

Research Article

Removal of Phenol Red Dye From Contaminated Water Using Barley (*Hordeum vulgare* L.) Husk-Derived Activated Carbon

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Abstract

Background and Objective: Phenol red, a finishing textile dye, enters the water bodies through the industrial effluents. Due to its toxic nature, phenol red present in drinking water, is harmful to the human health. The high cost of activated charcoal restricts its use, as adsorbent, for the treatment of water contaminated with organic pollutants such as dyes. In the present study, low cost barley (*Hordeum vulgare* L.) husk-derived activated carbon has been used for the removal of phenol red dye from aqueous solution.

Materials and Methods: Chemical method was used for preparing activated carbon from barley husk powder. The synthesized material was characterized for surface area, morphology and elemental composition by pH-metric, XRD and EDX techniques. Using batch method, decoloration of phenol-red solution over synthesized activated carbon was monitored, spectro photometrically. The effects of parameters such as: adsorbent dosage, pH, adsorbate initial concentration, contact time and temperature on the removal of dye from its aqueous solution have been investigated. **Results:** Synthesized activated carbon was an amorphous powder with surface area $90.2 \text{ m}^2 \text{ g}^{-1}$. The observed data have been interpreted in the light of Langmuir and Freundlich adsorption isotherms. The pseudo second order kinetic model better fits for the adsorption of phenol-red on the studied adsorbent and the surface adsorption as well as intra-particle diffusion concurrently occur during the adsorbate-adsorbent interaction. Though the enthalpy of adsorption of phenol red is endothermic, yet the process becomes feasible due to the predominant entropy gain. **Conclusion:** The results show that barley husk derived activated carbon powder can be used as an efficient and cost effective adsorbent for the removal of phenol red dye pollutant from the contaminated water.

Key words: Adsorption, activated carbon, intra-particle, diffusion

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Effluents from dyeing, paper and textile industries, contaminated with non-degradable organic dyes, due to their mutagenic and carcinogenic effects, are not only a serious human health hazards but also cause adverse effect to aquatic life^{1,2}. Activated carbon, due to its micro porous structure, high specific surface area and reactivity, has an important role in adsorption process for removing toxic organic and inorganic chemicals from the contaminated water³. Activated carbon can be prepared by physical as well as chemical methods. Whereas, physical activation involves carbonization of carbonaceous materials followed by activation of the resulting char using gas activating agents, in chemical activation carbonization as well as activation takes place simultaneously in the presence of suitable chemical agents. Compared to physical activation, chemical activation occurs at lower temperature, provides more carbon yield of higher porosity⁴.

Due to the high cost involved in the use of activated charcoal for the removal of dyes from contaminated water, several efforts have earlier been made to explore cost-effective adsorbents. Fly ash generated from thermal power plant was used by Mohan *et al.*⁵ for the removal of crystal violet and basic fuschin dyes from wastewater. They observed that the fly ash had sorption efficiency comparable to that of other commercially available adsorbents. Arami *et al.*⁶ used orange peel, a low-cost and eco-friendly adsorbent as an ideal alternative to the current expensive methods for removing Direct Red 23 and Direct Red 80 dyes as model compounds from wastewater. Hebeish *et al.*⁷ treated sawdust with sandene 850 (polyamine) in alkaline medium. The cationized sawdust thus obtained was used, effectively, for removing Direct Red 23 dye from aqueous solutions.

Yadav *et al.*⁸ have reported the kinetics and equilibrium study of rhodamine-B dye adsorption at activated rice husk carbon in aqueous solution. They observed that the adsorption of the dye follows the first order kinetics and the endothermic adsorption process is made feasible due to large entropy gain. For the removal of reactive yellow 2 and reactive blue 4 dyes from contaminated water, Said *et al.*⁹ used propionic acid modified bagasse. They observed that the hydroxyl groups of bagasse and the carboxylic group of propionic acid play an important role in the removal of the dyes from polluted water. Jibril *et al.*¹⁰ used coconut shell activated carbon (CSAC) and commercial activated carbon (CAC) adsorbents for the removal of Tartrazine E102 dye from wastewater. Their results showed that CSAC has the potential as a low cost alternative for colour removal. In another study

by Rajasekhar¹¹, corncob was used as sorbent for the removal of malachite green dye from aqueous solution.

Recently Sharma *et al.*¹² have reviewed work on adsorption of textile dyes by plant biomass. However, to the best of our knowledge, no work has earlier been reported on the use of barley (*hordeum vulgare* L.) husk-derived activated carbon for the removal of phenol red dye from contaminated aqueous solution. Barley husk is a waste product of flour mills and beverage industries. The use of barley husk as a cost-effective adsorbent for the removal of pollutants will also contribute towards solving the solid waste disposal problem. Phenol red, a finishing textile dye and a pH indicator is also used in most of tissue culture media. pH-dependent cytotoxicity of contaminants of phenol red for breast cancer cells has, earlier, been reported by Grady *et al.*¹³.

The present study reports the synthesis and characterization of activated carbon derived from barley (*Hordeum vulgare* L.) husk. The as-synthesized material has been used as adsorbent for the removal of phenol red dye in aqueous solution. Effects of dye initial concentration, adsorbent dose and pH on the kinetics and adsorption of phenol red have been investigated.

MATERIALS AND METHODS

Materials

Chemicals: Phenol red (C₁₉H₁₄O₅S) (MW: 354.38 g mol⁻¹, SD fine chemicals), sulfuric acid (H₂SO₄) (MW 98 g mol⁻¹, Abron Chemicals), sodium bicarbonate (NaHCO₃) (MW 84 g mol⁻¹, BLULUX). Hydrochloric acid (HCl) (MW 36.5 g mol⁻¹, SD fine chemicals), sodium hydroxide (NaOH) (MW 40 g mol⁻¹, BLULUX), sodium chloride (NaCl) (MW 58.5 g mol⁻¹, BLULUX), Phenol red dye (C₁₉H₁₄O₅S) (MW 354.37 g mol⁻¹, Berckland Scientific Supply), Chemical structure of phenol red dye is given in Fig. 1.

Methods

Preparation of Barley husk activated carbon (BHAC): Barley husk was immersed in minimum quantity of conc. H₂SO₄ and kept overnight at room temperature. The product was soaked

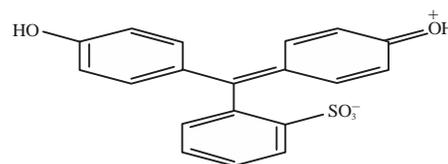


Fig. 1: Chemical structure of phenol red (Source: Wikipedia)

in 2% aqueous solution of NaHCO₃ for 24 h and then filtered. The residue was repeatedly washed with distilled water until it attained neutral pH and dried in hot air oven at 120°C for 10 h. The product was heated in a furnace at 500°C for an h, crushed in a mortar and fractionated into different mesh sizes.

Specific surface area of carbon: Surface area per g of barley husk derived activated carbon was obtained using Sears method¹⁴. As synthesized carbon powder (1.5 g) and NaCl (30 g) were added to 100 mL distilled water and stirred for 5 min. To this 0.1 N HCl was added to achieve final pH = 4.0 and then titrated against 0.1 N NaOH solution. The specific surface area was obtained using the formula:

$$A = 32.V-25 \quad (1)$$

Where, A = adsorbent's specific surface area (m² gm⁻¹), V = volume of 0.1 N NaOH required to raise the pH of reaction mixture from 4.0 to 9.0.

XRD analysis: The XRD pattern of synthesized carbon powder was carried out using X-ray diffractometer (BRUKER D8 Advanced XRD, West Germany) equipped with a Cu target emitting K_α radiation of wave length λ = 1.5405 Å. The measurements were made at room temperature using accelerating voltage and the applied current as 40 kV and 30 mA, respectively and scan rate 0.02° per sec over 2θ range 4-64°. The obtained XRD pattern was analyzed using DIFFPLUS, Eva, version 6.0 software.

Adsorption study: In a typical run, phenol-red dye solution (100 mL) of known concentration was mixed with a given amount of adsorbent in a 250 mL conical flask and magnetically stirred at room temperature and agitation rate 200 rpm. The reaction mixture, 5 mL each was withdrawn at regular interval, filtered using a whatman filter paper No. 1 and concentration of unabsorbed dye was determined at 435 nm using UV/Visible spectrophotometer (SP65). Percent of dye adsorbed is given by Eq. 2:

$$\text{Adsorption (\%)} (w/w) = \frac{(A_0 - A_t)}{A_0} \times 100 \quad (2)$$

RESULTS AND DISCUSSION

Physico-chemical properties of adsorbent: Physico-chemical properties such as bulk density, moisture and ash contents and specific surface area of synthesized activated carbon powder are presented in Table 1.

Table 1: Physico-chemical properties of barley husk activated carbon

Properties	Value
Bulk density (g cm ⁻³)	0.37
Moisture content (%)	1.95
Ash content (%)	1.63
Specific surface area (m ² g ⁻¹)	90.2

Table 2: Elemental composition in terms of weight percent (Wt %) and atom percent (At %) of synthesized barley husk derived activated carbon from EDX analysis

Elements	Wt (%)	At (%)
C	59.57	71.15
O	25.50	22.86
Si	2.00	01.02
S	6.21	02.78
Cl	3.49	01.41
Sn	1.59	00.19
Ca	1.65	00.59

XRD analysis: The XRD pattern of synthesized barley husk mediated activated carbon, used as adsorbent in the present study, is shown in Fig. 2. As no characteristic diffraction peak was observed, this suggests the amorphous nature of the adsorbent.

SEM analysis: Scanning electron microscopic (SEM) images of synthesized barley husk derived activated carbon powder at different magnifications presented in Fig. 3, clearly reveal its highly porous and non-uniform morphology with average particle size 6.7 μm.

Energy dispersive X-ray (EDX) analysis: Energy dispersive X-ray (EDX) spectra of synthesized barley husk derived activated carbon is exhibited in Fig. 4. The elements detected in the adsorbent are: C, O, Si, S, Cl, Sn and Ca, carbon being the major component followed by oxygen. The elemental composition in terms of weight percent (Wt %) and atom percent (At %) is given in Table 2.

Effects of parameters on adsorption

Effect of phenol red initial concentration: Plots of q_e (amount of phenol red adsorbed per g adsorbent) (mg g⁻¹) as a function of contact time (min) using varying initial concentrations (5-35 mg L⁻¹) of phenol red at the given adsorbent dose (5 g L⁻¹) and pH (2.0), are shown in Fig. 5.

Each plot exhibits initial increase of adsorption with contact time followed by an attainment of saturation, due to sorption-desorption equilibrium, at characteristic contact time suggesting the mono layer coverage of phenol red on the studied adsorbent. The amount of dye adsorbed and the time required to attain adsorption equilibrium, increase on increasing substrate (dye) initial concentration. It is obvious

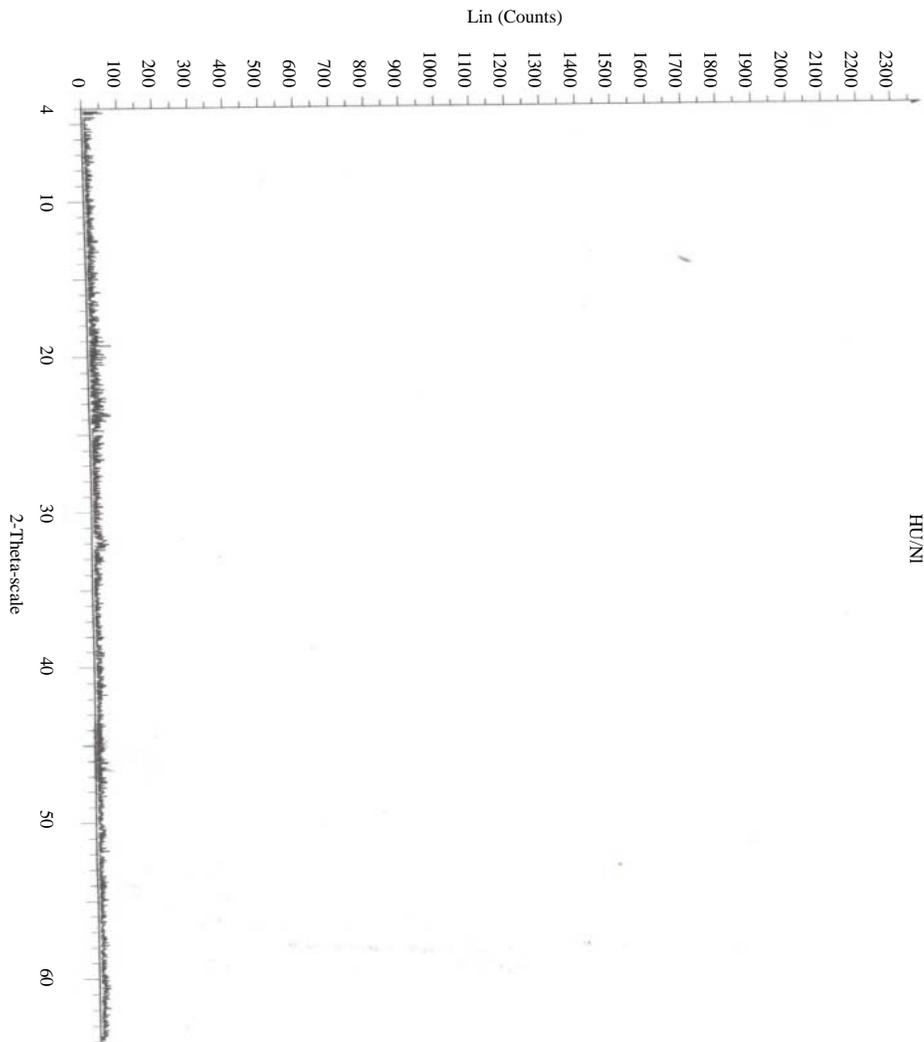


Fig. 2: X-ray diffraction of Barley Husk derived Activated Carbon

because at higher dye initial concentration, the number of interacting adsorbate molecules at the adsorbent surface, becomes higher.

Effect of pH on adsorption: Since the hydrogen and hydroxide ions are usually adsorbed quite strongly on the adsorbent surface thus the adsorption of the other ions is affected by the pH of the solution¹⁵. The pH also affects the degree of ionization of the dye as well as the surface properties of the adsorbent. Plot of Percent adsorption of phenol red dye at barley husk derived activated carbon as a function of pH is shown in Fig. 6. It is observed that (a) Adsorption of dye rapidly decreases on varying from 2 to 4, (b) The decrease in adsorption becomes slower in the pH range 4-8 and (c) A sharp dip in the adsorption over pH 8 to 10. Such unique pH dependent variation of dye

adsorption at the studied adsorbent may be due to the complex nature of both adsorbate's as well as adsorbent's net charge variation upon varying pH of the medium.

Effect of adsorbent dose: Plot of percent (w/w) of phenol-red dye adsorbed as a function of adsorbent load, using given initial concentration of the former and pH, is shown in Fig. 7. Adsorption of dye increases with the increasing adsorbent load over its studied range. It may be because on increasing the adsorbent load, its surface area and hence the availability of active sites/adsorbate (dye) molecule, increases^{16,17}. However, the increase of adsorption rate around 5 g L^{-1} slows down, an indication of the approaching optimum load of the adsorbent.

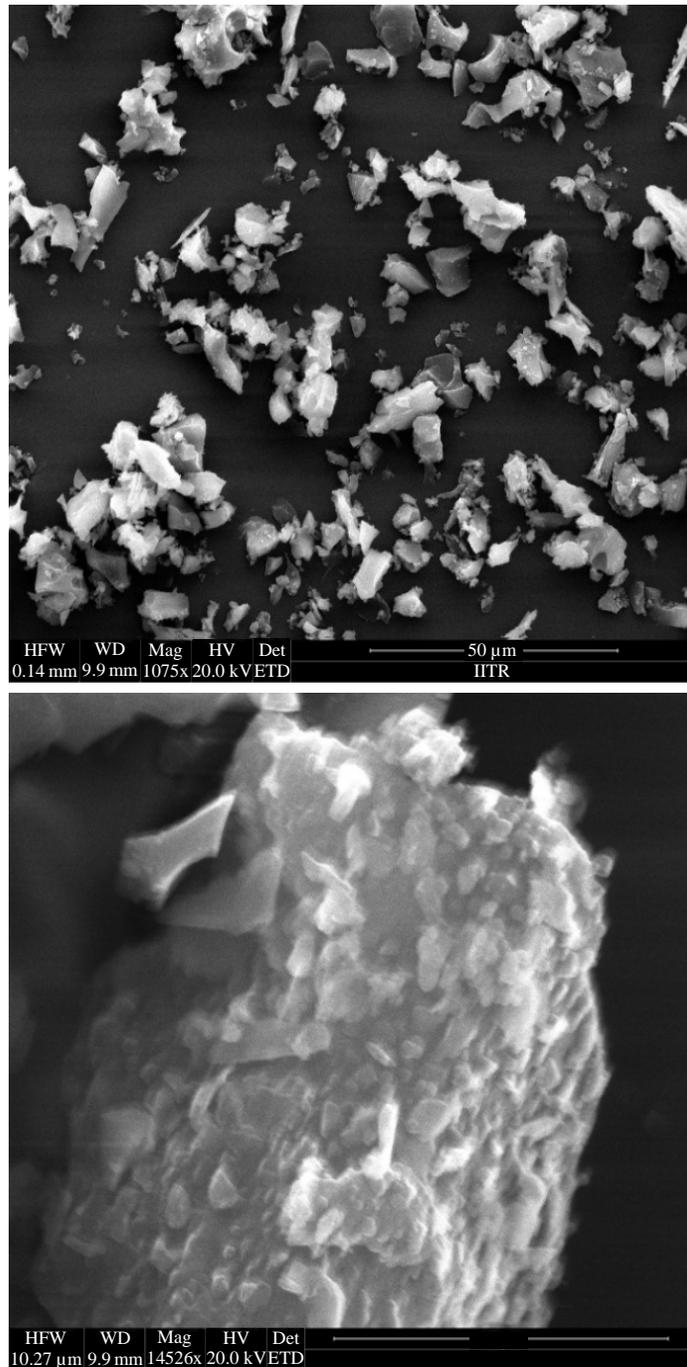


Fig. 3: Scanning electron microscopic (SEM) images of synthesized barley husk derived activated carbon powder at different magnifications

Adsorption isotherms models: The observed adsorption data was analyzed in the light of Langmuir's and Freundlich's adsorption isotherms. Adsorption isotherms are important in optimizing the use of adsorbents and to explore the suitable model that can be used for design purposes¹⁸.

Langmuir adsorption isotherm: Langmuir's model assumes that the binding sites at the adsorbent surface are homogeneously distributed and have similar affinity for the adsorbate molecules forming their monolayer and there is no interaction between molecules adsorbed at

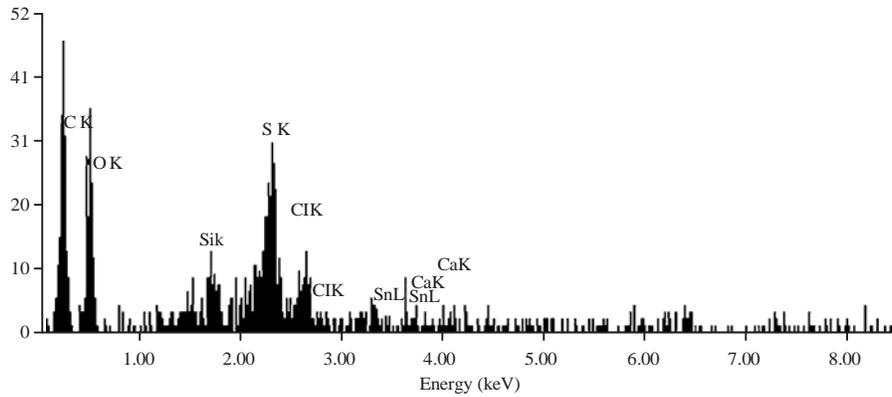


Fig. 4: Energy Dispersive X-ray (EDX) spectra of as-synthesized barley husk derived activated carbon

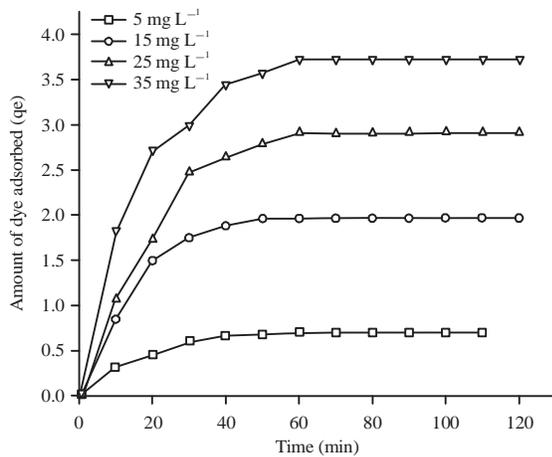


Fig. 5: Plots of q_e (amount of phenol red adsorbed per gram adsorbent) (mg g^{-1}) as a function of contact time (min) using varying initial concentrations of phenol red. (adsorbent dose: 5 g L^{-1} and $\text{pH} = 2.0$)

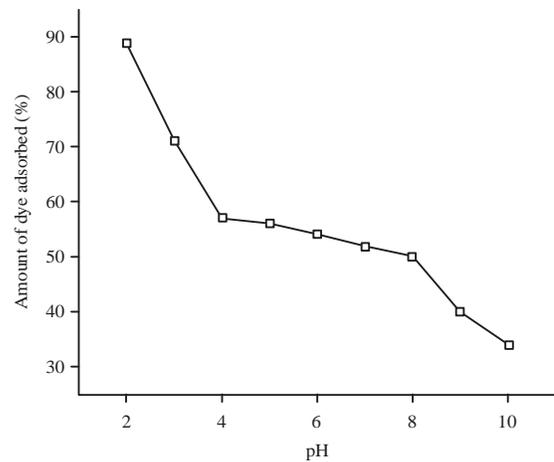


Fig. 6: Plot of Percent adsorption of phenol red dye at barley husk derived activated carbon as a function of pH (adsorbent dose = 2.5 g L^{-1} , dye initial concentration = 50 mg L^{-1})

neighbouring sites¹⁹. Langmuir adsorption isotherm can be represented by the relation Eq. 3:

$$1/q_e = (1/K_L \cdot Q_m) \cdot (1/C_e) + 1/Q_m \quad (3)$$

Where, q_e is the amount of adsorbate (mg) adsorbed per gram of adsorbent and is given by Eq. 4:

$$q_e = [(C_0 - C_e)/w]V \quad (4)$$

The C_0 and C_e are initial and equilibrium concentrations (mg L^{-1}) of adsorbate (dye), respectively, 'V' is the volume of dye solution (liter) and 'w' is mass of the adsorbent. In equation (3), K_L and Q_m are the Langmuir's adsorption equilibrium constant (L mg^{-1}) and maximum amount of adsorbate (mg) adsorbed per gram of adsorbent,

respectively. The parameter Q_m and Langmuir equilibrium constant K_L obtainable from the intercept and the slope of linear plot between $1/q_e$ and $1/C_e$ (Fig. 8), are recorded in Table 3. At the ambient temperature, maximum 6.756 mg of phenol red dye could be adsorbed per gram of the studied adsorbent.

Freundlich adsorption isotherm: Freundlich model for adsorption assumes heterogeneous nature of adsorbent surface having active sites with varying affinities^{20,21}. Freundlich adsorption isotherm can be written as Eq. 5:

$$\text{Log}q_e = \text{Log}K_f + (1/n)\text{Log}C_e \quad (5)$$

Where, parameters K_f and 'n' are measures of adsorption capacity and adsorption intensity, respectively. These

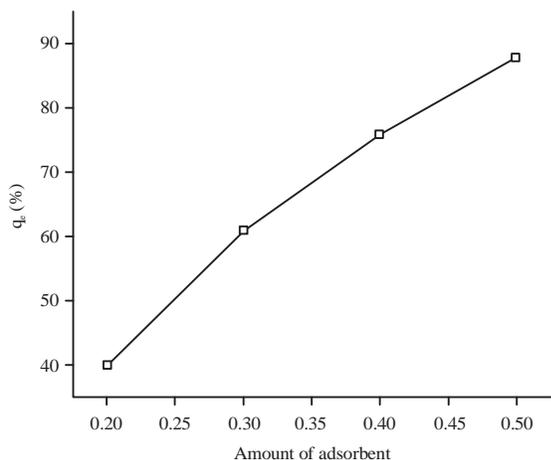


Fig. 7: Plot of percentage of phenol red adsorbed as a function of adsorbent (barley husk derived activated carbon) load (g L^{-1}) (initial concentration of phenol red = 25 mg L^{-1} , $\text{pH} = 2$)

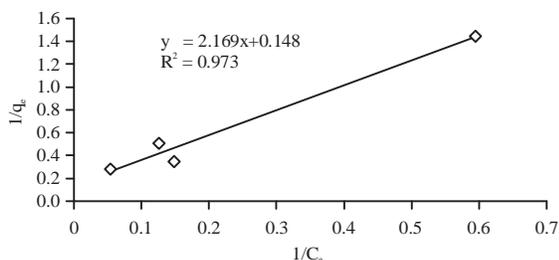


Fig. 8: Plot of $1/q_e$ versus $1/C_e$ for adsorption of phenol red dye on barley husk derived activated carbon at room temperature

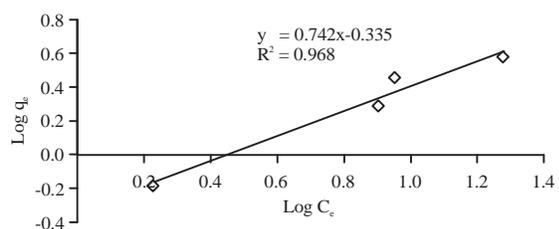


Fig. 9: Plot of $\text{Log } q_e$ versus $\text{Log } C_e$ for adsorption of phenol red on barley husk derived activated carbon at room temperature (q_e and C_e are defined in eq. 2 and 3, respectively)

parameters, obtainable from the slope and intercept, respectively, of the plot between $\text{Log } q_e$ and $\text{Log } C_e$ (Fig. 9), are recorded in Table 3. The magnitude of $1/n$ also indicates the feasibility of adsorption process, i.e., when, $0 < (1/n) < 1$, it is favorable and if $1/n > 1$, it is unfavorable²². For the studied adsorbate-adsorbent system the value of $1/n = 0.742$ being

Table 3: Langmuir and Freundlich parameters for adsorption of phenol red on barley husk derived activated carbon at ambient temperature

Langmuir parameters	
Q_m	6.756
K_a	0.068
R^2	0.973
Freundlich parameters	
K_F	0.4623
n	1.347
R^2	0.968

less than unity suggests the feasibility of adsorption of phenol red on barley husk derived activated carbon. The applicability of the linear form of Langmuir and Freundlich models to the studied adsorbate-adsorbent system is confirmed by their high correlation coefficients, 0.973 and 0.968, respectively.

Kinetic of adsorption

Pseudo-first order kinetic study: Lagergren pseudo-first-order kinetics of adsorption is represented by Eq. 6:

$$\text{Log}(q_e - q_t) = \log q_e - k_1 t / 2.303 \quad (6)$$

Where q_e and q_t are amounts (in mg) of adsorbate adsorbed per g of the adsorbent at the equilibrium and at time, respectively, k_1 (min^{-1}) is pseudo-first-order adsorption rate constant. Plots of $\text{Log}(q_e - q_t)$ versus t (min), using varying phenol red initial concentration, on barley husk derived activated carbon at room temperature are presented in Fig. 10. The adsorption rate constant (k_1) obtained from the slope of linear plot of $\text{Log}(q_e - q_t)$ versus time are recorded in Table 4. Pseudo first order rate constant decreases with the increase in the dye initial concentration. It may be because on raising the adsorbate (dye) initial concentration, the availability of active sites on the adsorbent surface/dye molecule, decreases.

Pseudo second-order kinetic study: The pseudo second-order adsorption kinetic rate equation in its integrated form is written as:

$$(t/q_t) = [1/(k_2 \cdot q_e^2)] + (1/q_e) \cdot t \quad (7)$$

The values of q_e and k_2 obtainable from the slope and the intercept of the linear plot between (t/q_t) and time (Fig. 11), are recorded in Table 4.

It can be seen that, though, the correlation coefficient (R^2) values for pseudo-first order plots (Fig. 10) are higher compared to those of pseudo-second order model plots (Fig. 11), yet the calculated values of q_e from the former model

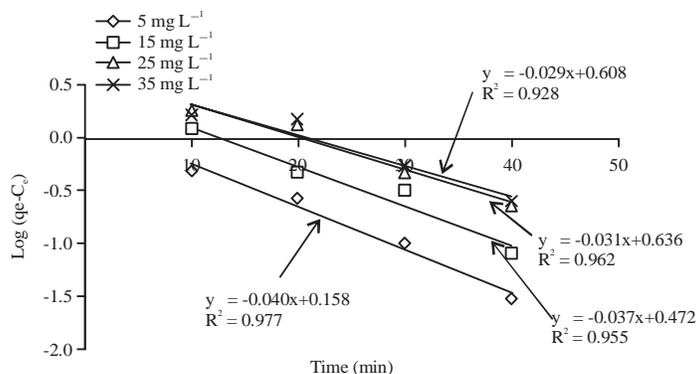


Fig. 10: Plot of $\text{Log}(q_e - q_t)$ versus time (min) using varying phenol red initial concentration on barley husk derived activated carbon at room temperature (adsorbent dosage = 5 g L^{-1} , $\text{pH} = 2$)

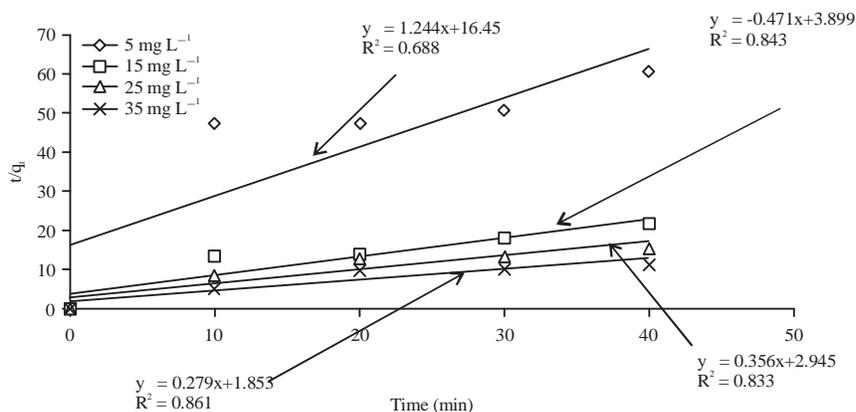


Fig. 11: Plot of t/q_t versus time (min) using varying phenol red initial concentration on barley husk derived activated carbon at room temperature (adsorbent dosage = 5 g L^{-1} , $\text{pH} = 2$)

Table 4: Parameters of different kinetic models for the adsorption of phenol-red on barley husk derived activated carbon using different initial concentrations (C_0)

C_0 (mg L^{-1})	Pseudo-first-order kinetic model				Pseudo-second-order model			Intra-particle diffusion model	
	q_e (mg g^{-1} (exptl.))	q_e (mg g^{-1} (calc.))	k_1 (min^{-1})	R^2	q_e (mg g^{-1} (calc.))	k_2 ($\text{g mg}^{-1} \text{ min}$)	R^2	K_d ($\text{mg g}^{-1} \text{ min}$)	R^2
5	0.690	1.438	0.092	0.977	0.801	0.094	0.688	0.153	0.945
15	1.968	2.964	0.085	0.955	2.123	0.057	0.843	0.353	0.951
25	2.876	4.325	0.071	0.962	2.808	0.043	0.787	0.528	0.962
35	3.496	4.055	0.067	0.928	3.573	0.041	0.891	0.483	0.905

dramatically differ more from the corresponding experimental q_e . Therefore, pseudo-second order model can be considered to fit better for the studied adsorbate-adsorbent system.

Intra-particle diffusion model: The intra-particle diffusion model, due to Weber and Morris²³, assumes that adsorption kinetics on a porous material such as activated carbon involves 2 processes viz. (a) External mass transfer from the solution to the liquid-solid interface and (b) the diffusion of the adsorbed species inside the porous particle. This model can be represented by Eq. 8:

$$q_t = k_d t^{1/2} + C \quad (8)$$

Where, q_t is the amount of adsorbate adsorbed per unit mass of adsorbent (mg g^{-1}) at time t , k_d is the intra-particle diffusion rate constant ($\text{mg g}^{-1} \text{ min}^{1/2}$), C is related to the adsorbate mass transfer across the boundary layer. Value of k_d obtained from the slope of the linear plot between q_t and $t^{1/2}$ (Fig. 12), is given in Table 4.

It is observed that (a) Values of k_d increase with the increase in the adsorbate (dye) initial concentration and (b) The linear plots of q_t and $t^{1/2}$ do not pass through the

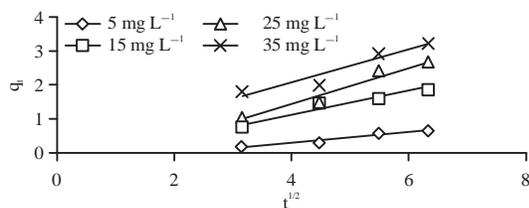


Fig. 12: Plot of q_t versus $t^{1/2}$ for adsorption of phenol red on barley husk derived activated using different initial concentrations of adsorbate (adsorbent dose, 5 g L^{-1} , $\text{pH} = 2$ at room temperature)

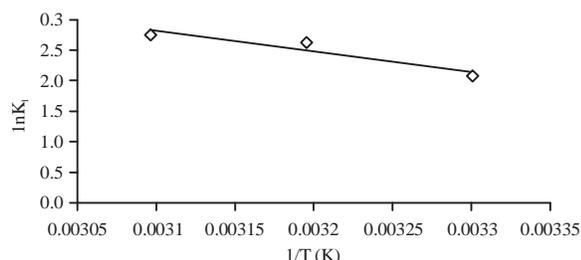


Fig. 13: Van't Hoff plot ($\ln K_L$ versus $1/T$) for adsorption of phenol red dye on barley husk derived activated carbon (dye initial concentration = 25 mg L^{-1} , Adsorbent load: 5 g L^{-1} and $\text{pH} = 2$)

Table 5: Thermodynamic parameters (ΔG^0_{ads} , ΔH^0_{ads} and ΔS^0_{ads}) for adsorption of phenol red on barley husk derived activated carbon

Temperature (K)	ΔH^0_{ads} (kJ mol^{-1})	ΔS^0_{ads} ($\text{J mol}^{-1} \text{K}^{-1}$)	$-\Delta G^0_{\text{ads}}$ (kJ mol^{-1})
303.15	28.4	111.6	5.4
313.15	28.4	111.6	6.5
323.15	28.4	111.6	7.7

origin. This indicates that the concentration gradient between the bulk and the adsorbent surface is the dominating force during adsorption of phenol red on the barley husk derived activated carbon and the intra-particle diffusion is not the sole adsorption rate-limiting step.

Thermodynamics studies: In order to determine thermodynamic parameters of adsorption, experiments were performed at three different temperatures viz., 303.15, 313.15 and 323.15 K ($\pm 0.10\text{K}$) and in each case Langmuir's equilibrium constant (K_L) was calculated. Van't Hoff plot ($\ln K_L$ versus $1/T$) for phenol red dye adsorption on barley husk derived activated carbon (dye initial concentration = 25 mg L^{-1} , Adsorbent load: 5 g L^{-1} and $\text{pH} = 2$) are presented in Fig. 13.

Standard Enthalpy (ΔH^0_{ads}) and entropy (ΔS^0_{ads}) of adsorption is related to the Langmuir adsorption constant (K_L) in the Van't Hoff's Eq. 9:

$$\ln K_L = -(\Delta H^0_{\text{ads}})/(RT) + (\Delta S^0_{\text{ads}})/R \quad (9)$$

Were obtained from the slope and intercept, respectively, of linear plot between $\ln K_L$ and $1/T$ (Fig. 13). The standard free energy of adsorption, ΔG^0_{ads} , was calculated from the Gibbs Helmholtz's Eq. 10:

$$\Delta G^0_{\text{ads}} = \Delta H^0_{\text{ads}} - \Delta S^0_{\text{ads}} \quad (10)$$

These thermodynamic parameters thus obtained are presented in Table 5.

The observed negative standard free energy of adsorption (ΔG^0_{ads}) indicates that the adsorption of phenol red at the barley husk derived activated carbon, is favourable and of spontaneous nature. As ΔG^0_{ads} decreases (becomes more negative) on raising temperature, it suggests the adsorption becomes more feasible at higher temperature. Though the observed endothermic enthalpy of adsorption opposes the adsorption process, yet it becomes feasible due to the predominate larger entropy gain.

CONCLUSION

Adsorption of phenol-red dye on barley husk derived activated carbon follows pseudo second order kinetic model and the process of adsorption, though endothermic, is predominately favored by entropy gain. Whereas, the dye removal efficiency of the adsorbent decreases with the increasing initial concentration of the former, an optimum adsorbent level is required to achieve maximum removal of the dye of specified initial concentration. The results show that the barley husk derived activated carbon can be used as an efficient and cost-effective adsorbent for the treatment of water contaminated with phenol red.

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