Modified coconut fiber used as adsorbent for the removal of 2-chlorophenol and 2, 4, 6-trichlorophenol from aqueous solution

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Keywords: chlorophenol, activated carbon, adsoption isotherm, waste water treatment, water pollution

Abstract-The aim of this work was to determine the potential application of adsorbent prepared from coconut fiber for the removal of organic water pollutants. Removal of 2-chlorophenol (2-CP) and 2, 4, 6-trichlorophenol (TCP) from aqueous solution by coconut fiber activated carbon (FAC) and acid treated coconut fiber activated carbon (ATFAC) was investigated. Equilibrium and kinetic studies were performed and the data was fitted to isotherm and kinetic models. Langmuir isotherm model fitted better than Freundlich to the adsorption data. The monolayer adsorption capacity of ATFAC (38.29 mg g^{-1} for 2-CP and 101 mg g^{-1} for TCP) at 25^0C was relatively higher as compared to that of FAC (37.11 mg g^{-1} for 2-CP and 49.80 mg g^{-1} for TCP). The adsorption trend was as follows: ATFAC (TCP) > ATFAC (2-CP) > FAC (TCP) > FAC (2-CP). The pseudo-second-order rate model fitted better to the adsorption kinetics as compared to the pseudo-first-order rate model in all the cases. Overall adsorption rate is controlled by film diffusion. The thermodynamic parameters (free energy, enthalpy, entropy changes) exhibited the feasibility and spontaneous nature of the adsorption process. The results of the study show that the carbon prepared from acid treated coconut fiber is more effective than thermally treated fiber in the removal of chloro phenols and can be used as a potential adsorbent for the removal of water pollutants.

INTRODUCTION

Chlorophenols have found widespread usage in petroleum refining, plastic, rubbers, pharmaceuticals, disinfectants, wood preserving, steel industries the pulp and paper industry. Due to their toxicity and recalcitrance, they form an important class of environmental pollutants (Colella et al, 1998). Because of their high solubility, they not only contaminate wastewaters and groundwater but also migrate within different aqueous environments (Snoeyink et al, 1997). Previous studies have classified them as one of the most hazardous water pollutants (Gupta et al, 2006; Srivastava et al, 1995; Fawell et al, 1998; Murialdo et al, 2003; Fu et al, 2004). Study for the removal of chlorophenols becomes important due to their carcinogenic and mutagenic nature (Fishbein, 1978) and their high resistance to biodegradation.

Many efforts have been made for the physicochemical and biological treatment of chlorophenol-rich wastewaters. Several studies have been carried out for the removal of chlorophenols from water/wastewater including biodegradation (Baker et al, 1980; Hsieh et al, 2000; Tseng et al, 2003), adsorption (Streat et al, 1995; Okolo et al, 2000; Paprowiez, 1990), ion exchange (Maslowska et al, 1984), ozonation (Stockinger et al, 1995; Adams et al, 1997; Shang et al, 2002) and electrochemical oxidation (Tahar et al, 1999; Korbahti et al, 2002; Yavuz et al, 2006). Many materials like coir pith (Namasivayam et al, 2003), dried activated sludge, fly ash (Aksu et al, 2001) palm seed coat (Rengaraj et al, 2002), bituminous coal (Podkoscielny et al, 2003) and redmud (Namasivayam et al, 1998) have been reported for the removal of chlorophenols. Adsorption utilizing activated carbons prepared from agricultural materials have generated much interest amongst researchers and practitioners of environmental engineering and science, attributed to its vast surface area and great affinity for organics. However at the present time, little information is available concerning the adsorption of chlorophenols onto activated carbons prepared from fibers (Wang et al, 2007; Mohan et al, 2005; Singh et al, 2008).

This work builds upon earlier publications in which removal study of pyridine derivatives and chromium metal were done by adsorbents prepared from coconut fiber (Mohan et al, 2005; Mohan et al, 2006). Continuing our research in this direction, we have used activated carbons prepared from coconut fibers for the adsorption and kinetic studies of 2-chlorophenol (2-CP) and 2, 4, 6-trichlorophenol (TCP) from aqueous solutions.

MATERIALS AND METHODS

Adsorbent development

Activated carbon from coconut fiber was prepared following the procedure reported elsewhere (Mohan et al, 2005; Mohan et al, 2006). In brief, two different types of activated carbons were developed. The acid treated fiber activated carbon (ATFAC) was prepared by treating one part of coconut fibers with two parts (by weight) of commercially available concentrated sulfuric acid (36N) and kept in an oven maintained at 150–165°C for a period of 24 h. The carbonized material was washed well with double distilled water to remove the free acid and dried at 105–110°C for 24 h. Dried coconut fibers were subjected to thermal activation at different temperatures viz., 200, 400, 600 and 800°C for 1 h in an inert atmosphere.

The fiber activated carbon (FAC) was prepared by simply activating the coconut fibers without any chemical treatment at different temperatures (200, 400, 600 and 800°C) in an inert atmosphere. The temperature and time were optimized after observing the surface properties of the activated products obtained. In both cases (with and without chemical treatment) the products obtained at temperatures higher or lower than 600°C exhibited poor adsorption capacities. The activation was carried out under closely controlled conditions to obtain optimum properties. The products so obtained were sieved to the desired particle size of 30–200 microns. Finally, the product was stored in vacuum desiccators until required. The point of zero charge $pH_{(PZC)}$ of FAC and ATFAC was calculated using the method described by Noh et al (1989). This was done by placing various amounts of the carbon in 10 ml solution of 0.1 M NaCl. The sealed conical flasks were placed in a constant temperature (25°C) shaker overnight. The equilibrium pH values of the mixtures were measured. The limiting pH was taken as $pH_{(PZC)}$. The composition of both the carbons with their pH and $pH_{(PZC)}$ are given in Table 1.

Table 1 : Characteristics of activated carbons derived from coconut fibers

Samples	**SBET m^2/g**	**pH**	**pH_{PZC}**	**C (%)**	**N (%)**	**H (%)**	**Ash (%)**
FAC	343	7.80	8.20	71.54	0.32	1.83	7.12
ATFAC	512	5.80	7.50	76.38	0.38	1.95	7.22

Adsorbate preparation

Two chloro-substituted phenols namely 2-chlorophenol (2-CP) and 2, 4, 6-trichlorophenol (TCP) were selected for the study of adsorption on both the activated carbons (FAC and ATFAC). All the reagents were AR-grade chemicals. Stock solutions ($1x10^{-3}$ mol L^{-1}) of 2-CP and TCP were prepared in double distilled water. Subsequent test solutions of the two chlorophenols were prepared through appropriate dilution of the respective stock solutions with double distilled water. The pH of the test solutions were adjusted using dilute HCl (0.1 N) and NaOH (0.1 N). The pH measurements were made using a pH meter (model 744, Metrohm).

Adsorption studies

Adsorption studies were performed by the batch technique to obtain the rate and equilibrium data. In order to select the optimum pH for experiments, a series of batch experiments with the FAC and ATFAC were conducted at different pH ranging 2–10. The equilibrium isotherm studies were conducted by contacting a constant mass of adsorbent with adsorbate solutions of different initial concentrations (10^{-4}-10^{-3} mol L^{-1}) at optimum pH and agitated intermittently. Blank samples without adsorbent were used under the same conditions. Adsorption studies were performed at different temperatures (10°C, 25°C and 40°C) and at optimum pH to obtain data on the rate and extent of adsorption. For isotherm studies, a series of 100 mL Erlenmeyer stopper conical flasks containing 50 mL of adsorbate (2-CP and TCP) solution (optimum pH) of varied concentrations (10^{-4}-10^{-3} mol L^{-1}) and known amount of adsorbents were mixed together and agitated intermittently for a period of 30 h. The contact time and other conditions were selected on the basis of preliminary experiments, which demonstrated that the contact period of 24 h was sufficient to establish the equilibrium (Fig. 1a and 1b). Longer contact times gave practically the same uptake for both the chloro phenols.

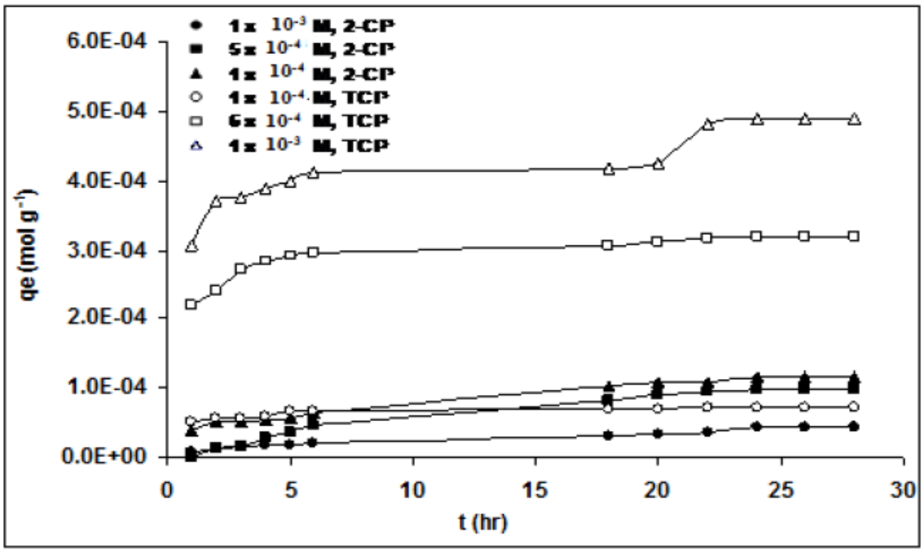

Figure 1a

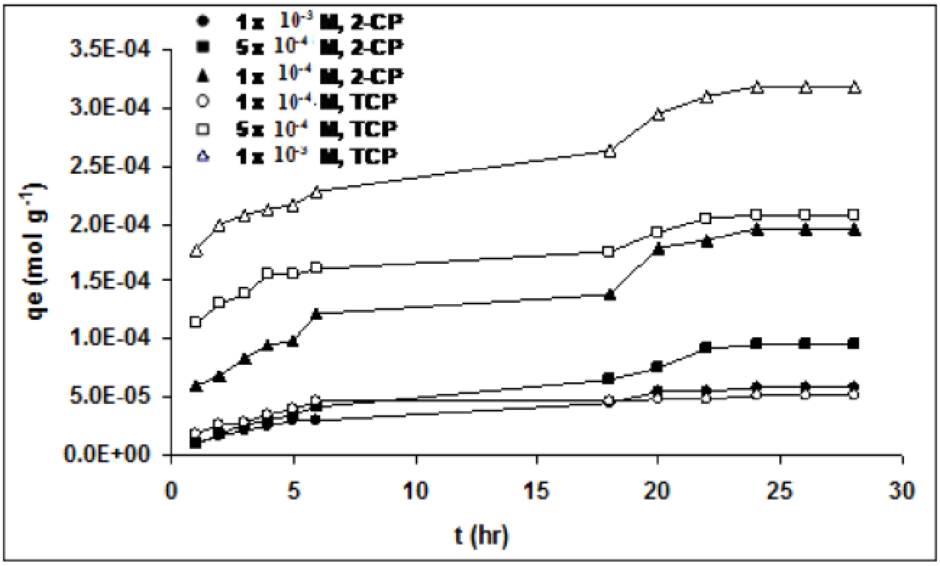

Figure 1b

Figure 1: Effect of contact time on the uptake of 2-CP and TCP on (a) FAC and (b) ATFAC at different initial concentrations; pH = 2; adsorbent amount = 1.0 g L^{-1}; temperature = 25°C

After this period the solution was filtered and adsorbate (2-CP and TCP) concentrations were determined spectrophotometrically at their corresponding λ_{max}. Absorbance measurements were made with a UV–visible spectrophotometer model GBC Cintra 40. The spectrophotometer response time was 0.1 s and the instrument had a resolution of 0.1 nm. Absorbance values were recorded at the wavelength for maximum absorbance i.e. 273 and 294 nm (λ_{max}), corresponding to 2-CP, and TCP, respectively. The absorbance was measured with a 1-cm path-length cell, with an accuracy of ±0.004. Absorbance was found to vary linearly in the concentration range of 10^{-4}-10^{-3} M. Agitation of the system under investigation was carried out on a thermostat-cum-shaking assembly (model MSW 275).

Kinetic studies

The adsorption kinetics of different adsorbates (2-CP and TCP) on the two adsorbents (FAC and ATFAC) was studied by the batch technique. The batch kinetic studies were performed at different temperatures, adsorbate concentrations, and adsorbent doses at optimum pH. For this purpose, a number of stoppered conical flasks containing a definite volume (50 mL in each case) of adsorbate solution of known concentration were placed in a thermostat controlled shaking assembly. When the desired temperature was reached, a known amount of adsorbent was added to each flask and the solutions were agitated mechanically. At pre-decided intervals of time, the solutions of the specified conical flasks were separated from the adsorbent and analyzed spectrophotometrically to determine the uptake of adsorbate at corresponding λ_{max}.

Theoretical background of models applied

Langmuir and Freundlich model

The Langmuir and Freundlich models (Singh et al, 2008) were used to fit the adsorption isotherms and to evaluate the isotherm parameters. The Langmuir isotherm is based upon the assumption of monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform energies of adsorption with no transmigration of adsorbate in the pores of the adsorbent surface. The Langmuir equation may be written as (Singh et al, 2008):

$$\frac{c_e}{q_e} = \frac{1}{Q^o b} + \frac{1}{Q^c} c_e \qquad (1)$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mol g^{-1}), C_e the equilibrium concentration (mol L^{-1}), Q^o the monolayer adsorption capacity (mol g^{-1}) and b is the constant related to the free energy of adsorption ($b\alpha e^{-\Delta G/RT}$). It is the reciprocal of the concentration of which half the saturation of the adsorbent is attained. The model parameters (Q^0 and b) can be determined from the linear plots of C_e/q_e and C_e.

The Freundlich model assumes heterogeneous surface energies, in which adsorption energy varies as a function of the surface coverage due to variation in the heat of adsorption. The Freundlich equation may be written as (Laszlo et al, 2004):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \qquad (2)$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mol g^{-1}), C_e the equilibrium concentration (mol L^{-1}), K_F the constant indicative of the relative adsorption capacity of the adsorbent (mol g^{-1}) and 1/n is the constant, indicative of the intensity of the adsorption. The model parameters (K_F and 1/n) can be determined from the linear plots of log q_e and log C_e.

Kinetic models

To analyze the adsorption rate of phenols onto the developed adsorbents, two kinetic models (pseudo-first-order and pseudo second order) were used. The pseudo-first-order kinetic equation (Tutem et al, 1998) may be written as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.808} t \qquad (3)$$

where q_e and q_t are the amounts adsorbed at equilibrium and at time t, respectively, and k_1 is the first-order rate constant. The adsorption rate parameter k_1 can be calculated by plotting log (q_e -q_t) versus t. The pseudo-second-order-equation based on equilibrium adsorption may be expressed as (Reddad et al, 2002):

$$\frac{1}{q_t} = \frac{1}{k_2 qe^2} + \frac{1}{q_e} t \qquad (4)$$

where q_e is the amount adsorbed at equilibrium, and k_2 is the pseudo-second-order rate constant. The values of k_2 and q_e can be calculated by plotting t/q_t versus t.

RESULTS AND DISCUSSION

Characterization of prepared adsorbents

The pH, pH_{PZC}, C, H, N, and ash content (%) of both the carbons are given in Table 1. The BET surface area of the FAC is 343 $m^2g^{-1}$and for ATFAC it is found to be 512 m^2g^{-1}. Scanning electron microscopic (SEM) photographs are widely used to study the morphological features and surface characteristics of the adsorbent materials. In the present study, scanning electron microscopic photographs of the two activated carbons (30–200 micron), revealed the surface texture, porosity and fibrous structure of the developed adsorbents (Fig. 2a and 2b). SEM results showed that as compared to the thermally treated fibers, in chemically treated fibers there were more small oval cavities, cracks and attached fine particles over the activated carbon surface forming a complicated pore network.

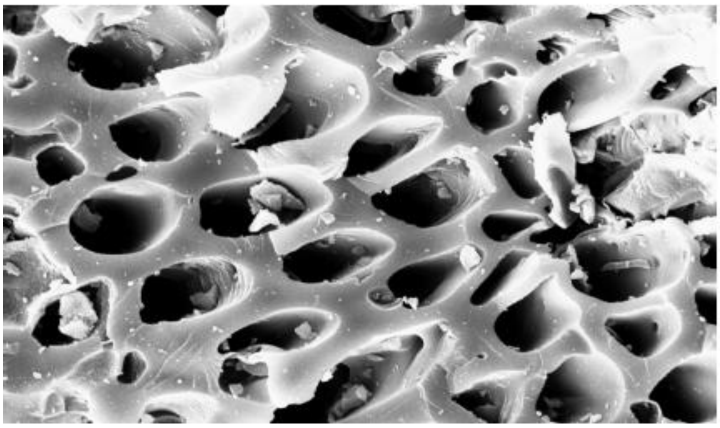

Figure 2a

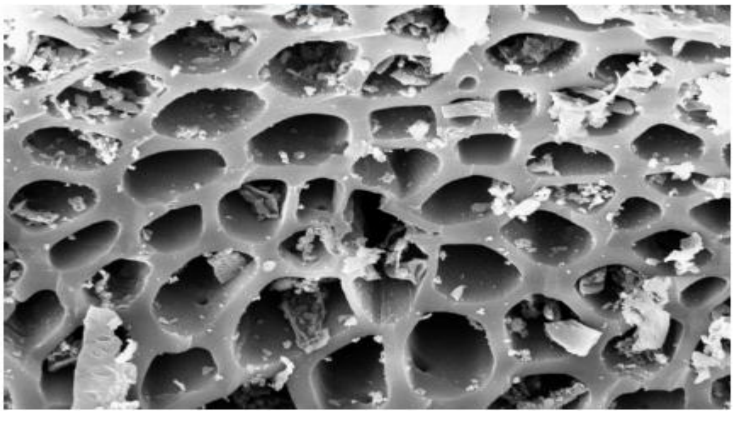

Figure 2b

Figure 2: Scanning electron micrographs of (a) FAC and (b) ATFAC at 1500x

Adsorption studies

The pH of the solution is one of the major factors influencing the adsorption capacity of compounds that can be ionized. Acid or alkali species may change the surface chemistry of the adsorbent by reacting with surface groups. These effects may lead to significant alterations in the adsorption equilibrium depending on the pH (Mohan et al, 2005; Garcia- Araya et al, 2003). At higher pH, phenol dissociate forming phenolate anions, whereas, surface functional groups may be either neutral or negatively charged. The electrostatic repulsion lowers the adsorption capacity of both the chlorophenols at high pH. The ionic fraction of phenolate ion, Ψ_{ions} can be calculated from the equation (Banat et al, 2000):

$$\Psi_{\text{ions}} = \frac{1}{1+10^{(pKa-pH)}} \qquad (5)$$

The Ψ_{ions} increases as the pH value increased. Thus, phenols being a weak acid will be adsorbed to a lesser extent at higher pH values due to the repulsive force prevailing at higher pH (Banat et al, 2000; Khalid et al, 2000). 2-chlorophenol and 2, 4, 6-trichlorophenol is associated with the electron withdrawing effect of the aromatic ring (Laszlo et al, 2004). Adsorption capacity of the activated carbons for the solute in molecular form depends on the electron density of the solute and the carbon surface because the dispersive interaction between the aromatic ring of the solute and those of the carbon surface are the main forces involved in the adsorption process (Nouri et al, 2004). The effect of pH on the removal of different adsorbates (2-CP and TCP) using adsorbents FAC and ATFAC is presented in Fig. 3.

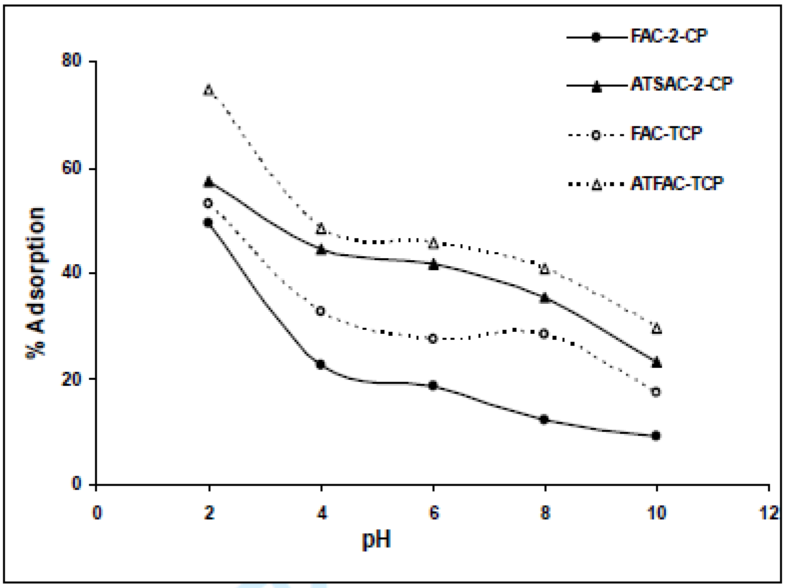

Figure 3: Effect of pH on the adsorption of 2-CP and TCP on FAC and ATFAC

These studies were carried out at the initial adsorbate concentration of 1×10^{-4} mol L^{-1}. It was observed that the removal decreases with an increase in the solution pH. The maximum adsorption was observed at the acidic pH for both the adsorbents, therefore, a pH of 2.0±0.2 was chosen for the adsorption of 2-CP and TCP on the FAC and ATFAC. At low pH high adsorption of phenols are also reported by some researchers (Gupta et al 2006; Singh et al, 2008; Garcia-Araya et al, 2003). At lower pH, the functional groups on the carbon surface are in the protonated form and high electron density on the solute molecules would lead to higher adsorption. The positive surface of the adsorbent in relation to $pH_{(PZC)}$ is also discussed by Boehm (2002). It was concluded that for amphoteric carbons having $pH < pH_{(PZC)}$, the surface of the carbon is considered to be positively charged.

The surface chemistry of the activated carbons also depends on their heteroatom content, mainly on their surface oxygen complex (Radovic et al, 2001). A negative charge will result from the dissociation of surface oxygen complexes of acid character such as carboxyl and phenolic groups and these surface sites are known to be of the Bronsted type. The positive surface charge may be due to surface oxygen complexes of basic character like pyrones or chromenes, or due to the existence of electron-rich regions within the grapheme layers acting as Lewis basic centers, which accept protons from the aqueous solution (Moreno-Castilla, 2004). The rise in the equilibrium concentrations results in the molecular interaction of the solute i.e. electrostatic interactions with the polar surface groups of activated carbons. This effect decreases with increase in the temperature enhancing the adsorption (Terzyk, 2003). The Langmuir isotherms for the adsorption of 2-CP and TCP on FAC and ATFAC at different temperatures are shown in Fig. 4a and 4b, respectively.

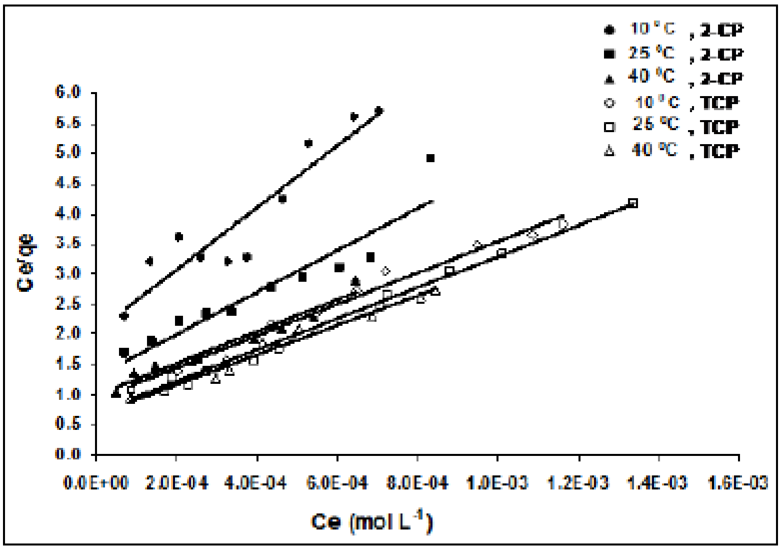

Figure 4a

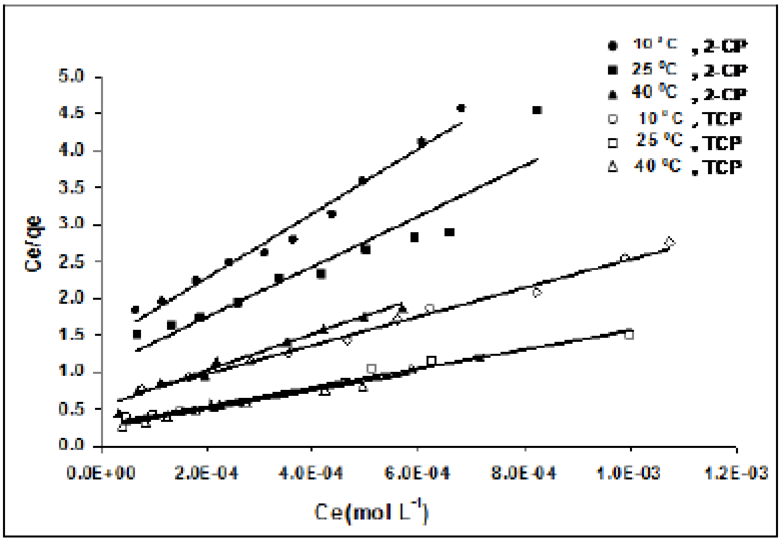

Figure 4b

Figure 4: Langmuir adsorption isotherms of 2-CP and TCP on (a) FAC and (b) ATFAC at different temperatures, pH = 2.

The monolayer adsorption capacity (Q^o) was found to be higher for ATFAC as compared to FAC for adsorption of both 2-CP and TCP as shown in Table 2. This signifies that the large surface area of ATSAC (512 m^2/g) is one of the parameters governing the high adsorption capacity of chloro phenols onto acid treated carbons.

The adsorbent developed and used in the study showed high adsorption capacity in the removal of phenols from aqueous solution as compared to other adsorbents developed by activation methods like palm seed coat (Rengaraj et al, 1999), sawdust (Mohanty et al, 2005), tamarind nutshell (Goud et al, 2005) and tendu leaf refuse (Nagda et al, 2009). Although the adsorption capacity of olive stone activated carbon reported by Mourad et al. (2006) is much higher than the adsorbent investigated in this study. The difference in the adsorption capacities of these adsorbents for the uptake of phenols suggests that the nature and composition of the agricultural waste material used as adsorbent and the activation process under which it was developed plays an important role in determining its adsorption capacity.

Table 2 : Langmuir isotherm constants for the adsorption of chlorophenols

	10 °C			**25 °C**			**40 °C**		
Adsorbent	**Q^o ($x10^4$ mol g^{-1})**	**b ($x10^{-3}$ mol g^{-1})**	**R^2**	**Q^o ($x10^4$ mol g^{-1})**	**b ($x10^{-3}$ mol g^{-1})**	**R^2**	**Q^o ($x10^4$ mol g^{-1})**	**b ($x10^{-3}$ mol g^{-1})**	**R^2**
2-CP									
FAC	1.96	2.49	0.98	2.89	2.63	0.99	3.80	2.68	0.95
ATFAC	2.27	3.16	0.98	2.98	3.11	0.98	4.05	4.65	0.99
TCP									
FAC	3.82	2.83	0.98	3.87	3.66	0.97	4.03	3.74	0.99
ATFAC	5.14	3.24	0.99	7.86	4.34	0.98	8.08	4.80	0.97

A donor-acceptor complex mechanism also reported earlier (Mattson et al, 1969) for the adsorption of phenol is one of the key factors in the explanation of adsorption of 2-CP and TCP on activated carbons. In this mechanism, the carbonyl oxygen group on the carbon surface acts as the electron donor whereas the aromatic ring of the phenol acts as the acceptor. The above donor-acceptor complex mechanism is more pronounced in the case of 2, 4, 6-trichlorophenol as the presence of the three Cl-group is in favor of the formation of a bond between the surface carbonyl groups and the electron–deficient aromatic ring of the phenol while only one Cl-group in 2-CP makes the aromatic ring of the phenol less electron deficient and thus weaken the bond formation between surface carbonyl groups of the adsorbent and aromatic ring of the phenol.

The Langmuir constant *b* reflects the affinity of the adsorbent for the solute. For the adsorption of 2-CP and TCP the values of *b* are relatively higher for ATFAC indicating more stable bond/complex with the carbon surface. The essential characteristic of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium factor R_L that is defined as $R_L = 1/(1+bC_o)$, where b is the Langmuir constant and C_0 the initial concentration of adsorbate. R_L values obtained (data not shown) at different concentrations and temperatures are between 0 and 1, indicating favorable adsorption of both adsorbates on activated carbons developed from the agricultural waste material. The Freundlich isotherms for the adsorption of 2-CP and TCP on FAC and ATFAC at different temperatures are presented in Fig. 5a and 5b, respectively. The corresponding Freundlich isotherm parameters along with the correlation coefficients are given in Table 3. The value of $0 < 1/n < 1.0$ exhibits the favorability of adsorption onto activated carbons (McKay et al, 1981). The regression coefficients showed that the Langmuir model adequately fitted to the adsorption data. The adsorption capacity of ATFAC for TCP is higher than that for 2-CP i.e. TCP > 2-CP. This could be explained by the electron withdrawing characteristic of the Cl-group also taken into account by Jain et al (2007); according to the study the greater the number of Cl-groups present, the more deficient the aromatic ring and this would result in higher adsorption of phenol onto carbon.

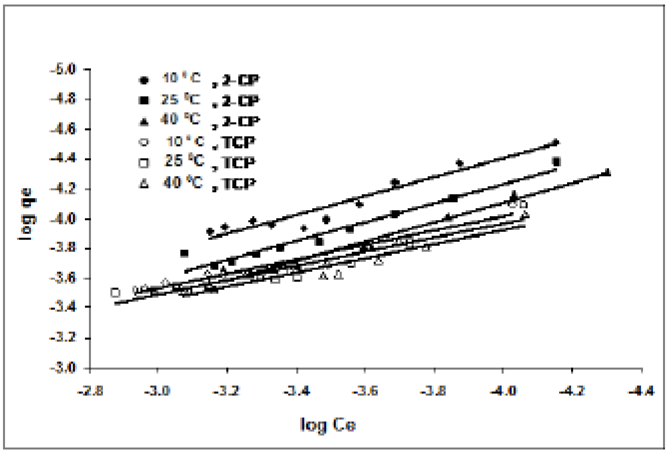

Figure 5a

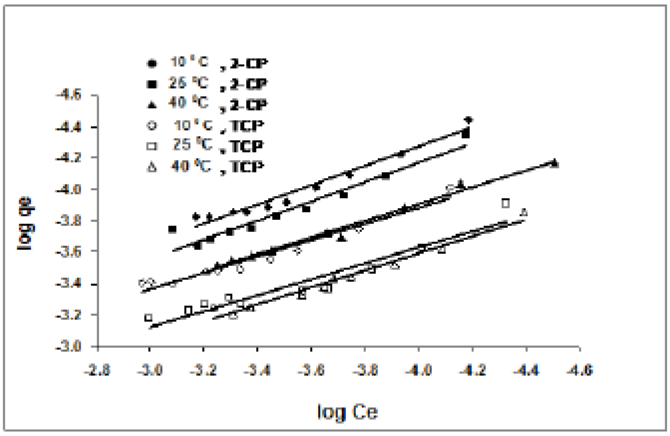

Figure 5b

Figure 5: Freundlich adsorption isotherms of 2-CP and TCP on (a) FAC and (b) ATFAC at different temperatures, pH = 2.

	10 °C			**25 °C**			**40 °C**		
Adsorbent	**K_F (mol g^{-1})**	**1/n**	**R^2**	**K_F (mol g^{-1})**	**1/n**	**R^2**	**K_F (mol g^{-1})**	**1/n**	**R^2**
2-CP									
FAC	0.014	0.64	0.93	0.021	0.64	0.95	0.030	0.66	0.98
ATFAC	0.015	0.61	0.96	0.018	0.62	0.95	0.021	0.54	0.99
TCP									
FAC	0.008	0.49	0.94	0.009	0.48	0.90	0.010	0.48	0.92
ATFAC	0.016	0.54	0.95	0.023	0.52	0.91	0.037	0.51	0.97

Thermodynamic studies

The thermodynamic parameters mainly free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) changes that mainly characterize the equilibrium of the system were calculated using equations (6) – (8), respectively. The reference state was defined based on adsorption density in mol g^{-1} of adsorbent and concentration in mol L^{-1} (Singh et al, 2008):

$$\Delta G = -RT \, lnK \qquad (6)$$

$$\Delta H = R\left(\frac{T_1 T_2}{T_1 - T_2}\right) ln\frac{K_2}{K_1} \qquad (7)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \qquad (8)$$

where K, K_1 and K_2 are the equilibrium constants at the temperatures 10 °C, 25 °C and 40 °C respectively and obtained from the slopes of adsorption isotherms at different concentrations. The values obtained from the thermodynamic analysis are given in Table 4.

Table 4: Thermodynamic parameters of the adsorption

Adsorbent	$-\Delta G^0$ (kJ mol^{-1}) 10 °C	25 °C	40 °C	ΔH (kJ mol^{-1})	ΔS^0 (kJ mol^{-1})
2-CP					
FAC	18.40	19.51	20.54	1.86	0.07
ATFAC	18.96	19.99	21.98	9.36	0.10
TCP					
FAC	18.70	20.33	21.41	6.96	0.09
ATFAC	19.02	20.75	22.06	9.75	0.12

The negative values of ΔG for adsorption of 2-CP and TCP indicate the feasibility and spontaneous nature of the process. The dipole-dipole interaction between the adsorbent and adsorbate may effectively reduce the hydration number of the adsorbate and adsorbent, increasing ΔS, as water molecules are released into the aqueous solution (Vinod et al, 2002). Physical adsorption and chemisorption can be classified, to a certain extent, by the magnitude of the enthalpy change. It is accepted that bonding strengths of < 84 kJ mol^{-1} are those of physical adsorption type bonds. The chemisorption bond strengths range from 84 kJ mol^{-1} to 420 kJ mol^{-1} (Terzyk, 2003). Based on this assumption the adsorption of chloro phenols on activated carbons (FAC and ATFAC) appear to be a physical adsorption process.

Kinetic studies

The extent of adsorption of both the adsorbates on FAC and ATFAC was found to increase with temperature indicating the endothermic nature of the process resembling the findings of the thermodynamic analysis. The effect of adsorbent amount and initial adsorbate concentration on the adsorption of chloro phenols for the different carbons has also been studied. The rate of uptake increased with an increase in adsorbent amount. The adsorption rate data for the studied adsorbates (2-CP and TCP) attaching onto the developed activated carbons were analyzed using two kinetic models viz., pseudo-first-order equation and pseudo-second-order equation. Both the models were studied at different temperatures as well as different initial concentrations of adsorbates to find out the effect of temperature and concentration on the rate-equation parameters. The results obtained from both the first and second-order rate equations at different temperatures are given in Tables 5 and 6 for different adsorbate concentrations; it is summarized in Tables 7 and 8, respectively. The pseudosecond- order rate model fitted better to the adsorption kinetics as compared to the pseudofirst- order rate model. The qe values obtained by using the pseudo-second order rate equation are more accurate to the experimental values (Tables 9 and 10) than that when using the pseudo-first order at all temperatures and concentrations for both the chloro phenols.

Table 5 : First-order rate constants for the adsorption at different temperature

	10 °C		**25 °C**		**40 °C**	
Adsorbent	**k_1 (x10^3 min^{-1})**	**R^2**	**k_1 (x10^3 min^{-1})**	**R^2**	**k_1 (x10^3 min^{-1})**	**R^2**
2-CP						
FAC	2.17	0.95	0.97	0.98	2.48	0.96
ATFAC	3.12	0.98	1.97	0.92	2.45	0.89
TCP						
FAC	0.22	0.90	2.60	0.77	2.22	0.86
ATFAC	1.90	0.89	1.67	0.82	1.16	0.84

Table 6 : Second-order rate constants for the adsorption at different temperature

	10 °C		**25 °C**		**40 °C**	
Adsorbent	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**
2-CP						
FAC	52.83	0.94	34.83	0.99	21.00	0.99
ATFAC	21.00	0.87	32.50	0.95	27.50	0.96
TCP						
FAC	296	0.98	368	0.99	308	0.99
ATFAC	166	0.98	149	0.99	81.67	0.99

Table 7 : First-order rate constants for the adsorption at different initial adsorbate concentrations

	1x10^{-4} mol L^{-1})		**(5x10^{-4} mol L^{-1})**		**(1x10^{-3} mol L^{-1})**	
Adsorbent	**k_1 (x10^3 min^{-1})**	**R^2**	**k_1 (x10^3 min^{-1})**	**R^2**	**k_1 (x10^3 min^{-1})**	**R^2**
2-CP						
FAC	0.97	0.98	1.82	0.96	2.03	0.99
ATFAC	1.97	0.92	1.97	0.73	1.35	0.88
TCP						
FAC	2.60	0.77	2.15	0.91	1.29	0.59
ATFAC	1.67	0.82	1.28	0.87	1.22	0.90

Table 8 : Second- order rate constants for the adsorption at different initial adsorbate concentrations

	1x10^{-4} mol L^{-1})		**(5x10^{-4} mol L^{-1})**		**(1x10^{-3} mol L $^{-1}$)**	
Adsorbent	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**	**k_2(g mol^{-1}min $^{-1}$)**	**R^2**
2-CP						
FAC	52.83	0.94	36.83	0.99	25.83	0.98
ATFAC	32.50	0.98	11.42	0.94	15.82	0.94
TCP						
FAC	368	0.99	87.67	0.99	39.33	0.98
ATFAC	149	0.99	47.50	0.99	25.17	0.98

Table 9 : Comparative evaluation of q_e as calculated experimentally and by using first and second-order rate equations at different temperatures

	$q_{e(exp)}$ (x10^4 mol g^{-1})			**$q_{e(exp)}$ (x10^4 mol g^{-1})**			**$q_{e(exp)}$ (x10^4 mol g^{-1})**		
Adsorbent	**10 °C**	**25 °C**	**40 °C**	**10 °C**	**25 °C**	**40 °C**	**10 °C**	**25 °C**	**40 °C**
2-CP									
FAC	0.31	0.45	0.52	0.48	0.74	1.39	0.36	0.42	0.54
ATFAC	0.58	0.95	2.05	0.74	1.24	02.19	0.55	0.98	2.07
TCP									
FAC	0.38	0.72	0.72	0.49	0.18	0.19	0.37	0.73	0.73
ATFAC	0.29	0.52	0.64	0.19	0.25	0.08	0.31	0.54	0.64

Table 10 : Comparative evaluation of q_e as calculated experimentally and by using first and second-order rate equations at initial adsorbate concentrations

	$q_{e(exp)}$ (x10^4 mol g^{-1})			**$q_{e(exp)}$ (x10^4 mol g^{-1})**			**$q_{e(exp)}$ (x10^4 mol g^{-1})**		
Adsorbent	**(1x10^{-4} mol L^{-1})**	**(5x10^{-4} mol L^{-1})**	**(1x10^{-3} mol L^{-1})**	**(1x10^{-4} mol L^{-1})**	**(5x10^{-4} mol L^{-1})**	**(1x10^{-3} mol L^{-1})**	**(1x10^{-4} mol L^{-1})**	**(5x10^{-4} mol L^{-1})**	**(1x10^{-3} mol L^{-1})**
2-CP									
FAC	0.45	0.59	1.16	0.36	0.54	0.95	0.48	0.59	1.14
ATFAC	0.58	0.95	2.05	0.65	1.02	1.56	0.59	0.94	2.09
TCP									
FAC	0.71	3.19	4.90	0.18	0.75	1.51	0.73	3.21	4.89
ATFAC	0.52	2.17	3.32	0.25	0.97	1.63	0.54	2.15	3.31

Mass transfer studies

The mass-transfer analysis of adsorbates during the process was studied by using the mass transfer diffusion model (McKay et al, 1981):

$$\ln\left(\frac{C_t}{C_o} - \frac{1}{1+mk}\right) = \ln\left(\frac{mk}{1+mk}\right) - \left(1+\frac{mk}{mk}\right) \beta_l S_s t \qquad (9)$$

where C_t is the concentration of solute at time t (mol L^{-1}), C_0 the initial concentration of the solute (mol L^{-1}), m the mass of the adsorbent per unit volume of particle-free solution of solute (g L^{-1}), k the Langmuir constant (obtained by multiplying Q^0 with b), β_l the mass transfer coefficient (cm s^{-1}) and Ss the outer surface area of the adsorbent per unit volume of particle-free slurry (cm^{-1}) and is calculated as:

$$S_s = \frac{6m}{(1-\tau_p)} d_p \rho_p \qquad (10)$$

where d_p is the particle diameter (cm), ρ_p the density of adsorbent (g cm^{-1}) and ε_p is the porosity of adsorbent particles. The values of β_l were determined from the slope and intercepts of the plots of ln{(Ct/C_0)-1/(1+mk)} versus t, for different temperatures and initial adsorbate concentrations (Fig. 6 and 7 respectively) using the least squares method. The linearity of the plots confirms the validity of the diffusion model for the studied adsorbateadsorbent systems. The values of the mass transfer coefficient (β_l) of the adsorbates for both the developed adsorbents (FAC and ATSAC) are presented in Tables 11 and 12. The values of β_l increased with an increase in the temperature, suggesting the endothermic nature of the reaction. Further, it was found that increasing the initial adsorbate concentration results in a decrease in the external mass transfer coefficient. These findings are similar to those reported for the adsorption of phenol by coconut-husk based activated carbon (Reichenberg, 1953).

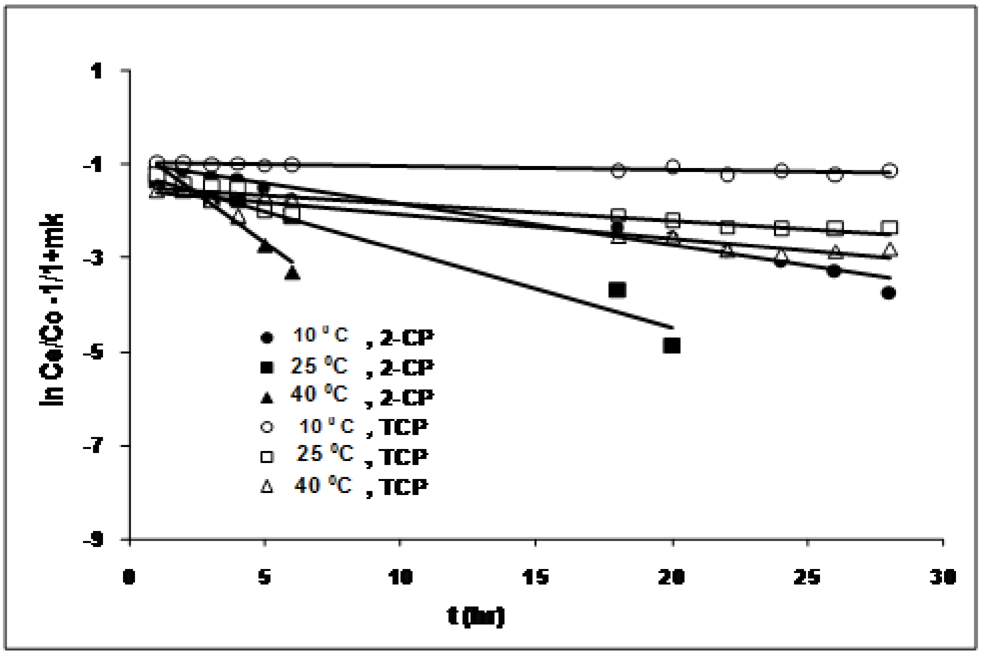

Figure 6a

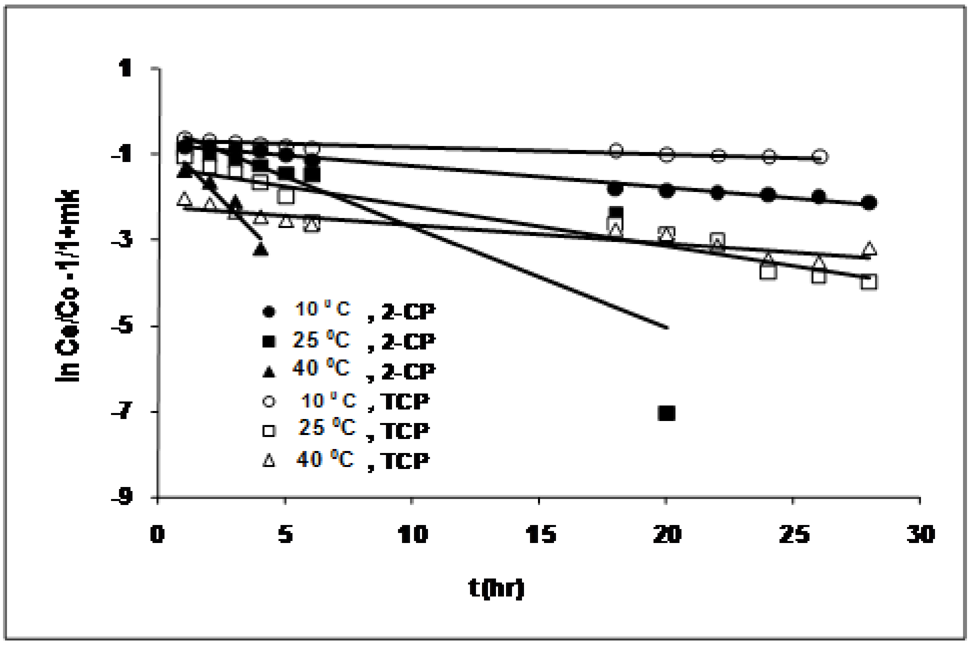

Figure 6b

Figure 6: Mckay plots for the adsorption of 2-CP and TCP on (a) FAC and (b) ATFAC at different temperatures (pH = 2, C_o = 1x 10^{-4} M)

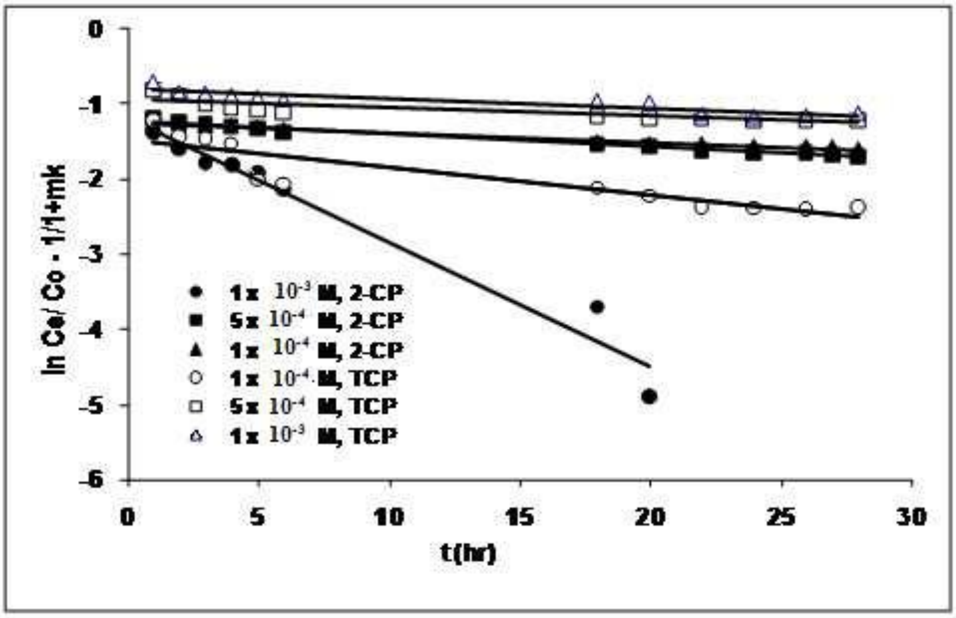

Figure 7a

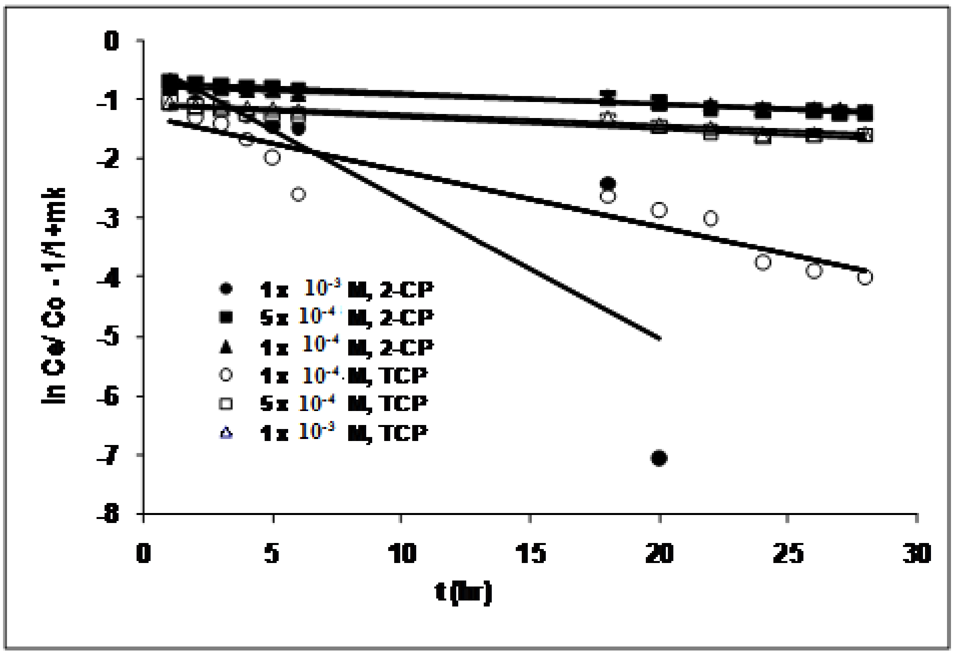

Figure 7b

Figure 7: Mckay plots for the adsorption of 2-CP and TCP on (a) FAC and (b) ATFAC at different initial concentrations (pH = 2, temperature = 25°C)

Table 11 : Mass transfer coefficient (β_l) at different temperatures

	10 °C		**25 °°C**		**40 °C**	
Adsorbent	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**
2-CP						
FAC	2.10	0.98	3.75	0.96	10.86	0.94
ATFAC	1.75	0.99	8.56	0.70	15.58	0.91
TCP						
FAC	0.10	0.77	2.00	0.80	2.69	0.93
ATFAC	0.66	0.92	3.33	0.90	8.86	0.86

Table 12 : Mass transfer coefficient (β_l) at different initial adsorbate concentrations

	($1x10^{-4}$mol L^{-1})		**($5x10^{-4}$mol L^{-1})**		**($1x10^{-3}$mol L^{-1})**	
Adsorbent	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**	**β_l (x 10^{-8}cm sec^{-1})**	**R^2**
2-CP						
FAC	3.77	0.96	3.72	0.98	2.86	0.99
ATFAC	8.56	0.80	6.83	0.98	5.44	0.96
TCP						
FAC	6.14	0.80	2.00	0.76	0.67	0.83
ATFAC	3.33	0.90	3.69	0.93	6.83	0.80

Rate limiting step in the process of uptake of chlorophenols

To interpret the experimental data, it is necessary to identify the rate-determining step controlling the removal rate in the adsorption process. The three consecutive steps involved in the adsorption of an organic/inorganic species by a porous adsorbent are:

(1) transport of the adsorbate to the external surface of the adsorbent (film-diffusion);

(2) transport of the adsorbate within the pores of the adsorbent except for a small amount of adsorption that occurs on the external surface (particle diffusion);

(3) adsorption of the adsorbate on the external surface of the adsorbent.

It is generally accepted that process (3) is very rapid and does not represent the ratedetermining step in the uptake of organic/inorganic species. Therefore, either steps (1) or (2) could be the rate controlling step and to determine the actual rate controlling step involved in the adsorption of chloro phenols, the quantitative analysis of the kinetic data was done with the help of the following equations also studied by Hasany et al (2000):

$$F = 1- \frac{6}{\Pi^2}\sum\frac{1}{n^2}\exp[-\frac{D_i n^2 n^2}{r_o^2}] \qquad (11)$$

or

$$F = 1- \frac{6}{\Pi^2}\sum\frac{1}{n^2}\exp[-n^2 B_t] \qquad (12)$$

where F is the fractional attainment of equilibrium at time t and is obtained by the expression:

$$F = \frac{Q_t}{Q^0} \qquad (13)$$

Q_t is the amount of adsorbate taken up at time t and Q_0 is the maximum equilibrium uptake

and;

B_t values were obtained for each observed value of F and using the following equation:

$$B_t = 2\Pi - \Pi^2 \frac{F(t)}{8} - 2\Pi \left[1 - \left(\frac{\Pi}{8}\right) F(t)\right]^{1/2} \qquad (14)$$

The results are plotted in Fig. 8 (a and b) and Fig. 9 (a and b).

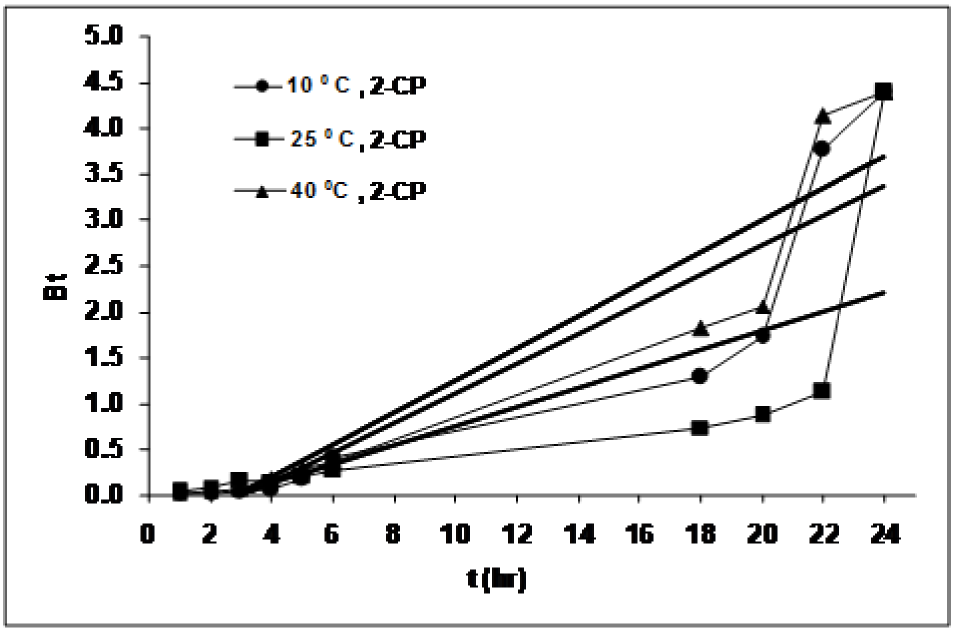

Figure 8a

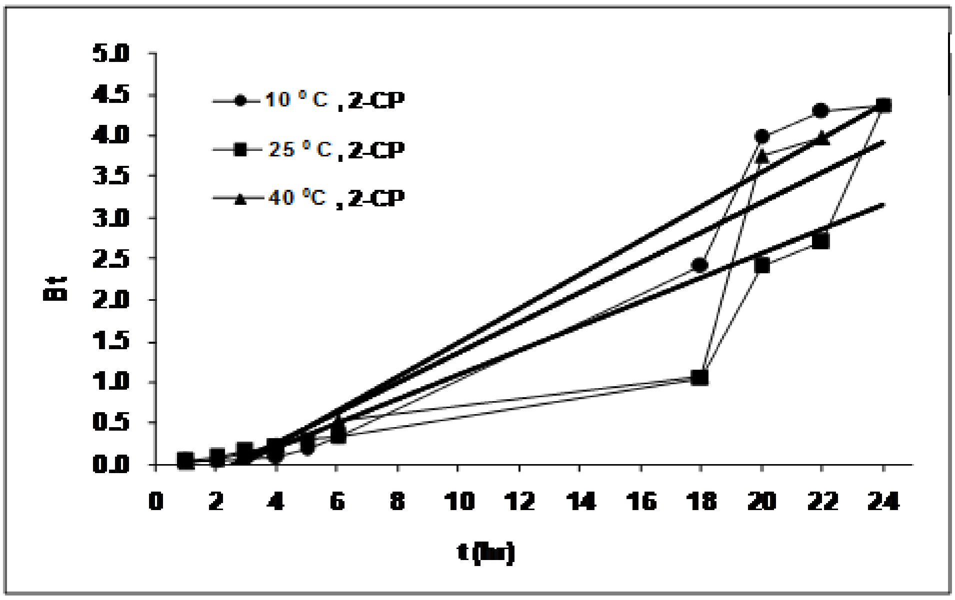

Figure 8b

Figure 8: B_t vs. *t* plots for the adsorption of (a) 2-CP and (b) TCP on FAC at different temperatures (pH = 2; $C_o = 1\text{x}\ 10^{-4}$ mol L^{-1})

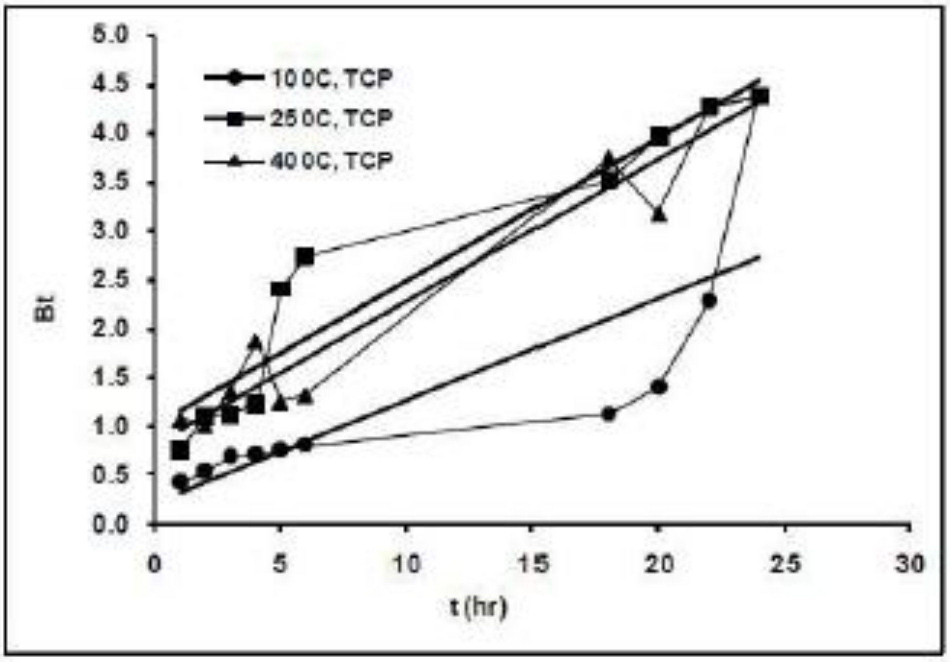

Figure 9a

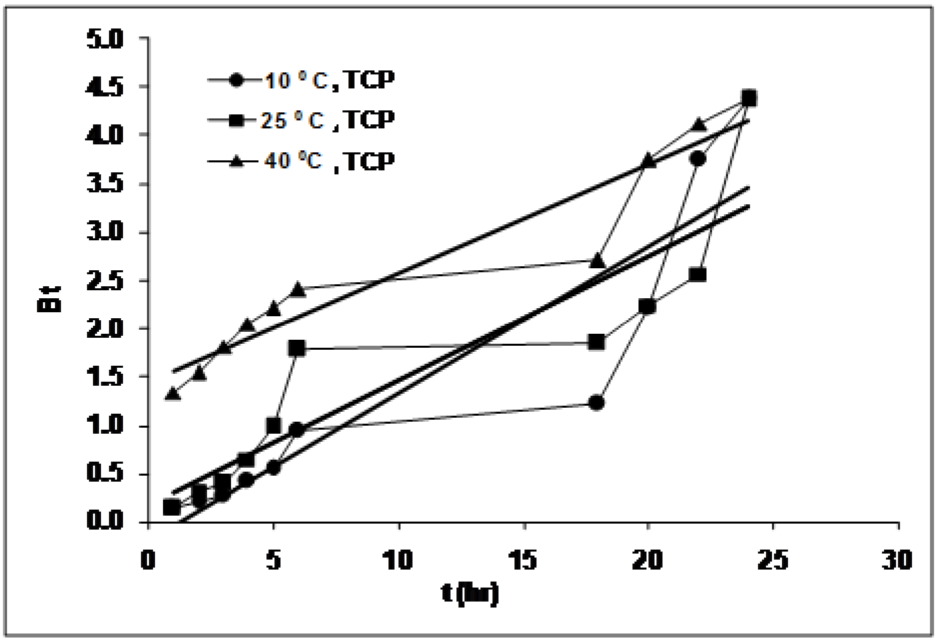

Figure 9b

Figure 9: B_t vs. t plots for the adsorption of (a) 2-CP and (b) TCP on ATFAC at different temperatures (pH = 2; C_o = 1x 10^{-4} mol L^{-1})

The linearity test of Bt versus t plots was employed to distinguish between the film diffusion and particle diffusion controlled adsorption. If the plots of Bt versus t is a straight line passing through the origin, then the adsorption rate is governed by particle diffusion, otherwise it is governed by film diffusion. In case of FAC and ATFAC the Bt versus t plot for adsorption of 2-CP and TCP, at all temperatures at initial concentration (1×10^{-4} mol L^{-1}) do not pass through the origin, suggesting that the rate controlling process may be the film diffusion (Singh et al, 2008). In all the cases the Bt versus t plots (curved at later stage) can be resolved into two plots with different slopes, indicating change in the adsorption mechanism with time. It has also been suggested that the change in the slope indicates the existence of different sizes of pores (Al-Asheh et al, 2003). Similar types of observations have been reported for adsorption of phenol (Vinod et al, 2002) and metals (Gupta et al, 2002).

CONCLUSIONS

Activated carbons developed from the coconut fibers were studied for the adsorption of 2-CP and TCP from aqueous solutions under different conditions. Langmuir isotherm model adequately fitted to the adsorption data. The monolayer adsorption capacity of ATFAC was high as compared to FAC. The pseudo-second-order rate model fitted better to the adsorption kinetics as compared to the pseudo-first-order rate model. Kinetic studies revealed that the adsorption occurs through film diffusion at different temperatures in both thermally activated (FAC) as well as chemically activated carbon (ATFAC). The computed values of Gibbs free energy, enthalpy change and entropy change showed an endothermic nature and spontaneity of the process. The study showed that the adsorbent prepared from acid treatment of the coconut fiber is more efficient in removing chloro phenols from aqueous solutions as compared to thermally activated fiber and can be exploited for commercial purposes. A novel method for maximum regeneration of adsorbents is a part of this work and shall be included in further studies.

ACKNOWLEDGEMENTS

The authors would like to thank the director, Indian Institute of Toxicology Research, Lucknow (India) for his keen interest in the work. Financial assistance from DUT, Durban (South Africa) to the author (PO) is thankfully acknowledged.

NOMENCLATURE

ATFAC	acid treated coconut fiber activated carbon
C_e	equilibrium concentration (mol L^{-1})
C_0	initial concentration of adsorbate (mol L^{-1})
C_t	concentration of solute at time t (mol L^{-1})
2-CP	2-chlorophenol
F	fractional attainment of equilibrium at time *t*
FAC	coconut fiber activated carbon
ΔG	Gibbs free energy
ΔH	Enthalpy
KF	constant indicative of the relative adsorption capacity of the adsorbent (mol g^{-1})
Q_o	monolayer adsorption capacity (mol g^{-1})
R	Universal gas constant
R_L	dimensionless equilibrium factor
SEM	Scanning electron micrograph
S_s	outer surface of the adsorbent per unit volume of particle-free slurry (cm^{-1})
T	Temperature (°C)
TCP	2,4,6-trichlorophenol
b	Langmuir constant
d_p	particle diameter (cm)
m	mass of the adsorbent per unit volume of particle-free solution of solute (g L^{-1})
k_1	first-order rate constant
k_2	pseudo-second-order rate constant
$pH_{(PZC)}$	point of zero charge
q_e	solute adsorbed per unit weight of adsorbent at equilibrium (mol g^{-1})
q_e	solute absorbed per unit weight of absorbent at time *t* (mol g^{-1})
1/n	constant, indicative of the intensity of the adsorption
Ψ_{ions}	ionic fraction of phenolate
β_l	mass transfer coefficient (cm s^{-1})
ρ_p	density of adsorbent (g cm^{-1})
ε_p	porosity of adsorbent particles
λ_{max}	wavelength for maximum absorbance (nm)

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