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Adsorptive Removal of 2,4-Dichlorophenol from Aqueous Solution by Low-Cost Carbon from an Agricultural Solid Waste: Coconut Coir Pith

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ABSTRACT

Coconut coir pith, a solid waste generated in coir fibre industries, was carbonized and used as adsorbent for the removal of 2,4-dichlorophenol (2,4-DCP) from water. Varying parameters such as agitation time, 2,4-DCP concentration, adsorbent dose, pH, and temperature were investigated. Adsorption equilibrium reached at 60, 80, 100, and 120 min for 2,4-DCP concentrations of 10, 20, 30, and 40 $\mathrm{mg\,L^{-1}}$, respectively. Adsorption followed pseudo-second order kinetics. The adsorption equilibrium data moderately obeyed Langmuir and Freundlich isotherms.

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The adsorption capacity was found to be 19 mg g^{-1} of carbon. Increase of temperature increased adsorption. Acidic pH was favorable for the adsorption of 2,4-DCP. Studies on pH effect and desorption show that both physisorption and chemisorption were involved in the adsorption process.

Key Words: 2,4-Dichlorophenol; Adsorption; Coir pith carbon; Isotherms; pH effect; Desorption studies.

INTRODUCTION

Phenol and phenolic compounds in wastewaters are hazardous pollutants, which are present in effluents from industries such as pharmaceutical, petrochemical refinery, herbicide, pesticide, and coal conversion industries,[1] and paper and pulp industries.[2] Phenolic compounds have been found to accelerate tumor formation, cancer, and mutation.[3] According to the Indian standards, the permissible limit for phenolic compounds in industrial effluents before discharging into municipal sewers and surface waters are 5 and 1 mg /L, respectively.[4] The guideline of WHO[5] and EPA[6] for maximum admissible concentration for 2,4-dichlorophenol (2,4-DCP) in drinking water is 40 $\mu g L^{-1}$. Removal of phenolic compounds from wastewater is therefore of utmost importance to prevent pollution of water in the receiving watercourse.

Biological treatment, adsorption, and solvent extraction are widely used conventional methods for removing phenols from wastewaters. Commercially available activated carbons (Calgon Corp., Pittsburgh, PA) are used for the adsorption of chlorinated phenols.[7] But these are expensive and the regeneration costs are high. Hence this has led many workers to search for more economic, practical, and efficient adsorbents. Bituminous coal, pine bark, red mud, fly ash, anion exchange resins, montmorillonite and silica, iron-loaded marble, polystyrene, paper mill sludge, biological activated carbon, straw, coconut shell, wood, tyre, etc. are some of the new adsorbents used for phenols.[8] Catalytic[9] and sonochemical[10] degradation of chlorophenol have also been investigated. Coir pith is a byproduct of coconut coir processing industry. More than 7.5 million tonnes of coir pith is generated annually in India.[11] The aged heaps of coir pith are readily available in the coir factory sites in southern India. Different types of activated carbon were prepared from coir pith by physical and chemical methods in our laboratory[12] and used for the removal of dyes,[13] 2-chlorophenol[14] and heavy metals.[15] The purpose of this study was to investigate the feasibility of using carbonized coir pith for the removal of 2,4-DCP from aqueous solution by adsorption method.

EXPERIMENTAL

Materials

Coir pith was collected from nearby coir industries, dried in sunlight for 5 hr and ground. The dried coir pith powder was sieved to 250–500 μm size. It was subjected to carbonization at 700°C for 1 hr 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.[13] 2,4-DCP was obtained from G.S. Chemical Testing Lab and Allied Industries, New Delhi. The characteristics of the carbon are shown in Table 1. For comparison the characteristics of activated carbon made from coconut husk is also given in Table 1.[16]

Methods

Adsorption Studies

Adsorption experiments were carried out by agitating 100 mg of adsorbent with 50 mL of 2,4-DCP solution of desired concentration and pH at 200 rpm, 35°C in a thermostated rotary shaker (ORBITEK, Chennai, India). 2,4-DCP concentration was estimated spectrophotometrically by monitoring the absorbance at 283.8 nm using UV-VIS spectrophotometer (Hitachi, model U-3210, Tokyo, Japan). pH was measured using pH meter (Elico, model LI-107, Hyderabad, India). The samples were withdrawn from the shaker at predetermined time intervals and the 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 2,4-DCP solutions using dilute HCl and NaOH solutions and the solutions were agitated with 100 mg 50 mL^{-1} adsorbent dose at equilibrium time for 10 and 20 mg L^{-1} 2,4-DCP concentrations. Effect of adsorbent dose was studied with different adsorbent doses (20–400 mg) and 50 mL of 10, 20, 30, 40 mg L^{-1} 2,4-DCP solutions at equilibrium time. Langmuir and Freundlich isotherms were employed to study the equilibrium adsorption and they were obtained from the data of time curves and adsorbent dose effect, respectively. Effect of temperature was studied using 10 mg L^{-1} of 2,4-DCP and 50 mg of adsorbent at 35°C, 40°C, 50°C, and 60°C.

Table 1. Comparison of the characteristics between coir pith carbon and activated carbon from coconut husk.

Physical parameter	Coir pith carbon	Activated carbon from coconut husk[16]
Specific surface area ($m^2 g^{-1}$)	167	421
Micropore volume ($cm^3 g^{-1}$)	0.0913	—
Total pore volume ($cm^3 g^{-1}$)	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	3.45
pH (1% solution)	10.1	—
Conductivity (1% solution) ($mS\,cm^{-1}$)	2.3	—
Bulk density ($g\,mL^{-1}$)	0.12	1.2
Mechanical moisture content (%)	5.88	6.0
Ash content (%)	79.87	2.3
Specific gravity	1.742	—
Porosity (%)	93.11	51
Volatile matter (%)	58.38	—
Fixed carbon (%)	41.62	—
Decolorising power ($mg\,g^{-1}$)	21.0	—
Iodine number ($mg\,g^{-1}$)	101.52	579
Ion exchange capacity	Nil	—
Chemical parameters		
Sodium (%)	0.14	—
Potassium (%)	0.18	—
Calcium (%)	0.22	—
Phosphorous (%)	0.01	—
Iron (%)	0.18	—

Desorption Studies

The adsorbent ($100\,mg\,50\,mL^{-1}$) that was used for the adsorption of 10 or $20\,mg\,L^{-1}$ of 2,4-DCP solution was separated from the solution by centrifugation. The 2,4-DCP-loaded adsorbent was filtered using Whatman filter paper and washed gently with water to remove any unadsorbed 2,4-DCP. Several such samples were prepared. Then the spent adsorbent was mixed with 50 mL of distilled water, adjusted to different pH values and agitated at equilibrium time for 10 and $20\,mg\,L^{-1}$ of 2,4-DCP. The desorbed 2,4-DCP was estimated as before.

RESULTS AND DISCUSSION

Effects of Agitation Time and Concentration of 2,4-DCP on Adsorption

Effects of agitation time and 2,4-DCP concentrations on removal of 2,4-DCP by coir pith carbon show that the amount adsorbed ($\mathrm{mg\,g^{-1}}$) increased with increase in agitation time and 2,4-DCP concentration and remained nearly constant after equilibrium time. The equilibrium time increased with increase in 2,4-DCP concentration. The equilibrium time for 10, 20, 30, and 40 $\mathrm{mg\,L^{-1}}$ of 2,4-DCP was found to be 60, 80, 100, and 120 min, respectively (figures not shown).

Adsorption Dynamics

Adsorption Rate Constant

The rate constant of adsorption is determined from the first order rate expression given by Lagergren:[17]

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

where q_e and q are the amounts of 2,4-DCP adsorbed ($\mathrm{mg\,g^{-1}}$) at equilibrium and at time t (min), respectively and k_1 is the rate constant of adsorption ($1\,\mathrm{min^{-1}}$). Values of k_1 were calculated from the plots of $\log(q_e - q)$ vs. t (figures not shown) for different concentrations of 2,4-DCP. The experimental q_e values do not agree with the calculated ones, obtained from the linear plots (Table 2). This shows that the adsorption of 2,4-DCP onto coir pith carbon is not a first order reaction.

The second order kinetic model[18] is expressed as

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

where k_2 ($\mathrm{g\,mg^{-1}\,min^{-1}}$) is the rate constant of second order adsorption. Values of k_2 and q_e were calculated from the intercept and slope of the plots of t/q vs. t. The linear plots of t/q vs. t (Fig. 1) show a good agreement between experimental and calculated q_e values (Table 2). Also the correlation coefficients for the second order kinetic model are good. This indicates that the adsorption system belongs to the pseudo-second order kinetic model. Similar

Table 2. Comparison of the first and second order adsorption rate constants and calculated and experimental q_e values for different initial 2,4-DCP concentrations and temperatures.

		First order kinetic model			Second order kinetic model		
Parameter	q_e (exp, mg g^{-1})	k_1 (L min^{-1})	q_e (cal, mg g^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	q_e (cal, mg g^{-1})	R^2
2,4-DCP conc. (mg L^{-1})							
10	4.767	0.059	2.407	0.987	0.108	4.675	0.997
20	9.480	0.033	4.269	0.998	0.021	9.533	0.997
30	12.82	0.026	5.750	0.987	0.014	12.72	0.996
40	16.74	0.032	7.581	0.993	0.011	17.18	0.999
Temperature (°C)							
35	6.877	0.032	4.152	0.973	0.018	7.067	0.991
40	7.112	0.034	4.096	0.970	0.019	7.309	0.994
50	7.268	0.035	3.602	0.964	0.024	7.413	0.995
60	7.519	0.051	2.367	0.996	0.053	7.698	0.999

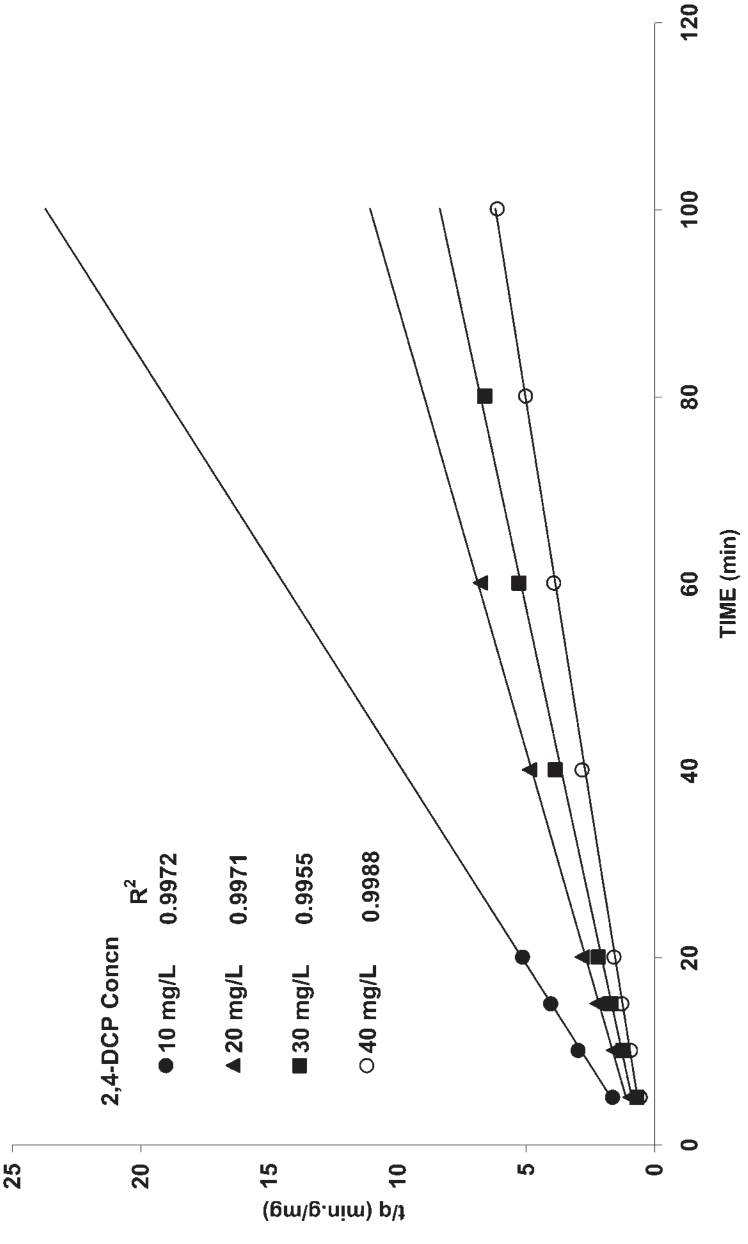

Figure 1. Plot of the pseudo-second order model at different initial 2,4-DCP concentrations: adsorbent dose, 100 mg 50 mL^{-1}; initial pH, 2.0; temperature, 35°C.

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phenomenon has been observed in the adsorption of 2-chlorophenol by coir pith carbon,[14] and dyes and heavy metals by activated carbon.[18]

Adsorption Isotherms

Two widely used isotherms for modeling the adsorption equilibrium are Langmuir and Freundlich isotherms.

Langmuir isotherm is represented by the following equation:[19]

$$\frac{C_e}{q_e} = \frac{l}{Q_o b} + \frac{C_e}{Q_o} \tag{3}$$

where C_e is the concentration of 2,4-DCP solution ($\mathrm{mg\,L^{-1}}$) at equilibrium. The constant Q_o signifies the adsorption capacity ($\mathrm{mg\,g^{-1}}$) and b is related to the energy of adsorption ($\mathrm{L\,mg^{-1}}$). The Langmuir model[20] (p. 83) represents chemisorption on a set of well-defined localized adsorption sites having the same sorption energies independent of surface coverage and no interaction between adsorbed molecules. Langmuir isotherm assumes monolayer coverage of adsorbate on the adsorbent. A plot of C_e/q_e vs. C_e shows a correlation coefficient of only 0.9648, which shows that adsorption moderately follows Langmuir isotherm (Fig. 2). Values of Q_o and b were calculated from the slope and intercept of the linear plots and are presented in Table 3 along with the literature data. Though the activated carbon made from apricot stone shells and H_3PO_4 shows highest removal, the cost for making this carbon is high due to the inclusion of H_3PO_4. The essential characteristics of Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter R_L, defined by Weber and Chakkravorti:[24]

$$R_L = \frac{1}{1 + bC_0} \tag{4}$$

where b is the Langmuir constant and C_0 is the initial 2,4-DCP concentration ($\mathrm{mg\,L^{-1}}$), R_L values were found to be 0.12, 0.07, 0.04, and 0.03 for the concentration of 10, 20, 30, and 40 $\mathrm{mg\,L^{-1}}$, respectively. The obtained R_L values between zero and one indicate favorable adsorption.[24]

Freundlich isotherm was also applied to plot the equilibrium data of the adsorption:[25]

$$\log_{10}\left(\frac{x}{m}\right) = \log_{10} k_f + \left(\frac{1}{n}\right)\log_{10} C_e \tag{5}$$

where x is the amount of 2,4-DCP adsorbed (mg), m is the weight of the adsorbent used (g), C_e is the equilibrium concentration of 2,4-DCP in solution ($\mathrm{mg\,L^{-1}}$), k_f ($\mathrm{mg^{1-1/n}\,L^{1/n}\,g^{-1}}$) and n are constants incorporating all factors

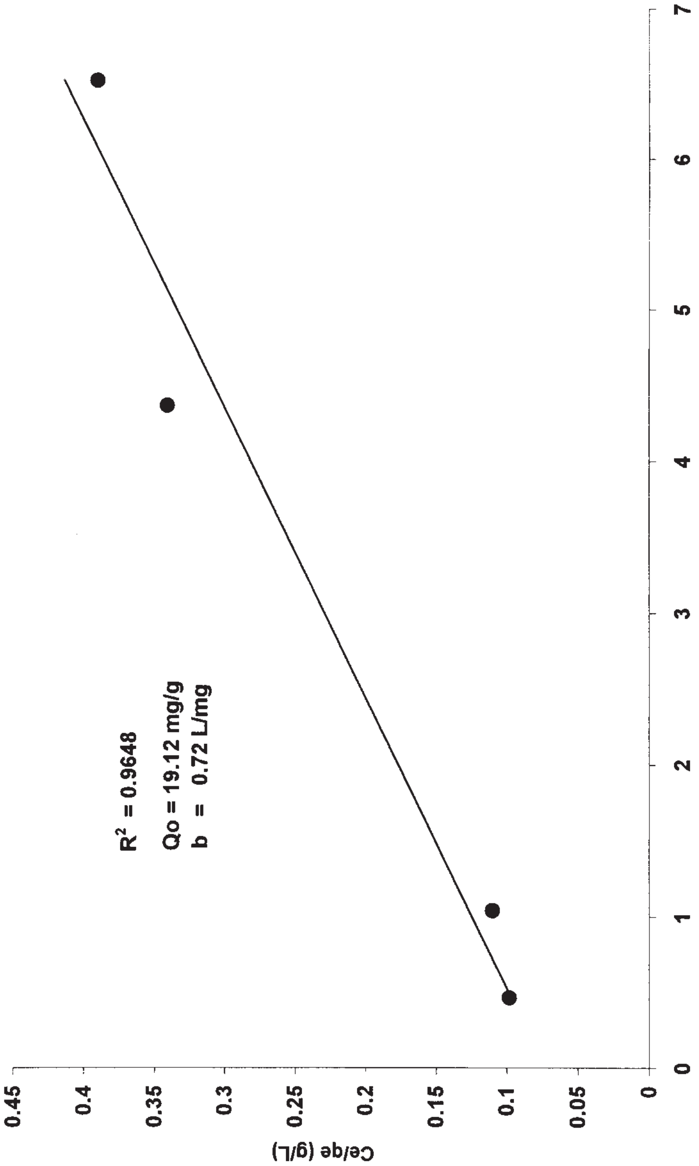

Figure 2. Langmuir plot for the adsorption of 2,4-dichlorophenol by coir pith carbon.

Table 3. Langmuir constants.

Adsorbent	Q_o (mg g^{-1})	b (L mg^{-1})	Reference
Coir pith carbon	19.12	0.72	This work
Activated carbon (apricot stone shells-H_3PO_4 impregnated)	339	0.197	[21]
Paper mill sludge	4.49	0.003	[22]

affecting the adsorption process (adsorption capacity and intensity). The Freundlich sorption isotherm[20] (p. 108), one of the most widely used mathematical descriptions, usually fits the experimental data over a wide range of concentration. The isotherm gives an expression encompassing the surface heterogeneity and the exponential distribution of active sites and their energies. This isotherm does not predict any saturation of the sorbent surface; thus, infinite surface coverage is predicted mathematically, indicating physisorption on the surface. Figure 3 shows plots of $\log(x/m)$ vs. $\log C_e$. Good correlation coefficient (0.9876) was obtained only for 10 mg L^{-1} concentration. Values of k_f and n were calculated from the intercept and slope of the plots and are presented in Table 4. Values of k_f and n for the adsorption of 2,4-DCP by commercial activated carbons and others reported in literature are shown in Table 5. In general, as the k_f value increases, the adsorption capacity also increases. n values between 1 and 10 show beneficial adsorption.[28] Activated carbon made from apricot stone shells and H_3PO_4 shows highest k_f value among all the adsorbents, indicating the highest adsorption capacity. Comparing the k_f values of commercial activated carbons from Norit N. V. Amersfort, The Netherlands with that of the carbonized coir pith, it is obvious that they are in same range. This means that the efficiency of the carbonized coir pith is on a par with the commercial activated carbons.

Effect of pH

The pH of the adsorption medium is the most significant parameter in the treatment of chlorophenols by the adsorbent.[29] The percent removal decreased with increase in pH (Fig. 4). The degree of ionization of phenols depends on the pH of the medium. Based on the relationship.[30]

$$\mathrm{CP}_0 = \frac{\mathrm{CP}_\mathrm{T}}{1 + 10^{(\mathrm{pH}-\mathrm{p}Ka)}} \tag{6}$$

(where CP_0 is the concentration of unionized 2,4-DCP species, CP_T is the total concentration of 2,4-DCP taken, pH is the final pH, i.e., equilibrium

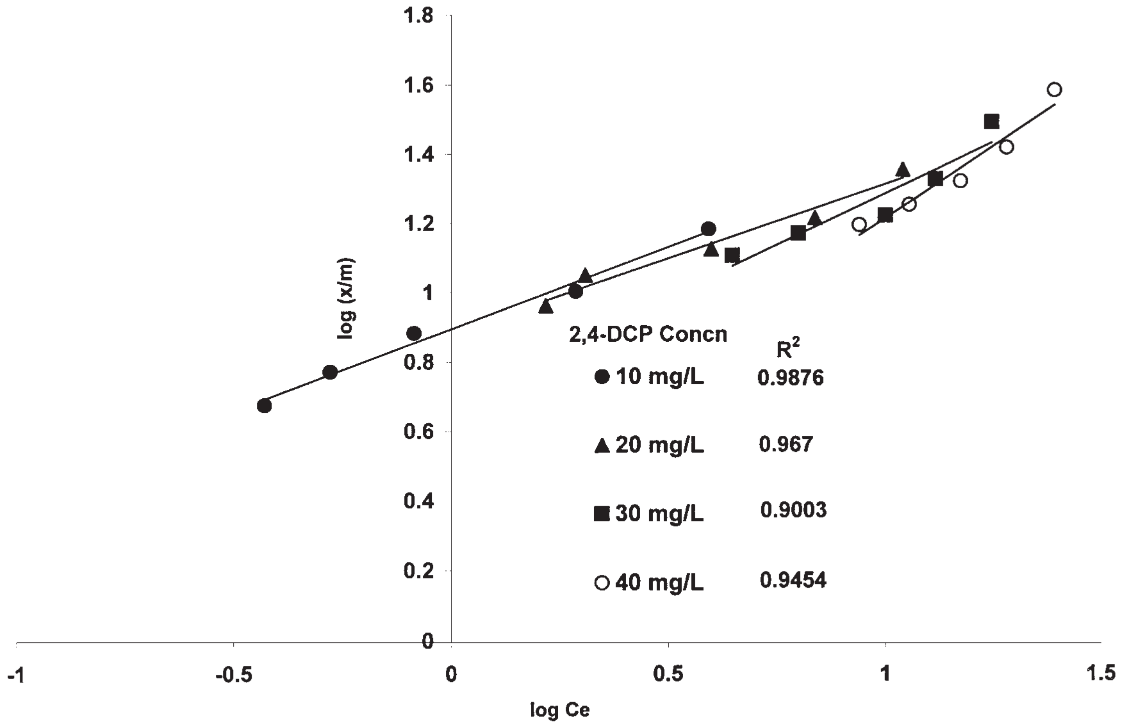

Figure 3. Freundlich plots for the adsorption of 2,4-dichlorophenol by coir pith carbon.

Table 4. Freundlich constants.

2,4-DCP concentration ($mg\,L^{-1}$)	k_f ($mg^{1-1/n}\,L^{1/n}\,g^{-1}$)	n
10	7.86	2.12
20	7.67	2.33
30	4.94	1.69
40	2.45	1.21

pH after adsorption and p*K*a is 7.85) the concentration of ionized 2,4-DCP species (dichloro2,4-DCPate) were calculated at different final pH values and a plot of the per cent ionized 2,4-DCP vs. final pH is shown in Fig. 5. Effect of initial pH on final pH in the presence and absence of 2,4-DCP shows (figure not shown) that the final pH is generally higher than the initial pH in the blank, i.e., in the absence of 2,4-DCP. This is due to the leaching of cations like Na, K from the carbon and subsequent replacement of protons from the solution on the adsorbent surface. Adsorption of neutral 2,4-DCP at pH 2 (unionized; see Fig. 5) did not cause any change in pH, as expected.

Table 5. Freundlich constants for 2,4-dichlorophenol adsorption by various adsorbents reported in literature.

Adsorbent	k_f ($mg^{1-1/n}\,L^{1/n}\,g^{-1}$)	n	Reference
GACs from Norit N. V. Amersfoort, The Netherlands			[26]
RGM 1	6.934	1.828	
RB 2	5.794	1.961	
ROW .8 supra	9.333	2.294	
CGran	1.750	1.859	
Fly ash	7.20	86.96	[27]
Activated carbon (apricot stone shells-H_3PO_4 impregnated)	43.65	1.50	[21]
Activated sludge			[23]
Biomass I	0.94	0.72	
Biomass II	0.37	0.90	
Biomass III	0.44	0.90	

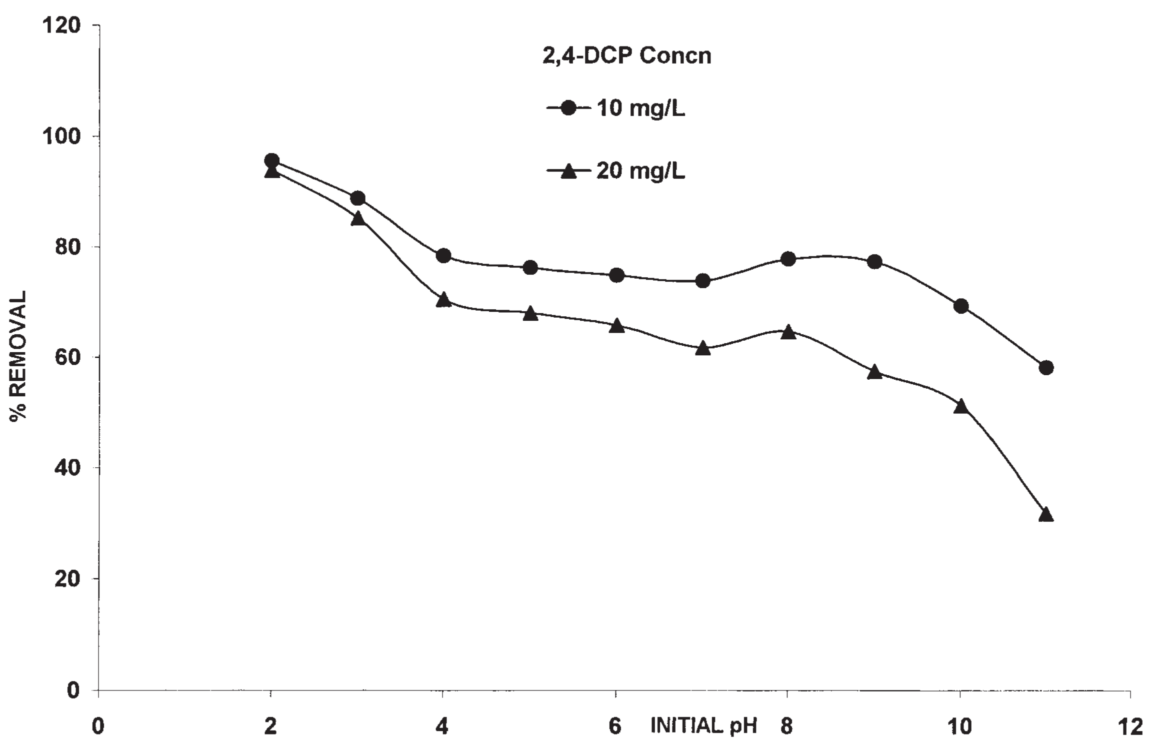

Figure 4. Effect of initial pH on removal of 2,4-dichlorophenol by coir pith carbon; Adsorbent dose, 100 mg 50 mL^{-1}; temperature, 35°C.

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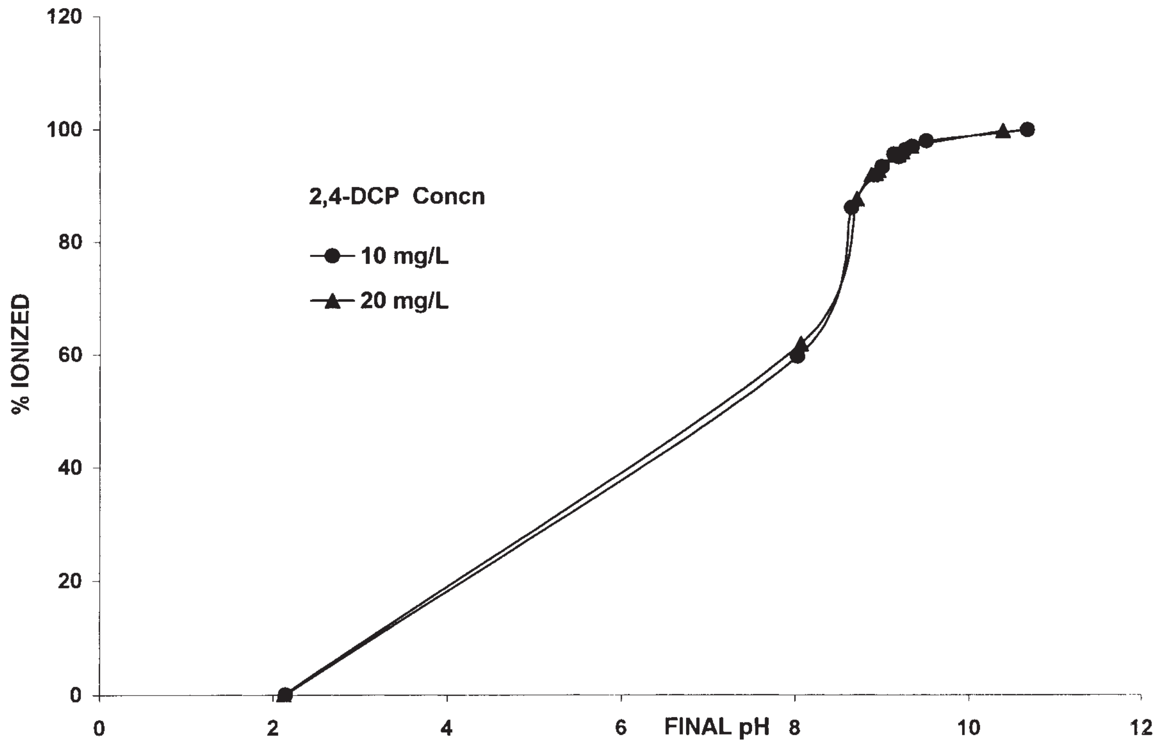

Figure 5. Effect of final pH on ionization of 2,4-dichlorophenol; temperature, 35°C.

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It appears that chemisorption is the only mode in the adsorption process at pH 2. At pH 2, adsorption of 2,4-dichlorophenolate ions release OH^- ions from the adsorbent surface and this increases the final pH further compared to blank. Since at acidic pH values 2,4-DCP is present mostly as the neutral species and the adsorbent surface carries positive charge, there is no electrostatic repulsion between the adsorbate and the adsorbent. In effect the removal is higher at lower pH values. When the pH is increased the concentration of the ionized 2,4-DCP and the number of negatively charged sites on the adsorbent also increase. As a result adsorption is lowered due to repulsive force between the adsorbate and the adsorbent.[29,31] Despite the electrostatic repulsion at alkaline pH, a significant removal was observed. This indicates that chemisorption might also be involved in the removal process.

Desorption Studies

Regeneration of exhausted carbon and recovery of 2,4-DCP contribute to the economy of wastewater treatment. Also desorption studies help elucidate the mechanism of adsorption. The percent desorption increased from 0.7 when the pH was increased from 2.0 for $10\,\mathrm{mg\,L^{-1}}$ 2,4-DCP concentration and it was 40% at 1 M NaOH. At alkaline pH, OH^- ions displaced the 2,4-dichlorophenolate ions from the adsorbent surface. This shows that physical adsorption occurs in the adsorption process. Studies on both pH effect and desorption suggest that both physisorption and chemisorption are involved in the adsorption process.

Effect of Temperature

Effect of agitation time and temperature on 2,4-DCP shows (figure not shown) that increase in temperature slightly increased the percent removal. The change in standard free energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0) of adsorption were calculated using the following equations:

$$\Delta G^0 = -RT \ln K_c \tag{7}$$

where R is gas constant and K_c is the equilibrium constant and T is the temperature in K. According to van't Hoff equation,

$$\log_{10} K_c = \frac{\Delta S^0}{2.303R} - \frac{\Delta H^0}{2.303RT} \tag{8}$$

Values of ΔH^0 and ΔS^0 were evaluated from the linear van't Hoff plot of $\log K_c$ vs. $1/T$ (Table 6; figure not shown). The positive values of ΔH^0 confirm the endothermic nature of adsorption. Exothermic adsorption has been

Table 6. Thermodynamical parameters.

Temperature (°C)	K_c	ΔG^0 (kJ mol^{-1})	ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})
35	2.202	−2.02	10.18	39.78
40	2.463	−2.35		
50	2.660	−2.63		
60	3.031	−3.07		

reported for the adsorption of 2-chlorophenol[14] and 2,4-DCP by fly ash.[27] The negative values of ΔG^0 at 35°C, 40°C, 50°C, and 60°C indicate spontaneous nature of adsorption for 2,4-DCP. The positive values of ΔS^0 suggest the increased randomness at the solid/solution interface during the adsorption of 2,4-DCP on coir pith carbon.

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

CONCLUSION

Wastewaters containing phenolic compounds present a serious problem. The present study shows that the coir pith carbon is an effective adsorbent for the removal of 2,4-DCP from aqueous solution. Adsorption equilibrium follows moderately both Langmuir and Freundlich isotherms. Kinetics of adsorption follows pseudo-second order model. Studies of pH effect and desorption show that both physisorption and chemisorption are involved in the adsorption process. The efficiency of the carbon is on a par with commercial activated carbon. Complete removal of 2,4-DCP 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 2,4-DCP. Since the raw material, coir pith, is freely available in large quantities in coir industries, the treatment method seems to be economical.

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