

ADSORPTION OF PHENOL FROM AQUEOUS MEDIA BY AN AGRO-WASTE (*HEMIDESMUS INDICUS*) BASED ACTIVATED CARBON

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Abstract. The adsorption of phenol by an agro-waste based activated carbon prepared from the root residue of *Hemidesmus Indicus* (HIC) was investigated to assess its possible use as adsorbent. The effect of various factors, namely, pH, initial adsorbate concentration, adsorbent dosage and contact time were studied to identify adsorption capacity of HIC. The results were compared to that obtained from adsorption of phenol by commercial activated carbon (CAC). Adsorption data were modeled with the Langmuir and Freundlich isotherms. The kinetic models were also applied for the pseudo-first-order, pseudo-second-order, intra-particle diffusion and pore diffusion coefficients. Although HIC and CAC showed much similar isotherm models and kinetics, yet HIC was found to show much higher boundary layer effect and pore-diffusion coefficients, in relation to CAC.

Keywords: *Phenol, agro-waste, adsorption, Isotherms, adsorption kinetics*

Introduction

Since 1860, phenol has been in production, with its basic use as an antiseptic. During late 19th century and thereafter the use of phenol has been further extended to the synthesis of dyes, aspirin, plastics, pharmaceuticals, petrochemical and pesticide chemical industries. In fact, by 2001, the global phenol production has reached an impressive 7.8 million tons [1].

Normally, discharges from the aforesaid phenol-yielding industries find their way to water bodies and subsequently affect the water quality adversely. As per the studies by various authors, phenols and its derivatives have been reported to cause undesirable and deleterious effect in water, even at a concentration as low as 0.1 ppm [2]. In fact, the adverse effects of phenol have been observed on the central nervous system, cardiovascular system as well as urino-genital systems of human being, often expressed by the multiple symptoms: convulsions, coma, cardiac disorders, respiratory failure and collapse [3]. As per literature, the various analytic methods attempted for dephenolation of wastewater include steam stripping, solvent extraction, oxidation (O₃, H₂O₂, and ClO₂), ion exchange, biodegradation and adsorption methods [4, 5, 6]. Out of all these, adsorption methods are the most-widely used techniques and activated carbon has been the predominant adsorbent all over the world [7, 8]. However, due to its high cost and low regeneration capacity, since the last three decades, extensive researches have been directed towards investigating the adsorption characteristics and potentials of cheaper materials and solid wastes, such as fly ash, peat, soil, rice husk, sawdust, bagasse and so forth [9, 10, 11, 12].

In the present study, the extracted residue of a root of an Indian plant, namely *Hemidesmus indicus* (also called Sveta sariva, Ananda mul or Nannari and is popularly used as energy stimulant soft drink), is studied for its adsorption capacity. In fact,

Hemidesmus indicus has been known to possess various favourable medical characteristics, such as promoting good circulation, clearing toxins, balancing the glandular system, regulating hormone secretion, providing calories, protecting against UV-radiation, stimulating metabolism and curing several stomach and kidney disorders. It has also been indigenously used for water purification (mainly turbidity removal) in certain traditional communities in India. However, the residue of the root of the plant has been considered wastes, with apparently no specific utilization (probably, except combustion and composting to a limited degree). The present paper is aimed at evaluation of the effectiveness of *Hemidesmus indicus* root residue and determination of its optimization condition for phenol-sorption.

Experimental Methods

Preparation of activated carbon

Roots of *Hemidesmus indicus*, a locally available slender twining herb, was collected and after extraction of its juice by traditional method (of grinding, boiling with water and filtering), was cleaned and washed thoroughly to remove water-soluble substances (so as not to affect the aqueous characteristics of phenolated wastewater). The material was acidulated using 1:1 wt ratio of concentrated H₂SO₄ and subsequently allowed to soak for 24 hours at room temperature. Thereafter, the sample materials were placed in an oven and heated to 200 °C for 24 hours, followed by cooling, washing (with distilled water) and soaking with 1% NaHCO₃ solution (to remove any excess acid). The washing with distilled water was continued till the uniform characteristics of both filtering and filtered water (with respect to colour, pH and turbidity). The thoroughly washed sample was dried and subsequently subjected to pyrolysis at 850–900 °C for 30 minutes. The samples, thus obtained, were ground in a ball mill and the particles having an average diameter of 0.5 mm (using the sieves of 20–50 mesh, ASTM), were collected and stored for further studies.

Characterization of Activated Samples

Activated carbon, obtained as above, was characterized by adopting the standard procedures [13, 14, 15]. The moisture content of the carbon was determined by heating a known weight of the sample in an air oven maintained at 105 ±5 °C for about 4 h. The residue was ignited in a muffle furnace at 1000°C for about 3 h to determine the ash content. Surface acidity of the sample was studied using boiled water and decolourising power, using methylene blue solution. The adsorption characteristics was studied in terms of phenol number (the amount of carbon required for 90% removal of phenol, which indicates the ability to remove taste and odour of the activated carbon) and iodine number (the ability of the carbon to adsorb low molecular weight substances, defined as the milligrams of iodine (I₂) that are adsorbed per gram of carbon when the equilibrium concentration of the bulk saturation (C_{eq}) is 0.02 N). The surface area of the activated carbons was carried by BET (Brunauer Emmett Teller) Nitrogen adsorption method. The characteristics of the activated carbons are presented in Table 1.

Table 1. Characteristics of HIC and CAC

Property	HIC	CAC
Bulk Density (g.ml ⁻¹)	0.48	0.65
Moisture content (%)	10.35	12.57
Ash content (%)	2.08	2.78
Fixed Carbon (%)	97.92	97.22
Solubility in water (%)	0.80	1.55
Solubility in 0.25M HCl acid (%)	1.2	3.0
pH of 5% slurry	6.3	8.2
Decolourising power (mg.g ⁻¹)	82	74
Phenol number (mg)	5.3	5.2
Iodine number (mg.g ⁻¹)	204	192
Surface area (m ² .g ⁻¹)	627	596

Batch experiments

A 100 ml each of synthetic phenol solution (30 ppm) adjusted to different pH values by 0.1 N NaOH or 0.1 N H₂SO₄, were placed in 250 ml leak-proof reaction bottles and a known amount of *Hemidesmus indicus* carbon (HIC) was added to each bottle. The solution was equilibrated for 24 hours at room temperature, followed by filtration of the adsorbent and subsequent analysis of filtrate for phenol concentrations were determined by spectrophotometric analysis of the colour resulting from the reaction of phenol with 4- aminoantipyrine at a wavelength of 500 nm [13]. Kinetic experiments were conducted using a known weight of carbon dosage for a phenol concentration in the range of 5–40 ppm. Periodically, after regular intervals of time, samples were analysed for phenol concentration. The rate constants were calculated by using the various models (viz. pseudo-first-order, pseudo-second-order, intra-particle diffusion and pore diffusion coefficients).

Results and Discussion

The adsorbent prepared from HIC was studied for adsorption and the results thus obtained were compared with that of CAC and the removal efficiency of phenol from aqueous solution was estimated, under different experimental conditions.

Effect of carbon dosage

Figure 1 shows the amount of phenol removed as a function of carbon dosage from the solution at neutral pH. Carbon dosage was varied from 5 to 500 mg and equilibrated for 24 hours. As evident from the figure, to remove the entire amount of phenol (with initial concentration of 30 ppm), the minimum adsorbent dosage required was found to be 35mg of CAC or 30mg of HIC. Thus, HIC seems to be better dephenolating agent than CAC.

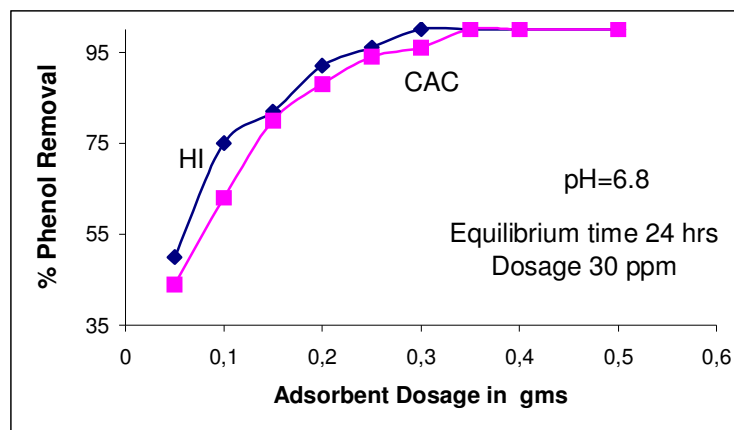


Figure 1. Effect of Adsorbent Dosage

Effect of pH on phenol adsorption

Since phenol sorption has been reported to be affected by the pH of the adsorbate [9], the adsorption of phenol by HIC and CAC were studied at various pH values of the phenol solution (100ml, 30-ppm). The amount of phenol adsorbed shows a declining trend with higher as well as with lower pH, with maximum removal of phenol (up to almost 100 % by both the adsorbents) at neutral pH (Figure 2). This reduction of phenol-sorption may be because of the suppression by hydrogen ions (at lower pH), and hydroxyl ions (at higher pH) in addition to formation of various phenolic compounds at both acidic and alkaline conditions [16, 17].

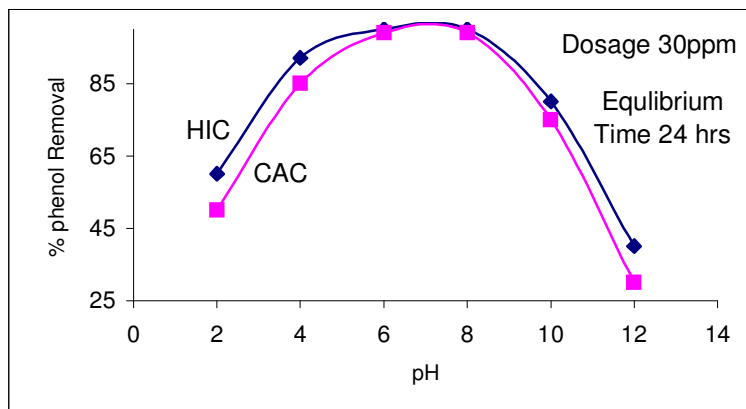


Figure 2. Effect of pH on Phenol Adsorption

Effect of contact time

Figure 3 shows the effect of contact time on the removal of phenol by HIC and CAC. For different duration of time (ranging from 0.5 to 24 hours), well-shaken phenol solution (100 ml, 30 ppm) was studied for equilibrium. The study revealed 5 hour and 6 hours as the equilibrium time for HIC and CAC, respectively.

Adsorption Isotherms

In order to determine the adsorption potential of both the adsorbents (viz. HIC and CAC) for the removal of phenol, study of adsorption isotherm was carried out and

tested against the Freundlich and Langmuir isotherm models, using standard procedures used by various authors [9, 18, 19].

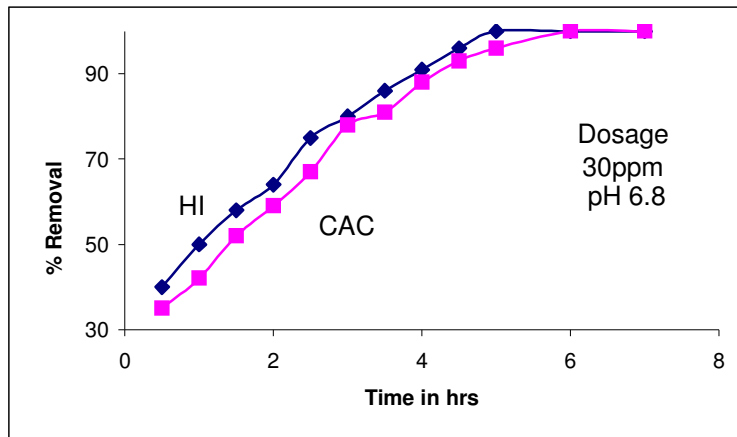


Figure 3. Effect of Time on phenol Adsorption

Langmuir Isotherm:

The Langmuir equation is given as:

$$\frac{C_e}{q} = \left(\frac{1}{Q_0 b} \right) + \left(\frac{C_e}{Q_0} \right) \quad \text{(Eq.1)}$$

Where, $q = x/m$, represents the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g) and ' C_e ', ' Q_0 ' and ' b ' refer to the equilibrium concentration (ppm), monolayer adsorption capacity (mg/g) and surface energy (g/l), respectively. A plot of C_e/q versus C_e as shown in Figure 4, where ' $1/(b Q_0)$ ' and ' $1/Q_0$ ' correspondingly represent the intercept and slope (comparing with the normal equation for straight line as ' $y = mx + c$ ').

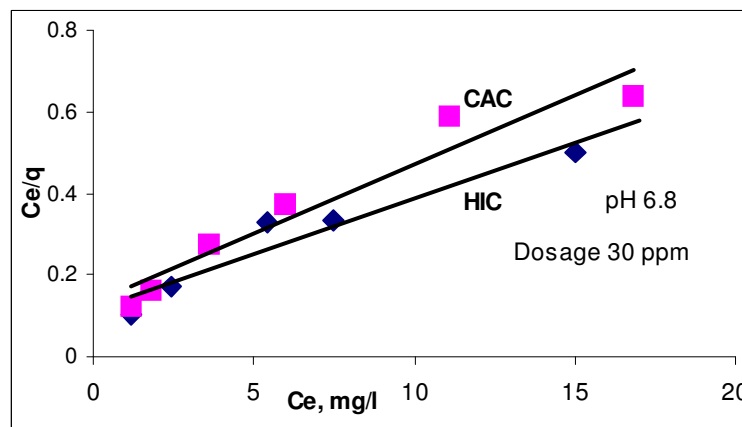


Figure 4. Langmuir Adsorption Isotherm for Phenol

The Langmuir isotherm can also be expressed by a separation factor (R_L), which is given by the equation [20].

$$R_L = 1 / (1 + b \cdot C_i) \quad \text{(Eq.2)}$$

Where, ‘ C_i ’ is the initial concentration of phenol in ppm and ‘ b ’ is the Langmuir constant in g/l. The separation factor ‘ R_L ’ indicates the nature of the adsorption process as given in Table 2.

Table 2. The process nature of separation factor.

S.No.	R_L Value	Type of process
1	$R_L > 1$	Unfavourable
2	$R_L = 1$	Linear
3	$0 < R_L < 1$	Favourable
4	$R_L = 0$	Irreversible

Freundlich Isotherm: The Freundlich equation is expressed as

$$\frac{x}{m} = q_e = kC_e^{1/n} \quad (\text{Eq.3})$$

Where, ‘ k ’ and ‘ n ’ are the measures of adsorption capacity and intensity of adsorption. ‘ q_e ’ is the amount of phenol adsorbed per unit mass of adsorbent and ‘ C_e ’ is the equilibrium concentration in ppm. The logarithmic form of **Freundlich** equation can be expressed by

$$\log(x/m) = \log q_e = \log k + 1/n \log C_e \quad (\text{Eq.4})$$

From the straight line (obtained by plotting ‘ $\log(x/m)$ ’ against ‘ $\log C_e$ ’ as shown in Figure 5 the corresponding slope and intercept can be determined from ‘ $1/n$ ’ and ‘ $\log k$ ’ values. Table 2 indicates the values of Langmuir and Freundlich constants.

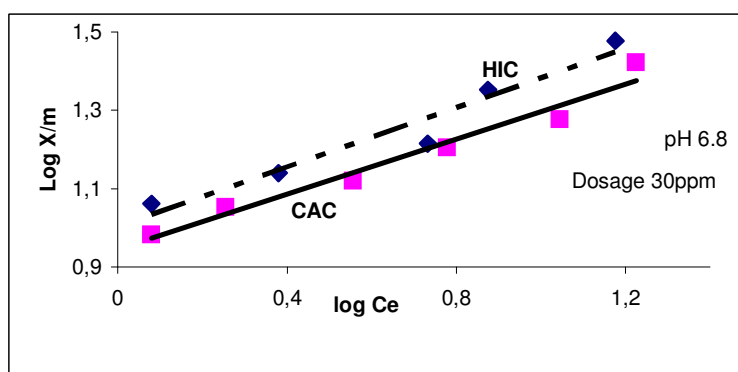


Figure 5. The Freundlich Adsorption Isotherm for Phenol

The results reveal that the adsorption of phenol on HIC and CAC obeys both Freundlich and Langmuir adsorption isotherms, as indicated by high R^2 Values (>92%). Greater the value of k (the Freundlich constant), higher the phenol uptake from aqueous solution [9,18]. As shown in Figure 5, higher k -value of HIC (1.04 mg/g) indicates greater affinity for phenol compared to that of CAC (0.57 mg/g), which shows better effectiveness of the phenol-HIC system than phenol-CAC system. The adsorption

intensity, ‘n’ is found to be 2.65 and 2.84 for HIC and CAC, respectively. It is observed that, in both these systems, the n-values satisfy the condition(s) of heterogeneity, i.e., $1 < n < 10$ as well as $0 < 1/n < 1$ [16]. The higher magnitude of ‘ Q_0 ’ indicates that the amount of phenol per unit weight of sorbent (to form a complete monolayer on the surface) seems to be significantly higher for phenol–HIC and phenol–CAC systems. A relatively lower ‘b’ – value (< 0.3) implies low surface energy in both the systems, thus indicating a probable stronger bonding between phenol and sorbents [18]. In fact, fairly low to moderate ‘b’ - values have been reported in many of the sorbent-phenol systems, involving palm-seed-coat-activated-carbon, bentonite, and rice husk [9, 11, and 19].

Adsorption kinetics

Kinetics of adsorption, a standard analysis in defining adsorption efficiency, describes the solute uptake rate, which in turn governs the residence time of adsorption reaction. The study the adsorption kinetics of the two phenol-sorbent systems (viz. -HIC and -CAC) were carried out using batch experiments. The adsorbents (0.3g) were separately exposed to the synthetic phenol solution (100ml, 30ppm) and the amount of phenol adsorbed was estimated for a time period of 30 min to 360 min. (or, 6 hours, which refers to the equilibrium time, Figure 1.)

Table 3. Details of Isotherm Constants

Name of Adsorbent	Langmuir Constants			Freundlich Constants			Separation Factor
	Q_0	b	R^2	k	n	R^2	R_L
Hemidesmus Indicus Carbon (HIC)	370.37	0.23	0.92	1.04	2.65	0.94	0.12
Commercial Activated Carbon (CAC)	294.11	0.26	0.93	0.57	2.84	0.96	0.11

Pseudo- First – Order Kinetics: A widely-used Lagergren model was employed to study the pseudo first order kinetics [21,22, 23].

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (\text{Eq.5})$$

where ‘ q_e ’ and ‘ q_t ’ refers to the amounts of phenol (ppm) adsorbed on the activated carbon at equilibrium time and time t (min) and k_1 as rate constant (min^{-1}), respectively .

Integrating the above equation between the limits from $t = 0$ to $t = t$ and from $q_t = 0$ to q_t

= q_t

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (\text{Eq.6})$$

Thus, the rate constants (k_1) were obtained from slope of the plots of $\log(q_e - q_t)$ Vs t ($R^2 > 0.9$, Eq.6) and presented in Table 4. HIC was found to be better rate-limited (0.0104) by pseudo-first order model, than CAC (0.0092).

Table 4. Rate constants for the phenol removal by HIC and CAC

Type of Carbon	Pseudo-First-Order model			Pseudo-Second-Order model		
	q_e	R^2	$k_1(\text{min}^{-1})$	$k_2(\text{g/mg.min})$	$h(\text{mg/g.min})$	R^2
Hemidesmus Indicus Carbon(HIC)	16.10	0.93	0.0104	0.0003	0.4166	0.97
Commercial Activated Carbon(CAC)	11.01	0.94	0.0092	0.0002	0.3246	0.94

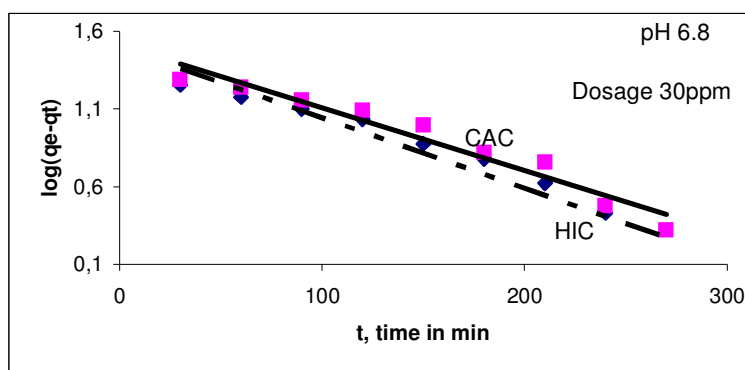


Figure 6. Pseudo-First-Order Kinetics for HIC and CAC

Second order Kinetics:

The sorption data were also studied by second order kinetics [24,21]

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (\text{Eq.7})$$

Where, ' k_2 ' refers to the rate constant of second order adsorption (g/mg.min).
 On Integration,

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (\text{Eq.8})$$

Expressing in the form of a standard straight-line equation ($y = mx + c$);

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e}, \text{ 'h' being } k_2 q_e^2.$$

The plot of t/q_t vs t (Figure 7) gives a linear relationship ($R^2 > 0.9$), from which the constants k_2 , q_e and h were determined (Table 3). As in the case of the pseudo-first order model, the present analysis also indicate HIC as marginally better rate-limited (0.0003) by pseudo-second order model, than CAC (0.0002).

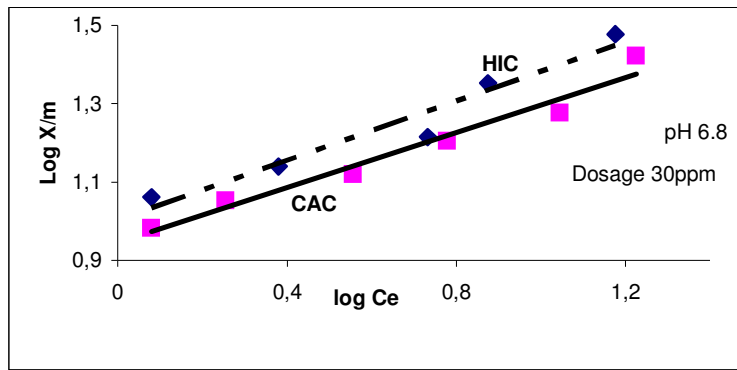


Figure 7. Pseudo-Second-Order Kinetics for HIC and CAC

Intra-particle diffusion

The structure of the adsorbent and its interaction with the diffusing adsorbate (Intra-particle diffusion) influence the rate of transport, where the solute movement is a function of concentration gradient [25] and the rate constant (k_i) can be determined by Intra-particle diffusion model [26].

$$q_t = k_i t^{(0.5)} + C \quad (\text{Eq.9})$$

Where, q_t refers to the amount of phenol adsorbed in mg/g at time, t ; intercept C , indicating the boundary layer effect [27] and k_i , the Intra-particle diffusion rate constant ($\text{mg/g} \cdot \text{min}^{1/2}$). A plot between the amount of phenol adsorbed and square root of time gives the rate constant (Figure 8, Table 4). The results indicate the intra-particle diffusion as a rate-determining step for HIC and CAC. The boundary layer effect of HIC (5.19) was found to be more than twice to that of CAC (2.16).

The pore diffusion coefficient, D , for the removal of phenol by HIC and CAC were calculated (assuming a spherical-geometry of the adsorbents; 20-50 ASTM; average mesh size 0.5mm):

$$t_{1/2} = 0.03 r_o^2 / D \quad (\text{Eq.10})$$

Where, $t_{1/2}$ refers to the time for half adsorption (sec); r_o , the diameter of the particle (cm); D , the pore diffusion constant (cm^2/s).

Pore diffusion in the adsorbents (Table 5) is assessed to be less significant due to relatively higher values of the coefficients, in contrast to the rate-limiting range (10^{-11} to $10^{-13} \text{ cm}^2/\text{s}$) [28]. However, the pore diffusion coefficient for HIC ($2.08 \times 10^{-8} \text{ cm}^2/\text{s}$) shows almost 1.5 times that for CAC ($1.39 \times 10^{-8} \text{ cm}^2/\text{s}$).

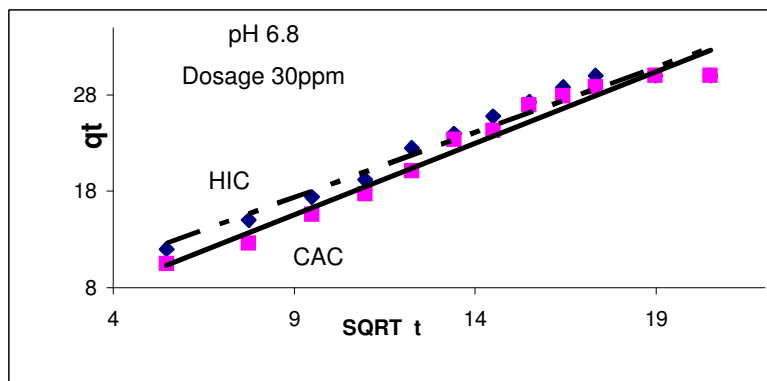


Figure 8. Intra-particle Diffusion plot of phenol on HIC and CAC

Table 5. Intra particle and pore diffusion constants

Type of Carbon	Intra-particle Diffusion model			Pore Diffusion Coefficient
	C	R ²	k _i (mg/g. min ^{1/2})	D (cm ² /s)
Hemidesmus Indicus Carbon (HIC)	5.19	0.95	1.35	2.08 x 10 ⁻⁸
Commercial Activated Carbon (CAC)	2.16	0.96	1.48	1.39 x 10 ⁻⁸

Conclusions:

The present studies indicate *Hemidesmus indicus* Carbon (HIC) as a better adsorbent than the commercial activated carbon (CAC), as indicated by its higher adsorption at lower adsorption dosage (and period of equilibrium). Adsorption characteristics of HIC and that of CAC showed notable similarity as reflected by (i) their obedience to both Langmuir and Frueundlich isotherm models, (ii) similar rate-characteristics (in both first- and second- order kinetics, with correlation coefficients greater than 0.9), (iii) rate-determining character of intra-particular diffusion, and (iv) less significant role by pore-diffusion process. However, HIC was found to be marginally better rate-limited (than CAC) by both pseudo-first and pseudo-second order models. Besides, ‘the boundary layer effect of’ and ‘the pore diffusion coefficient for’ HIC was found to be almost double to and 1.5 times of CAC, respectively.

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