔG^o with the increasing temperature indicates the spontaneity of the process at higher temperatures. The endothermic nature was also confirmed from the positive values of enthalpy change (ΔH^o), while good affinity of dye towards the adsorbent material is revealed by the positive value of ΔS^o.

Table 3. Values of Thermodynamic Parameters for the Adsorption of Indigo Carmine on Hen Feathers.

$-\Delta G^o$ (kJ mol^{-1})			ΔH^o	ΔS^o
30°C	40°C	50°C	(kJ.mol^{-1})	(JK^{-1} mol^{-1})
23.02	26.21	30.75	94.60	387.41

To evaluate nature of on-going adsorption, energy of sorption (E) was evaluated from the values of β, the activity coefficient obtained by the slopes of the straight lines of D-R isotherms (Figure 6) using following expression:

$$E = (-2\,\beta)^{-1/2} \qquad (10)$$

The values of energy of absorption are found as 10.00, 11.18 and 12.91 kJ/mol at 30, 40 and 50 °C temperatures. These values are falling within the range of values 8 - 16 kJ/mol, indicating thereby involvement of chemical adsorption in the present case [43, 44].

Kinetic studies

For effectual designing and representation of the ongoing process the study of kinetics of the adsorption was carried out, which involves effect of some major parameters like contact time, amount of adsorbent and concentration of adsorbate solution.

Preliminary studies suggested that almost four hours were sufficient to attain the equilibrium (Figure 7).

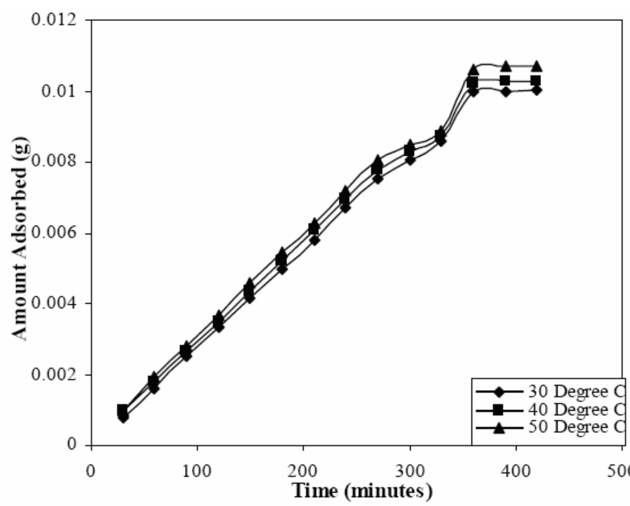

Figure 7. Effect of Contact Time for the Adsorption of Indigo Carmine by Hen Feathers at Different Temperatures and at a Concentration of 1×10^{-3} M.

Moreover, the kinetics of adsorption process at different temperatures (30, 40 and 50 ^oC) exhibited an increase in adsorption with the increase in temperature. The half-life of each process was also calculated and was found to decrease with increase in temperature. These results once again confirm endothermic nature of the on-going process.

Kinetic adsorption studies were carried by varying the adsorbent dosage. It has been observed that when amount of adsorbent is increased from 0.005 to 0.05 g the amount of the adsorbed dye increases from 0.003 to 0.011 g. This indicates an increase in rate of removal with increasing dosage of adsorbent. The half-life of the process was also calculated and found to decrease from 53.80 to 7.21 hour with the increase in dosage of the hen feather from 0.005 to 0.05 g.

Adsorption rate constant study

In order to study the specific rate constant of Indigo Carmine – Hen Feather system, the well-known Lagergren's first-order rate equation was employed [45]. Values of log (q_e – q_t) were calculated for each time interval at different temperatures.

$$\log (q_e - q_t) = \log q_e - \frac{k_{ad}}{2.303} \times t \qquad (11)$$

where, q_e and q_t signify the amount adsorbed at equilibrium and at any time t. The graph of log (q_e – q_t) vs time (Figure 8) exhibits straight lines at 30, 40 and 50 ^oC and confirms the first-order rate kinetics for the on-going adsorption process. The k_{ad} values evaluated, from Lagergren plots are found to be 0.00622, 0.00622 and 0.00576 sec^{-1} at 30^{o}C, 40^{o}C and 50^{o}C, respectively.

To further verify the order of the process following Ho – Mckay's pseudo second order rate expression was also applied [46]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (12)$$

where, q_e and q_t are adsorption capacities at equilibrium and time t ($mg.g^{-1}$), respectively and k_2 is the rate constant of the pseudo second order adsorption ($g.mg^{-1}.sec^{-1}$). The graphs between t and t/q_t did not give proper straight lines and obtained regression coefficient values were also found very low. However, R^2 values are close to unity in case of Lagergren plots (Figure 8). Thus it is safely concluded that the adsorption of Indigo Carmine follows only first-order kinetics.

Rate limiting step in the process of dye uptake

In order to interpret the experimental data, it is necessary to identify the step that governs the overall rate of removal in the adsorption process. The mathematical treatments of Boyd et al. [47] and Reichenberg [48], that has laid the foundations of sorption/ion exchange kinetics, were used to distinguish between particle and film diffusion mechanism of adsorption process.

The kinetics of the adsorption process of Feathers was examined at two different concentrations (1 × 10^{-3} & 5 ×10^{-4} M) of Indigo Carmine and the values of Fractional Attainment of Equilibrium 'F' were calculated using equation (13).

$$F = \frac{Q_t}{Q_\infty} \qquad (13)$$

where, Q_t and Q_∞ are amounts adsorbed after time t and after infinite time respectively.

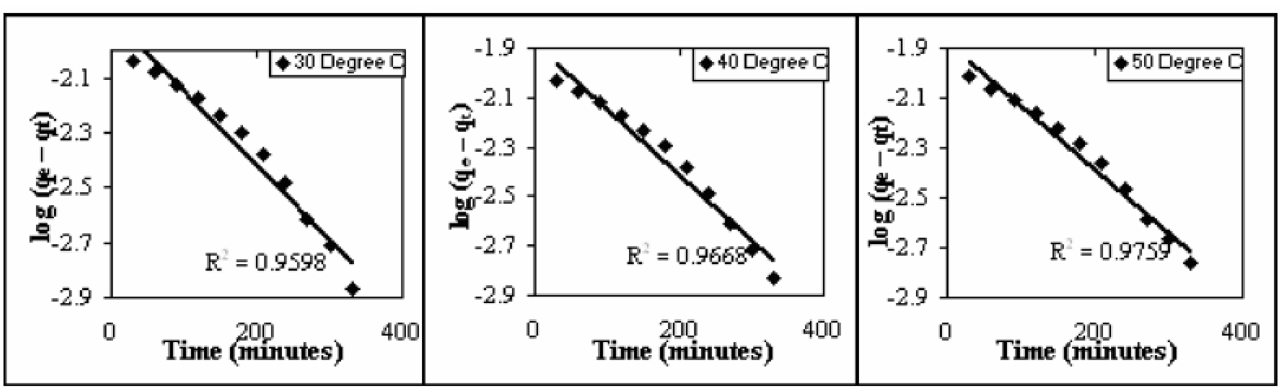

Figure 8. Lagergren First Order Plots for Indigo Carmine Adsorption on Hen Feathers at Different Temperatures.

$$F = 1 - \frac{6}{\pi^2} \sum_{1}^{\infty} (1/n^2) \exp(-n^2 B_t) \quad (14)$$

where, F is the fractional attainment of equilibrium at time 't', and n is Freundlich constant of the adsorbate. From Reichenberg's table [48] B_t values were obtained for each observed value of F and the results are plotted in Figure 9. The linearity test of B_t versus time plots was employed to distinguish between the film diffusion and particle diffusion controlled adsorption. The graph shows that for all the concentrations, film diffusion mechanism is active as the plots obtained shows deviation from linearity.

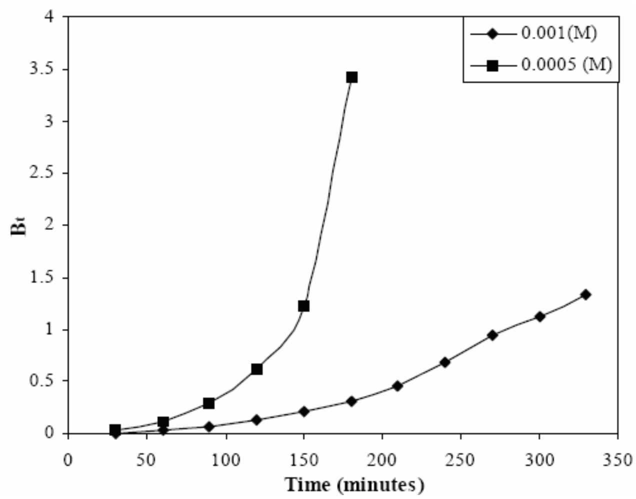

Figure 9. B_t Versus Time Plot for Indigo Carmine Adsorption on Hen Feathers at Different Concentrations at 50 °C.

CONCLUSIONS

The results presented in the paper clearly suggest that Hen Feathers can be efficiently used for the removal of Indigo Carmine, without posing any threat to the quality of water. Different operational parameters observed during the process of investigations reveal that the pH, temperature, contact time, adsorbent dose and concentrations of the adsorbate govern the overall process of adsorption. The batch studies clearly suggest that Feathers exhibit almost 100% adsorption at lower concentrations of 1×10^{-4} to 3×10^{-4} M, whereas at a dye concentration of 10×10^{-4} M removal of 96.6, 91.9 and 87.9% of the dye takes place, at 30, 40 and 50 °C temperatures, respectively. The results obtained are well fitted in the linear forms of Freundlich, Langmuir and D-R adsorption isotherms and adsorption of Indigo Carmine over Hen Feathers follows a chemical adsorption process. Thermodynamic parameters obtained accounts for the feasibility of the process at each concentration. The kinetic evaluation suggests that the uptake of dye follows first order rate equation and is controlled by film diffusion mechanism at all the concentrations. Thus it can be safely concluded that the Hen Feathers may be considered as a good replacement in place of commercially available adsorbents.

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