



Adsorptive removal of 2-chlorophenol by low-cost coir pith carbon

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Abstract

Adsorption of 2-chlorophenol (2-CP) by coir pith carbon was carried out by varying the parameters such as agitation time, 2-CP concentration, adsorbent dose, pH and temperature. Adsorption equilibrium reached at 40, 60, 80 and 100 min for 2-CP concentration of 10, 20, 30 and 40 mg/l, respectively. Adsorption followed second-order kinetics. The adsorption equilibrium data obeyed Freundlich isotherm. Acidic pH was favorable for the adsorption of 2-CP. Desorption studies showed that chemisorption plays a major role in the adsorption process.

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1. Introduction

Among the various aquatic pollutants, organic compounds are of increasing concern due to their ecotoxicological effects. Some chlorinated organics are formed in the course of chlorination of water supplies [1]. Among the chlorinated organic pollutants, chlorophenols are of major concern in view of their widespread contamination of soil and potable groundwater supplies and their harmful effects on man and animals [2]. The sources of chlorophenols to the environment are wastewaters from pulp [3], pharmaceutical, petrochemical refinery, herbicide, pesticide, and coal conversion industries [4] and accidental release [5].

It has been reported that 0.01 mg/l of chlorophenols imparts extremely disagreeable taste and odor to water [6]. Residues of chlorophenols have been found in soil, water and air

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samples, food products, human and animal tissues and body fluids [7]. Though they have beneficial biocidal properties, they are toxic at higher concentrations. Toxicity generally increases with the degree of chlorination [8]. It has been reported that the OH group plays an important role in the toxicity of chlorophenols in fish [9]. The permissible limits for phenolic compounds in industrial effluents before discharging into municipal sewers and surface water in India is 1–5 mg/l [10,11].

Commercially available activated carbons like F300 granular activated carbon (Calgon Corp., Pittsburgh, PA) [12–14] are used for the adsorption of chlorinated phenols. But these are expensive and the regeneration costs are high. Hence, low-cost activated carbons based on agricultural solid wastes are investigated for a long time. As organic agricultural wastes are available in large quantities, there is no shortage of starting materials to make activated carbons. Agricultural byproducts and waste materials used for the production of activated carbons include waste apple pulp [15], straw, coconut shell, wood, tyre [16], apricot stone shell [17], olive stones [18], bituminous coal [19], peanut hull [20], etc. In developing countries like India, industries cannot afford to use conventional wastewater treatment chemicals like alum, ferric chloride, polymer flocculants and coal-based activated carbon because they are not cost-effective. Agricultural wastes like coir pith are discarded in the agricultural sector in India. Its accumulation around coir-processing centers is creating a menace. Annual production is around 7.5 million tonnes [21]. Coir pith, which constitutes as much as 70% of the coconut husk, is a light fluffy material that is generated in the separation process of the fiber from the coconut husk. Carbonized coir pith has been investigated for the removal of heavy metals [22], dyes [23,24] and toxic anions and pesticides [25] in our laboratory before. The objective of this study was to investigate the feasibility of using carbonized coir pith for the removal of 2-chlorophenol (2-CP) from wastewaters by adsorption method.

2. Experimental

2.1. Materials

Coir pith was collected from nearby coir industries, dried in sunlight for 5 h and grounded. The dried coir pith powder was sieved to 250–500 μm size. It was subjected to carbonization at 700 °C for 1 h using a muffle furnace under closed conditions. The carbonized material was taken out, sieved to 250–500 μm size again and used for adsorption studies. The characteristics of the carbon are presented in Table 1. Specific surface area and pore size distribution was determined using Quantachrome surface area analyzer. Pore size distribution is shown in Fig. 1. Surface functional groups [26] (Table 2) and other parameters were determined using standard methods [27,28]. For the tests [29] with molasses, the residual product of unknown water content from a sugar factory was used, which is not a standard molasses. A dilution to the ratio 1:200 was made from the molasses with distilled water. Carbon samples of 100, 200, 300, 400 and 500 mg were used and added to 50 ml of the molasses solution. The optical density was measured spectrophotometrically at $\lambda = 315$ nm. Coir pith carbon and Unicarbon UP25 (Unique Carbon and Chemicals, Rajpipla, Gujarat, India) were used as adsorbents. The results are presented in Table 3.

Table 1
Characteristics of coir pith carbon

Physical parameters	
Specific surface area (m ² /g)	167
Micropore volume (cm ³ /g)	0.0913
Total pore volume (cm ³ /g)	0.1217
Micropore area (%)	89.3
Micropore volume (%)	75
Average pore diameter (nm)	2.75
Average micropore diameter (nm)	1.2
pH _{ZPC}	8.0
pH (1% solution)	10.1
Conductivity (1% solution) (mS/cm)	2.3
Bulk density (g/ml)	0.12
Mechanical moisture content (%)	5.88
Ash content (%)	79.87
Specific gravity	1.742
Porosity (%)	93.11
Volatile matter (%)	58.38
Fixed carbon (%)	41.62
Decolorizing power (mg/g)	21.0
Iodine number (mg/g)	101.52
Ion exchange capacity	Nil
Chemical parameters	
Sodium (%)	0.14
Potassium (%)	0.18
Calcium (%)	0.22
Phosphorous (%)	0.01
Iron (%)	0.18

2.2. Methods

2.2.1. Adsorption studies

Adsorption experiments were carried out by agitating 100 mg of adsorbent with 50 ml of 2-CP solution of desired concentration and pH at 200 rpm, 35 °C in a thermostated rotary shaker (ORBITEK, Chennai, India). Concentration of 2-CP was estimated

Table 2
Surface functional groups

Functional group	Amount (meq./g)	Percentage
Carboxylic	2.304	64.87
Lactones	0.672	19.08
Phenolic	0.576	16.22
Total	3.552	100
Basic	2.2	38.25
Total capacity	5.752	

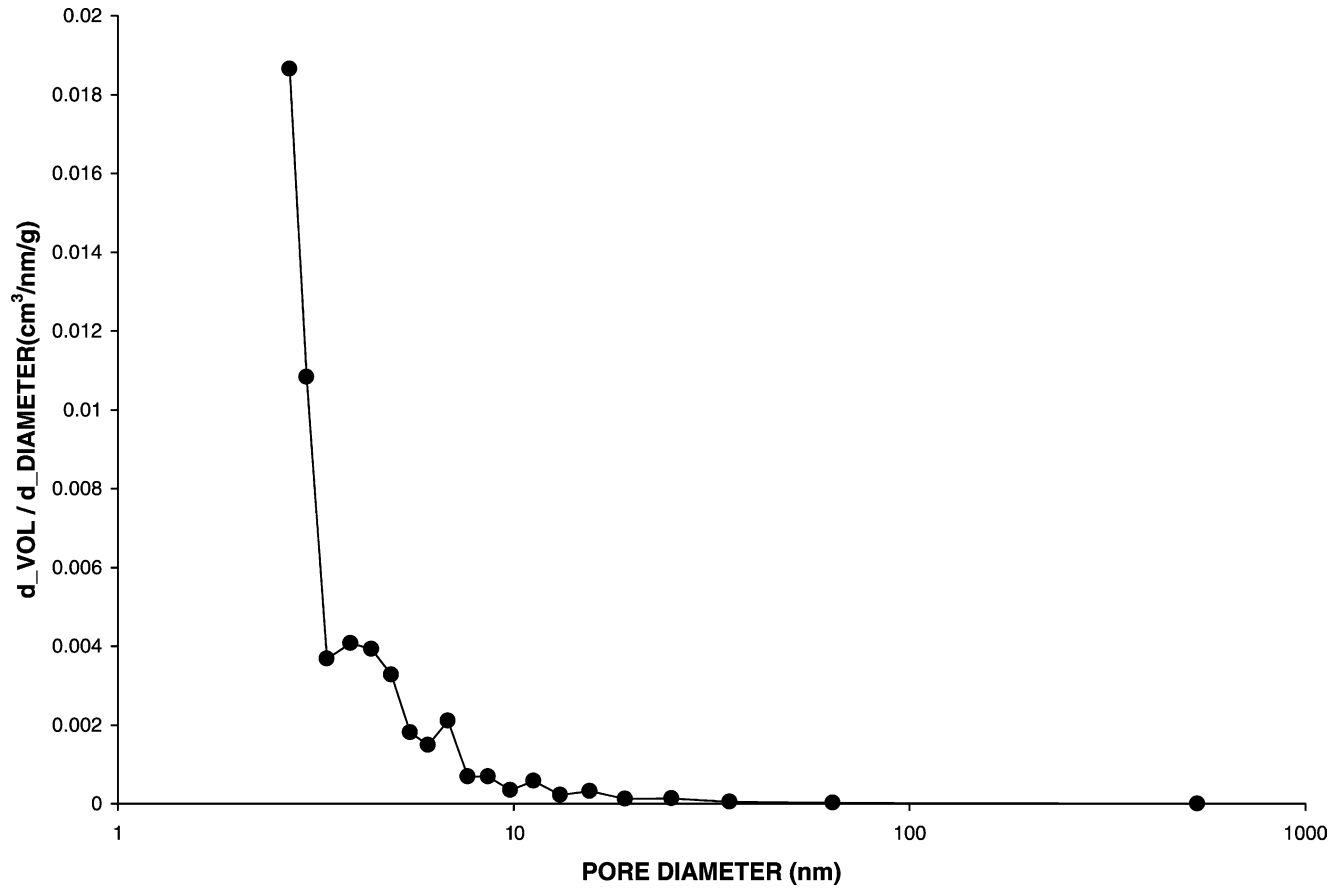


Fig. 1. Pore size distribution of coir pith carbon.

Table 3
The results of the molasses test

Amount of adsorbent (mg/50 ml)	Absorbance at 315 nm	
	Unicarbon UP25	Coir pith carbon
0	4.878	4.878
100	0.879	4.146
200	0.583	3.805
300	0.459	3.678
400	0.388	3.562
500	0.344	3.538

spectrophotometrically by monitoring the absorbance at 273 nm using UV-Vis spectrophotometer (Hitachi, model U-3210, Tokyo). The pH was measured using pH meter (Elico Instruments, model LI-107, Hyderabad, India). The samples were withdrawn from the shaker at predetermined time intervals and the 2-CP solution was separated from the adsorbent by centrifugation at 20,000 rpm for 20 min. The absorbance of supernatant solution was measured. Effect of pH was studied by adjusting the pH of 2-CP solutions using dilute HCl and NaOH solutions. Effect of adsorbent dosage was studied by agitating 50 ml of 10, 20, 30 or 40 mg/l phenol solutions with different adsorbent doses (25–500 mg) at equilibrium time. Langmuir, Freundlich and Dubinin–Radushkevich isotherms were employed to study the equilibrium adsorption. Effect of temperature was studied using 10 mg/l of 2-CP and 50 mg of adsorbent at 35, 40, 50 and 60 °C.

2.2.2. Desorption studies

The adsorbent that was used for the adsorption of 10 mg/l of 2-CP solution was separated from the solution by centrifugation. The 2-CP-loaded adsorbent was filtered using Whatman filter paper and washed gently with water to remove any unadsorbed 2-CP. Several such samples were prepared. Then the spent adsorbent was agitated for 40 min with 50 ml of distilled water, adjusted to different pH values. The desorbed 2-CP was estimated as before.

3. Results and discussion

3.1. Effects of agitation time and concentration of 2-CP on adsorption

Effects of agitation time and 2-CP concentration on removal of 2-CP by coir pith carbon are presented in Fig. 2. The amount of adsorbed (mg/g) increased with increase in agitation time and concentration and remained nearly constant after equilibrium time. The equilibrium time increased with increase in concentration. The equilibrium time for 10, 20, 30 and 40 mg/l of was found to be 40, 60, 80 and 100 min, respectively. It is clear that the removal depends on its concentration. The removal curves are single, smooth and continuous leading to saturation.

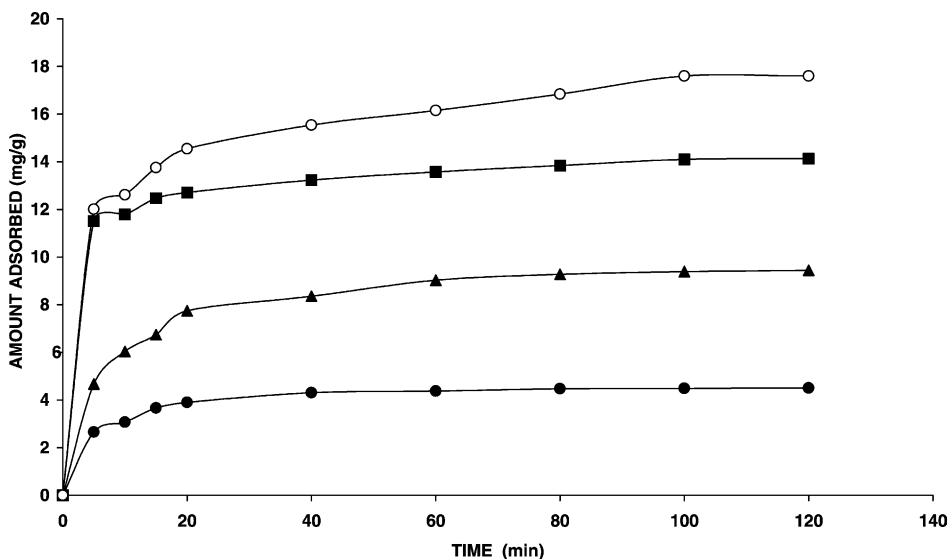


Fig. 2. Effect of agitation time and concentration of 2-CP on removal. Adsorbent dose, 100 mg/50 ml; pH 2.0; 2-CP concentration: (●) 10 mg/l, (▲) 20 mg/l, (■) 30 mg/l, (○) 40 mg/l; temperature, 35 °C.

3.2. Adsorption dynamics

3.2.1. Adsorption rate constant

The rate constant of adsorption is determined from the first-order rate expression given by Lagergren and Svenska [30]:

$$\log(q_e - q) = \log q_e - \frac{k_1 t}{2.303} \quad (1)$$

where q_e and q are the amounts of phenol adsorbed (mg/g) at equilibrium and at time t (min), respectively, and k_1 the rate constant of adsorption (min^{-1}). Values of k_1 were calculated from the plots of $\log(q_e - q)$ versus t (figures not shown) for different concentrations of phenols. The experimental q_e values do not agree with the calculated ones, obtained from the linear plots (Table 4). This shows that the adsorption of phenol onto coir pith carbon is not a first-order reaction.

The second-order kinetic model [31] is expressed as:

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where k_2 is the rate constant of second-order adsorption ($\text{g}/(\text{mg min})$). If the second-order kinetics is applicable, then the plot of t/q versus t should show a linear relationship. There is no need to know any parameter beforehand and the equilibrium adsorption capacity q_e can be calculated from Eq. (2). Also, it is more likely to predict the behavior over the whole range of adsorption [31]. Values of k_2 and q_e were calculated from the intercept and slope of the plots of t/q versus t . The linear plots of t/q versus t (Fig. 3) show a good agreement

Table 4

Comparison of the first- and second-order adsorption rate constants and calculated and experimental q_e values for different initial phenol concentrations and temperatures

Parameter	First-order kinetic model				Second-order kinetic model		
	q_e (experimental; mg/g)	k_1 (min^{-1})	q_e (calculated; mg/g)	R^2	k_2 (g/(mg min))	q_e (calculated; mg/g)	R^2
Initial phenol concentration (mg/l)							
10	4.309	0.10	2.888	0.981	0.05	4.737	0.990
20	9.025	0.05	4.966	0.946	0.02	9.524	0.998
30	13.57	0.04	2.706	0.989	0.05	13.89	0.999
40	17.58	0.03	6.179	0.975	0.02	17.48	0.998
Temperature ($^{\circ}\text{C}$)							
35	8.874	0.03	2.401	0.942	0.13	7.880	0.998
40	9.095	0.05	2.715	0.965	0.09	8.621	0.998
50	9.182	0.05	2.443	0.919	0.10	8.598	0.997
60	9.288	0.10	3.049	0.999	0.07	9.515	0.999

between experimental and calculated q_e values (Table 4). The correlation coefficients for the second-order kinetic model are greater than 0.990. These indicate that the adsorption system belongs to the second-order kinetic model. Similar phenomena have been observed in the biosorption of dye Remazol Black B on biomass [32,33] and adsorption of Congo Red on coir pith carbon [24].

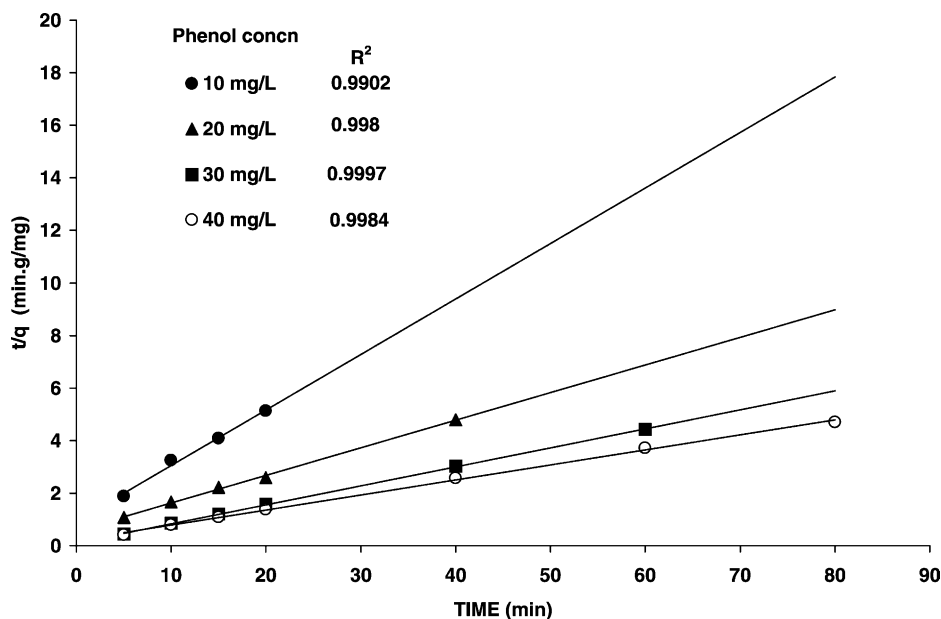


Fig. 3. Plot of the pseudo-second-order model at different initial 2-CP concentrations. Adsorbent dose, 100 mg/50 ml; pH 2.0; 2-CP concentration: (●) 10 mg/l, (▲) 20 mg/l, (■) 30 mg/l, (○) 40 mg/l; temperature, 35 $^{\circ}\text{C}$.

The rate limiting step may be due to intraparticle diffusion. The intraparticle diffusion rate is described by Weber and Morris [34]. If intraparticle diffusion occurs, then q versus $t^{1/2}$ will be linear and if the plot of q versus $t^{1/2}$ passes through the origin, then the rate limiting process is only due to the intraparticle diffusion. Otherwise, some other mechanism along with intraparticle diffusion is also involved. It was found that q versus $t^{1/2}$ plots for all the four concentrations of 2-CP were linear (figures not shown) and they did not pass through the origin. It shows that intraparticle diffusion is not the only rate limiting mechanism. Adsorption occurs in three steps [34]: (1) mass transfer across the boundary layer surrounding the adsorbent particle; (2) mass transfer within the internal structure of the particle; and (3) adsorption at a site.

3.3. Effect of adsorbent dosage

Fig. 4 shows the removal of 2-CP by coir pith carbon at different adsorbent doses (25–500 mg/50 ml) for the 2-CP concentrations of 10, 20, 30 and 40 mg/l. Increase in adsorbent dosage increased the percent removal of 2-CP, which is due to the increase in adsorbent surface area of the adsorbent.

3.4. Modeling of isotherm curves

The different experimental isotherms were modeled according to three equations: the Freundlich, Langmuir and Dubinin–Radushkevich models [35].

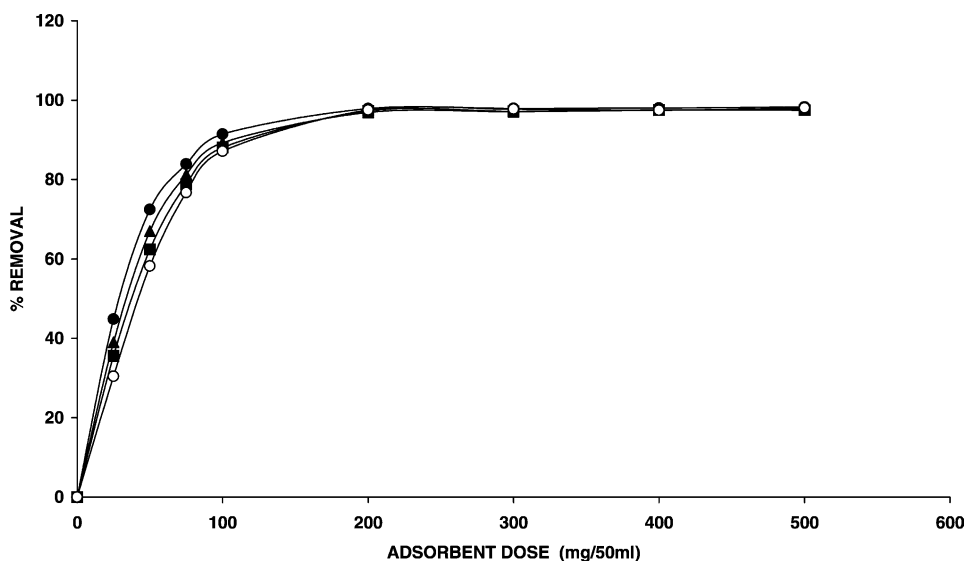


Fig. 4. Effect of adsorbent dosage on removal of 2-CP by coir pith carbon. 2-CP concentration: (●) 10 mg/l (agitation time, 40 min), (▲) 20 mg/l (agitation time, 60 min), (■) 30 mg/l (agitation time, 80 min), (○) 40 mg/l (agitation time, 100 min); temperature, 35 °C.

The Freundlich empirical model [36] is represented by:

$$q_e = k_f C_e^{1/n} \quad (3)$$

where C_e is the equilibrium concentration (mg/l), q_e the amount adsorbed at equilibrium (mg/g), and k_f ($\text{mg}^{1-1/n} \text{l}^{1/n}/\text{g}$) and $1/n$ the Freundlich constants depending on the temperature and the given adsorbent–adsorbate couple. n is related to the adsorption energy distribution, and k_f indicates the adsorption capacity.

Langmuir's model does not take into account the variation in adsorption energy, but it is the simplest description of the adsorption process. It is based on the physical hypothesis that the maximum adsorption capacity consists of a monolayer adsorption, that there are no interactions between adsorbed molecules, and that the adsorption energy is distributed homogeneously over the entire coverage surface. Langmuir's equation is:

$$q_e = \frac{b q_m C_e}{1 + b C_e} \quad (4)$$

where b is the equilibrium adsorption coefficient (l/mg) and q_m the maximum adsorption capacity (mg/g).

Dubinin's approach is based on the earlier potential theory of Polanyi and on the concept of micropore filling. The Dubinin–Radushkevich equation represents the isotherm in terms of fractional filling (q_e/q_{DR}) of the micropore volume in which the maximum adsorption capacity is q_{DR} :

$$\frac{q_e}{q_{\text{DR}}} = \exp(-B \varepsilon^2) \quad (5)$$

where q_{DR} is the maximum adsorption capacity in the micropore volume (mol/g), ε the Polanyi adsorption potential, and B the characteristic parameter related to adsorption energy for the given system (mol^2/kJ^2)

The Polanyi adsorption potential ε is an adsorption affinity defined in the liquid phase by:

$$\varepsilon = RT \ln \left(\frac{C_s}{C_e} \right) \quad (6)$$

where T is the temperature (K) and C_s the solubility of the solute (mol/l).

Compared with Langmuir's theory, the Dubinin approach assumes that there is a surface area where the adsorption energy is homogeneous. The characteristic adsorption energy E_0 (kJ/mol) is then given by:

$$E_0 = \frac{1}{B^{1/2}} \quad (7)$$

The three models were applied to experimental data.

Linear plots of $\log_{10}(x/m)$ versus $\log_{10} C_e$ show that the adsorption follows Freundlich isotherm (Fig. 5). In general, as the k_f value increases, the adsorption capacity also increases. Values of k_f and n were calculated from the intercept and slope of the plots and are presented in Table 5. Values of k_f and n for the adsorption of 2-CP by activated carbons reported in literature are shown in Table 6. The equilibrium adsorption capacity for different initial

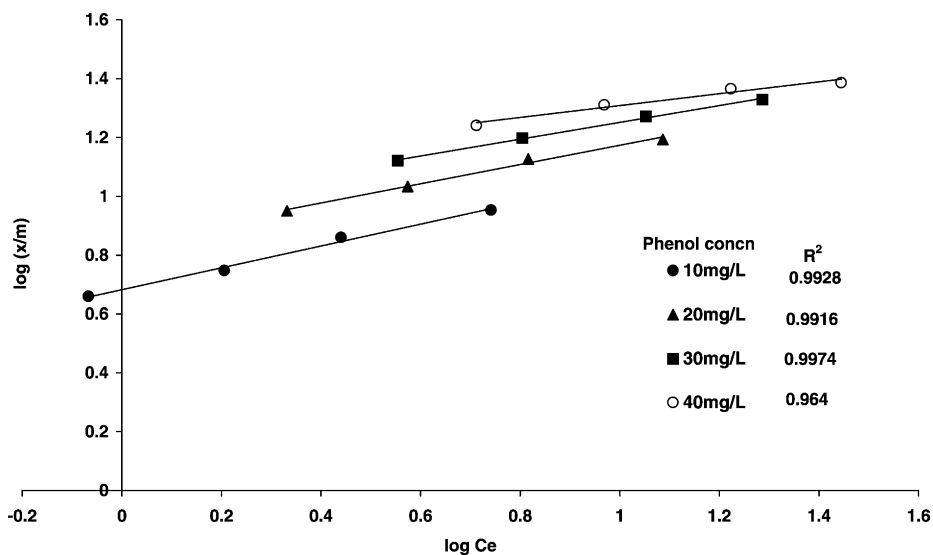


Fig. 5. Freundlich plots for adsorption of 2-CP by coir pith carbon. 2-CP concentration: (●) 10 mg/l, (▲) 20 mg/l, (■) 30 mg/l, (○) 40 mg/l.

Table 5
Freundlich constants

Phenol concentration (mg/l)	k_f ($\text{mg}^{1-1/n} \text{l}^{1/n}/\text{g}$)	n
10	4.81	2.70
20	7.02	3.06
30	9.24	3.50
40	12.8	4.96

Table 6
Freundlich constants for 2-chlorophenol adsorption by various adsorbents reported in literature

Adsorbent	k_f ($\text{mg}^{1-1/n} \text{l}^{1/n}/\text{g}$)	n	Reference
Dried activated sludge	13.50	2.07	[39]
Fly ash	1.49	1.00	[39]
Granular activated carbon (Nuchar WV-H, Westvaco, USA)	19.24	1.77	[39]
Red mud	1.134	1.165	[40]
GACs from Norit N.V., Amersfoort, The Netherlands			
RGM 1	2.512	2.066	[41]
RB 2	2.518	2.179	[41]
ROW 0.8 supra	3.750	2.439	[41]
CGran	0.667	2.717	[41]

Table 7

The equilibrium adsorption capacity (mg/g) for different initial concentrations of 2-CP (C_0) and outlet concentrations of 2-CP after treatment (C_e) based on Freundlich isotherm

C_0 (mg/l)	C_e (=1.0 mg/l)	C_e (=5.0 mg/l)
10	4.81	8.73
20	7.23	11.88
30	9.24	14.64
40	12.78	17.72

concentrations of 2-CP based on Freundlich isotherm is shown in Table 7. Adsorption equilibrium data do not follow Langmuir and Dubinin–Radushkevich isotherm models.

3.5. Effect of pH

Effect of pH on the removal of 2-chlorophenol is shown in Fig. 6. The percent removal decreased with increase in pH. Based on the relationship [37]:

$$CP_0 = \frac{CP_T}{1 + 10^{(pH - pK_a)}} \quad (8)$$

(where CP_0 is the concentration of unionized chlorophenol species, CP_T the total concentration of chlorophenol taken, pH the final pH (equilibrium pH) after adsorption, and pK_a is 8.35), the concentrations of ionized chlorophenol species (chlorophenolate) were calculated

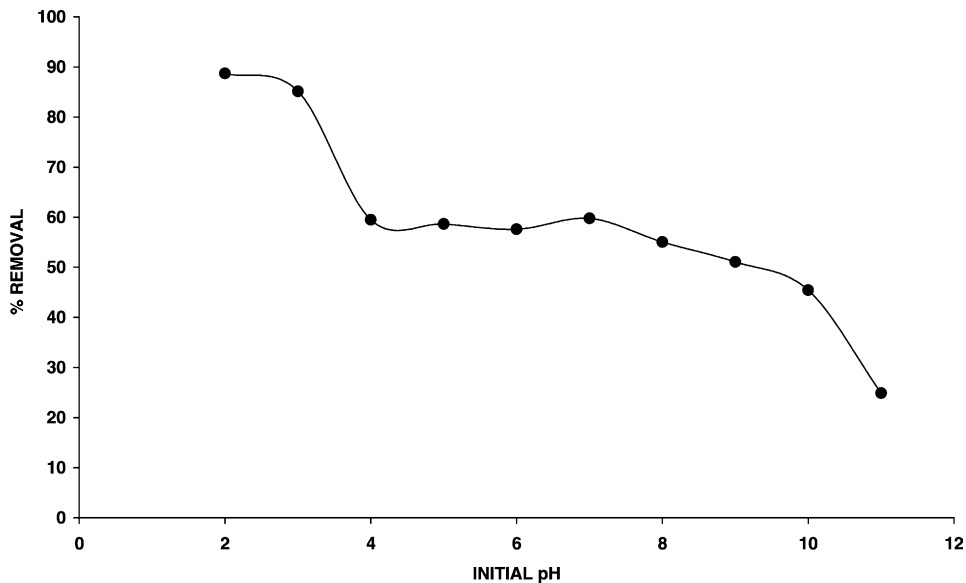


Fig. 6. Effect of pH on removal of 2-CP by coir pith carbon. Adsorbent dose, 100 mg/50 ml; 2-CP concentration, 10 mg/l; agitation time, 40 min; temperature, 35 °C.

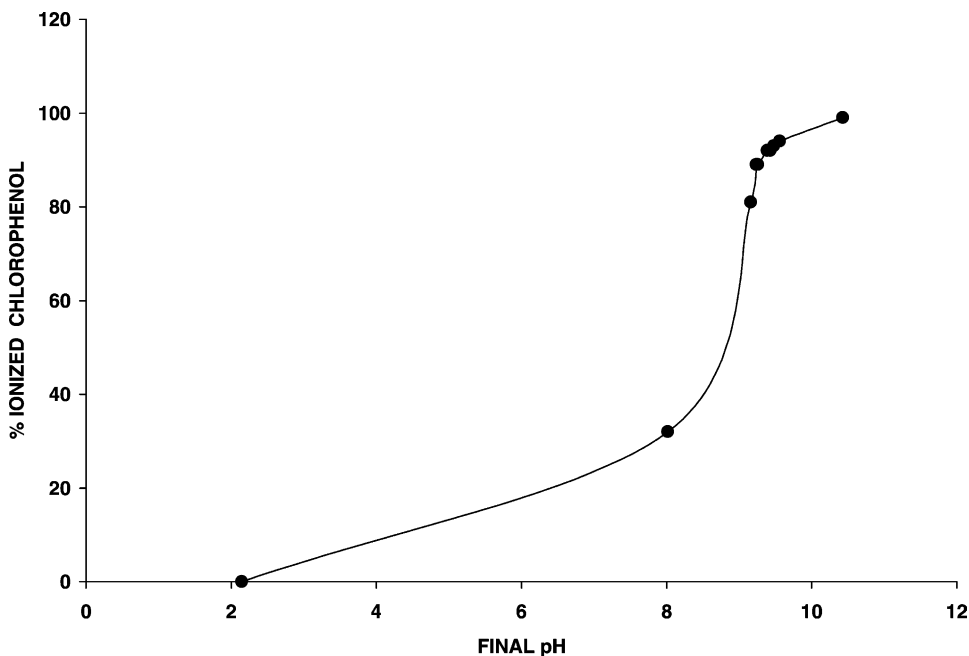


Fig. 7. Effect of final pH on ionization of 2-chlorophenol. 2-CP concentration, 10 mg/l; agitation time, 40 min; temperature, 35 °C.

at different final pH values and a plot of the percent ionized chlorophenol versus final pH is shown in Fig. 7. Fig. 7 shows an increase of ionized chlorophenol concentration from 0% at final pH 2.2 to 99% at final pH 10.4. At acidic pH ($<pH_{ZPC}$), the adsorbent surface is positively charged and at final pH 2.2 and 8.0, the percent unionized chlorophenol is 100 and 68, respectively. Since there is no electrostatic repulsion between the unionized chlorophenol species and the positively charged adsorbent surface at pH 2.0, the removal is higher. As the initial pH is increased, the percent of unionized species decreases and of the ionized species increases. At the same time the number of negatively charged sites on the adsorbent surface increases. At the initial pH of 11 (final pH 10.4), the percent ionized species is 99% and the percent removal observed by the highly negatively charged adsorbent surface is the least, i.e. 25%. At alkaline pH ($>pH_{ZPC}$), the adsorbent surface is negatively charged. Hence, decrease in adsorption is observed due to electrostatic repulsion between the negatively charged adsorbent surface and chlorophenolate species. Despite the electrostatic repulsion at the initial pH 11, a significant removal (25%) was observed. This indicates that chemisorption might be involved in the removal process.

3.6. Desorption studies

Regeneration of spent carbon and recovery of 2-CP would make the treatment process economical. Also desorption studies help elucidate the mechanism of adsorption. The

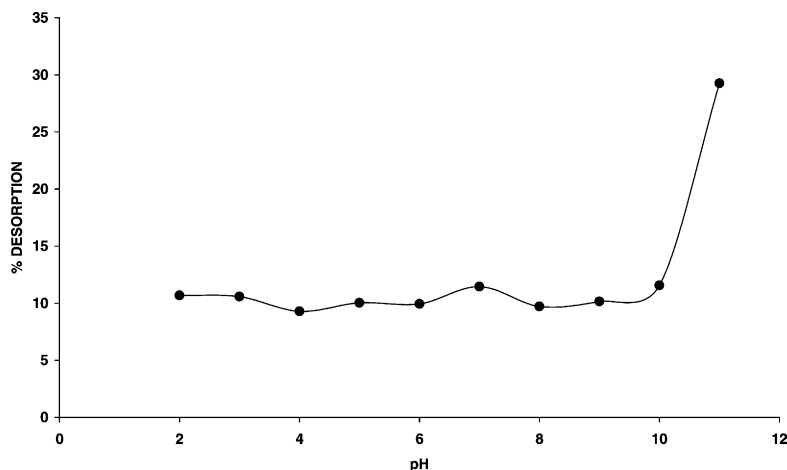


Fig. 8. Effect of pH on desorption of 2-CP from 2-CP-loaded adsorbent. Adsorbent dose, 100 mg/50 ml; 2-CP concentration, 10 mg/l; agitation time, 40 min; temperature, 35 °C.

percent desorption increased from 10 to 30%, when the pH was increased from 2 to 11 (Fig. 8). Only the physisorbed 2-CP unlike the chemisorbed species might be desorbed from the adsorbent surface. Both pH effect and desorption studies show that the chemisorption might be the major mode of removal of 2-CP by the coir pith carbon.

3.7. Effect of temperature

Effect of agitation time and temperature on 2-CP is shown in Fig. 9. Increase of temperature slightly increased the percent removal. The change in standard free energy, enthalpy and entropy of adsorption were calculated using the following equation:

$$\Delta G_0 = -RT \ln K_c \quad (9)$$

where R is the gas constant, K_c the equilibrium constant, and T the temperature (K).

According to van't Hoff equation:

$$\log_{10} K_c = \frac{\Delta S^0}{2.303R} - \frac{\Delta H^0}{2.303RT} \quad (10)$$

where ΔS^0 and ΔH^0 are the changes in entropy and enthalpy of adsorption, respectively. Plot of $\log K_c$ versus $1/T$ is linear (Fig. 10). Values of ΔH^0 and ΔS^0 were evaluated from the slope and intercept of van't Hoff plots (Table 8). The positive values of ΔH^0 confirm the endothermic nature of adsorption. The endothermic adsorption has also been reported for the adsorption of Direct Red 12B on biogas waste slurry [38]. The negative values of ΔG^0 at 35, 40, 50 and 60 °C indicates spontaneous nature of adsorption for 2-chlorophenol. The positive values of ΔS^0 suggest the increased randomness at the solid–solution interface during the adsorption of 2-chlorophenol on coir pith carbon.

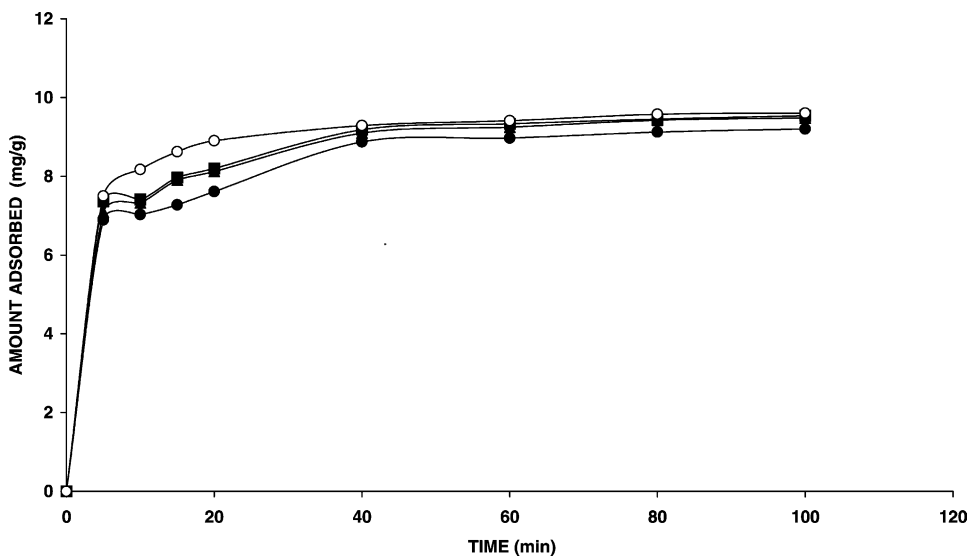


Fig. 9. Effect of temperature on removal of 2-CP by coir pith carbon. Adsorbent dose, 50 mg/50 ml; 2-CP concentration, 10 mg/l; pH 2.0; temperature: (●) 35 °C, (▲) 40 °C, (■) 50 °C, (○) 60 °C.

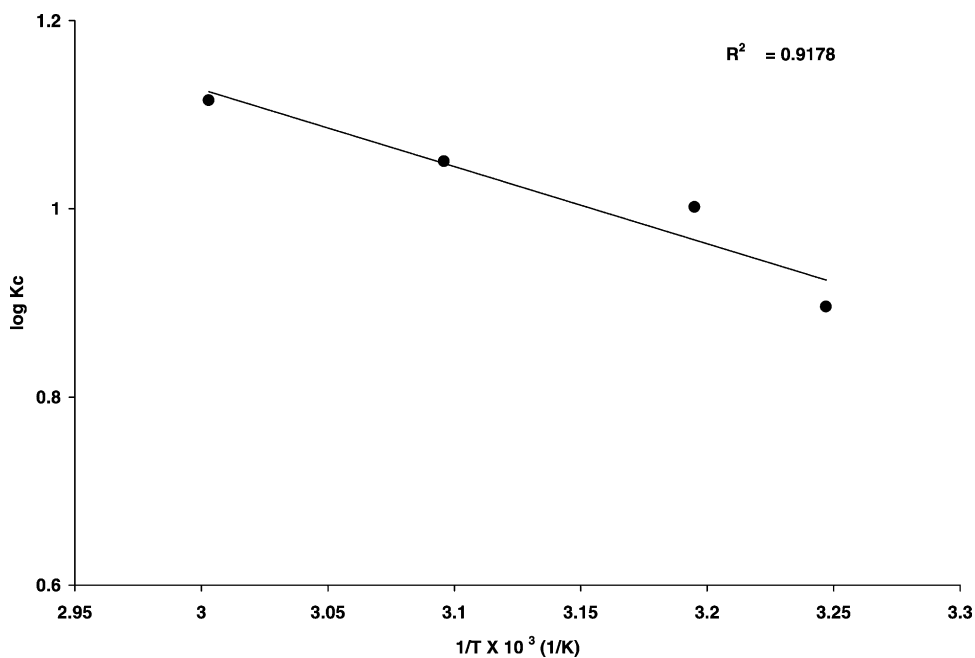


Fig. 10. The van't Hoff plot for 2-CP adsorption.

Table 8
Thermodynamical parameters

Temperature (°C)	K_c	ΔG^0 (kJ/mol)	ΔH^0 (kJ/mol)	ΔS^0 (J/(mol k))
35	7.88	−5.29	15.66	68.56
40	10.05	−6.01		
50	11.23	−6.50		
60	13.05	−7.11		

The first-order rate constants of adsorption (k_1) were evaluated from the plots of $\log(q_e - q)$ versus t for different temperatures using the Lagergren first-order rate expression. The calculated q_e values obtained from the first-order kinetic model do not agree with the experimental q_e values (Table 4). This shows that the adsorption of 2-chlorophenol on coir pith carbon at different temperatures does not follow the first-order kinetic model. Linear plots of t/q versus t corresponding to the second-order kinetic model were obtained (Fig. 11). The correlation coefficients are greater than 0.9970. Also, the calculated values of q_e agree very well with the experimental data. This indicates that the adsorption follows second-order kinetic model at different temperatures used in this study.

The conventional raw materials for the preparation of activated carbons are coconut shell, coal, lignite, peat, etc., which are available at a price. The price of the activated carbon made from these raw materials is due to the cost of both the raw material and the activation process.

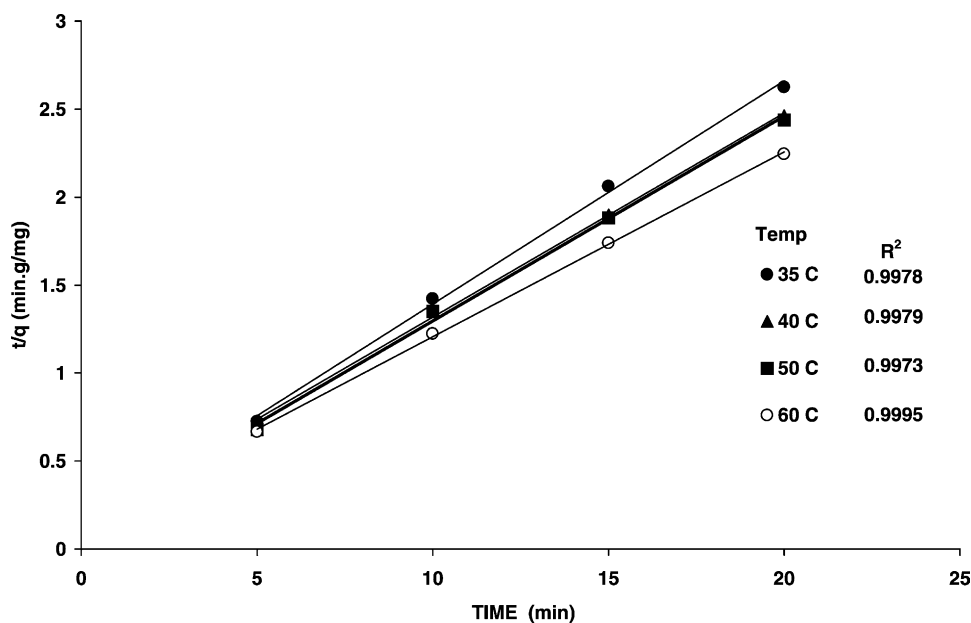


Fig. 11. Plot of the pseudo-second-order model at different temperatures. Adsorbent dose, 50 mg/50 ml; 2-CP concentration, 10 mg/l; pH 2.0; temperature: (●)35 °C, (▲) 40 °C, (■) 50 °C, (○) 60 °C.

Whereas, the non conventional raw material used here, namely, coir pith is available free of cost, as this is a solid waste discarded by coconut coir industry. So the cost of the activated carbon of coir pith arises only due to activation process. Also, the disposal cost for coir pith provided by the coir industry reduces the production cost of coir pith carbon. Hence, the coir pith carbon is considered to be economical.

4. Conclusion

The present study shows that the coir pith carbon is an effective adsorbent for the removal of 2-chlorophenol from aqueous solution. Adsorption equilibrium follows Freundlich isotherm. Kinetics of adsorption follows second-order model. Complete removal of phenol can be achieved using appropriate dosage of the adsorbent and pH of wastewaters. The results would be useful for the fabrication and designing of wastewater treatment plants for the removal of 2-chlorophenol. Also the treatment method is expected to be economical.

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