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Equilibrium, kinetic and thermodynamic study for the efficient removal of pentachloronitrobenzene from aqueous solution by an activated carbon derived from *Pinus ponderosa*

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Activated carbon has been synthesised from *Pinus Ponderosa* shell and used for adsorption of pentachloronitrobenzene. Initial pesticide concentration, initial pH of the solution and phases contact time are the investigated parameters. The equilibrium is attained in 20 and 30 min for diluted specimens (1 and 3.5 mg/L) and 75 min for concentrated specimens (6.5 and 9 mg/L). Linear, Langmuir and Freundlich isotherm models are applied to the experimental data. The maximum monolayer adsorption capacity is found at approximately 350 mg/g from the Langmuir model. This value of the novel carbonized adsorbent can be compared to the adsorption capacities of other materials. The kinetics of adsorption is analyzed via pseudo-first and second-order models. High Regression coefficients and the closeness of the calculated and experimental capacity values are found for pseudo first order model kinetic. The Gibbs energy shows the feasible and spontaneous adsorption. The activation energy and surface coverage relationship are evaluated by sticking probability factor. The results show that the pesticide was removed by the physical type adsorption mechanism and also good binding characteristic on the activated adsorbent.

Keywords: Active carbon, Isotherms, Kinetic, Pesticide removal, Thermodynamic parameter

Introduction

Pesticide usage covers an important role in modern agriculture. Agrochemicals are used to take the control of pests such as insects, plant diseases, worms and rodents. When pesticides are released into the environment, they may be broken down into other molecule groups or they may resist degradation and remain in the environment without decomposition for long periods[1].

Some adsorbents like graphite materials or activated carbons are popular for treating waste effluents[2,3,4]. Activated carbon is commonly employed in waste residues due to its huge adsorption capacity. A highly wide range of chemicals and harmful pollutants like dyes, organic molecules, pesticides and heavy metals are retained on the carbon surface easily[5,6]. Molecules that are slightly polar and molecules which have high molecular weight are well adsorbed on them. On the other hand, the hardest to retain on it, are the most polar molecules and the linear ones that have a low molecular weight like aliphatic alcohols and primary organic acids[7]. Solid-liquid adsorption treatments are very attractive for waste effluents due to their availability and they can further be increased by using sorbents instead of expensive activated carbon. To this scope, many materials having high carbon constituents have been recently studied deriving from either available bioresources or industrial wastes[8,9]. This paper aims to remove pentachloronitrobenzene (PCNB) from aqueous solutions by evaluating the carbonized biomass shells as a cost-free and environmentally friendly adsorbent material.

Experimental Section

Reagents and chemicals

PCNB (98%) was supplied from the Agriculture Provincial Directorate of Foresty in Istanbul (Turkey). *Pinus Ponderosa* was supplied from the Muğla area which is a city in the Aegean region of Turkey and carbonized under the nitrogen stream in the vertical reactor. The powdered *Pinus ponderosa* shell was placed in a stainless tubular reactor with 220 mm length and 75 mm internal diameter. The shell was heated up to the carbonization temperature of 900°C at a heating rate of 15°C/min for 60 min. The temperature was adjusted by using a Ni-Cr-Ni thermocouple. After being carbonized, the samples were allowed to cool down to 25°C under nitrogen

flow. The samples were then milled and sieved through 250 mesh. The carbonized biomass has no negative effect from the point of ecological approach on the environment and is also a beneficial material for employing the undesired constituent removal from the waste effluent. The surface area of the adsorbent was measured using the BET nitrogen adsorption technique by using Micromeritics Flow Sorb II-2300. The scanning electron microscope (SEM) was used to determine its surface morphology via Jeol JSM 5410LV Scanning Microscope. Fourier transform infrared spectroscopy (FTIR) was drawn from 4000 to 400 cm^{-1} by the Unicam Mattson model. The absorbance of PCNB was measured via UV visible double beam spectrometer (UV-150-02). The pH of the solution was recorded by WTW series inolab pH meter.

Adsorption equilibrium

Preliminary kinetic tests were carried out to determine the equilibrium time and adsorption capacity of the sorbent. It was assumed that the equilibrium state was attained when further changes in PCNB concentration were not observed after 75 min contact time.

Adsorption isotherms were obtained by mixing 0.1 g carbonized biomass with 100 mL having various PCNB concentrations. The experiments were done at some determined temperatures. Liquid samples were taken in predetermined intervals until the equilibrium state was achieved.

Adsorption kinetics

Batch adsorption kinetics was conducted and investigated the effects of various parameters such as the initial concentration of PCNB, medium temperature and pH. To explore the effect of initial PCNB concentrations, 1, 3.5, 6.5, and 9.5 mg pesticide concentrations were chosen. To determine the temperature effect, the adsorption was conducted at 20, 27, 34 and 41°C temperatures. The pH effect was searched at 2-8 pH ranges. A liquid volume of 100 mL pesticide containing aquatic solution in 150 mL sealed conical flasks was used for all experiments and the suspensions were agitated by a mechanical NÜVE brand shaker at 150 rev/min. The sample was centrifuged for 5 min at 1000 rpm and absorbance was determined at 268 nm wavelengths. Samples were withdrawn at regular intervals to analyze the residual PCNB concentration. Blank experimental tests were conducted simultaneously under the same conditions which contained the carbonized adsorbent in 100 mL aqueous solution. The stability of the PCNB solution was verified by agitating the PCNB solution without any adsorbent. There was no change in the concentration of PCNB in that control solution during the experiment.

The equation of PCNB removal efficiency (A%) can be calculated as

$$A\% = \frac{(C_0 - C_e)}{C_0} \times 100 \quad \ldots (1)$$

The adsorption capacity of PCNB (q_e)[10,11] can be determined as

$$q_e = \frac{(C_0 - C_e)\, V}{m} \quad \ldots (2)$$

Where, q_e (mg/g) is the equilibrium adsorption capacity; C_0 and C_e (mg/L) are the initial and equilibrium concentrations of PCNB in solution, respectively. V(L) is the volume of the aqueous PCNB containing solution and m (g) is the weight of the adsorbent. The experiments were performed in triplicate and average values were taken into account. The relative deviations between these results were less than 5%.

Adsorption kinetic theory

Adsorption rate constants for the pesticide were calculated by applying pseudo-first-order and pseudo-second-order models. The conformity between the experimental data and the model predicted values was expressed by the correlation coefficients (R^2) and closeness of experimental and calculated capacity values.

Pseudo first order and pseudo-second-order model

The nonlinear pseudo-first-order equation is shown below[12]

$$q_t = q_e\left[1 - \exp(-k_1 t)\right] \quad \ldots (3)$$

The equation may be expressed in a linear form as

$$\log(q_e - q_t) = \log q_e - k_1.t/2.303 \quad \ldots (4)$$

Where q_t (mg/g) is the amount of PCNB adsorbed at any time, t (min), q_e is mentioned above, and k_1 is the pseudo-first-order rate constant (1/min).

The nonlinear pseudo-second-order model is given by Ho and Mc Kay[13]

$$q_t = \frac{tk_2 q_e^2}{1 + tk_2 q_e} \quad \text{... (5)}$$

Which can be rewritten in linear form as

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad \text{... (6)}$$

Where k_2 is the pseudo-second-order rate constant (g/mg.min)

Adsorption isotherms

Three important isotherms were selected to describe the adsorption phenomenon. Those are:

The linear isotherm[14], the Langmuir isotherm [15] and the Freundlich isotherm[16] .

Linear Isotherm

Linear isotherm is expressed as

$$q_e = k_{Li} C_e \quad \text{... (7)}$$

Langmuir isotherm

The Langmuir theory is valid for monolayer adsorption onto a surface containing a finite number of identical sites and is one of the most popular isotherm models due to its suitability and its good agreement with experimental data. It is expressed as

$$q_e = \frac{Q_m k_L C_e}{1 + k_L C_e} \quad \text{... (8)}$$

The linearized form is

$$\frac{C_e}{q_e} = \left(\frac{1}{Q_m . k_L}\right) + \left(\frac{1}{Q_m}\right) C_e \quad \text{... (9)}$$

Freundlich isotherm

The Freundlich isotherm model is an empirical equation and the model is valid for heterogeneous surfaces. It assumes that the adsorption occurs on heterogeneous surfaces and the adsorption capacity is related to the concentration of pesticide at equilibrium. The Freundlich model is generally represented as follows:

$$q_e = k_F C_e^{1/n} \quad \text{... (10)}$$

The linearized form is

$$\log q_e = \log k_F + \left(\frac{1}{n}\right) \log C_e \quad \text{... (11)}$$

k_{Li}, k_L and k_F (l/mg) are the constants related to linear, Langmuir and Freundlich isotherms, respectively. Q_m(mg/g) is the maximum monolayer adsorption capacity, 1/n is the slope in Freundlich isotherm, ranging between 0 and 1, indicative of the degree of non-linearity between solution concentration and adsorption, b is a constant. q_e and C_e are defined in previous equations.

Thermodynamic parameters

The thermodynamic parameters of the adsorption were obtained from experiments at various temperatures given the Eqs (12-14)[17].

$$\log K_D = \frac{\Delta S^0}{2.303.R} - \frac{\Delta H^0}{2.303.R.T} \quad \text{... (12)}$$

$$\Delta G^0 = \Delta H^0 - T.\Delta S^0 \quad \text{... (13)}$$

$$K_D = \frac{x/m}{y/u} \quad \text{... (14)}$$

where K_D is the distribution coefficient for the adsorption and is equal to the ratio of the amount adsorbed (x/m in mg/g) to the adsorptive concentration (y/u in mg/L). The values of ΔH^0 and ΔS^0 were determined from the slope and intercept of the linear plot of log K_D vs. 1/T.

Results and Discussion

Characterization of materials

PCNB ($C_6Cl_5NO_2$) is a fungicide formally derived from nitrobenzene. The chemical structure is given in Fig. 1. It is employed for suppressing the fungi growth of crops like cotton, rice, seed grains, etc [18]. The residual traces and metabolites (Pentachloro-aniline, PCA ($C_6Cl_5NH_2$) can be seen in the soil and river sediments (Eqs 15 & 16). It is widely used in China and Japan. It is either an off-white or yellow solid depending on its purity, with a musty odour.

$C_6Cl_5NO_2 + 6\ H \rightarrow C_6Cl_5NH_2 + 2\ H_2O$ … (15)

$C_6Cl_5NO_2 + 6\ H_2O \rightarrow C_6Cl_5OH + HNO_2$ … (16)

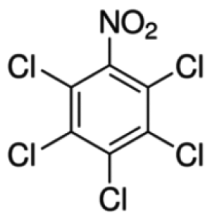

Fig. 1 — Chemical structure of PCNB

Functional groups present on the adsorbent were identified via FTIR spectra. Fig. 2 depicts the ponderosa as raw (a), carbonized form before the experiment (b) and carbonized form after the adsorption (c), respectively. The absorption peaks are seen at 750 (a), 840 (a), 712, 875 (b), 516, 660 and 780 (c), 880 (c), 970 (c) cm^{-1} are assigned to OH bending or C-O stretching vibration groups. 880 and 970 cm^{-1} are the indicator of the aliphatic C-Cl and aromatic C-Cl bands, respectively[19]. The absorption bands around 1119, 1050 (b), 1100 (c) and 1282 (c) cm^{-1} indicate C-O stretching. The peak intensity of the absorption bands in the spectral region 1250–1000 cm^{-1} is observed for the halogenated active carbon samples. 1100 cm^{-1} show the chloro group adsorption[19]. The small absorption band at 1282 cm^{-1} is associated with aromatic CO, phenolic OH and C-NO_2 stretching. The 1345 cm^{-1} peak is also due to the C-NO_2 interactions[20]. The peaks seen at around 1436 (a), 1429 (b), 1345, 1528 (c), 1631 (a), 1630 (b) and 1615 (c)cm^{-1} are due to the aromatic C=C ring stretching. 1744 (b) cm^{-1} is the indicator of aromatic carbonyl/carboxyl C=O stretching. Aliphatic CH stretchings are seen at 2853, 2922 (a), 2924 (b) and 2849 (c) cm^{-1}. The absorption band that is seen at 3438 (a), 3435 (b), and 3434 (c) cm^{-1} show OH stretching peaks.

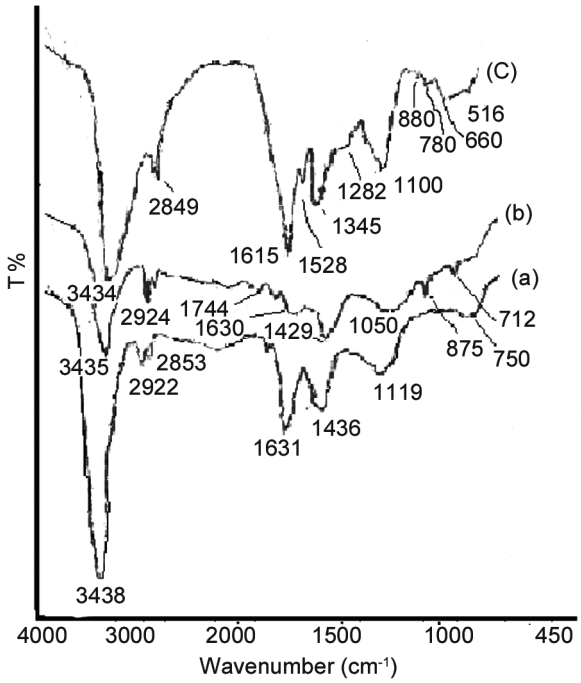

Fig. 2 — FTIR spectra of the samples (a) raw, (b) carbonized and (c) carbonized plus PCNB adsorbed

SEM images of the raw (a), carbonized (b) and carbonized plus PCNB adsorbed (c) activated carbons are shown in Fig. 3. There was a significant difference in the surface morphology of the materials after the carbonization treatment. The carbonized form gives more pores (b) on the surface than the raw one (a). The activated adsorbent has a considerable number of pores where there is a good possibility of the pesticide being trapped (c). The surface area and pore volume of the adsorbent measured by the BET method were 288.15 m^2/g and 78.24 cm^3/g, respectively.

Effect of contact time (t) on PCNB adsorption

Fig. 4 shows the effect of contact time on the removal of PCNB by activated biomass material. As shown in Fig. 4, when the initial PCNB concentration increased from 1 to 9.5 mg/L, the adsorption capacity increased. The adsorption equilibrium was obtained in 20 mins for 1 mg/L. No obvious concentration changes occur after the equilibrium state. As seen from the figure, the amount of adsorbed PCNB (mg/g) is increased with increased PCNB concentration from

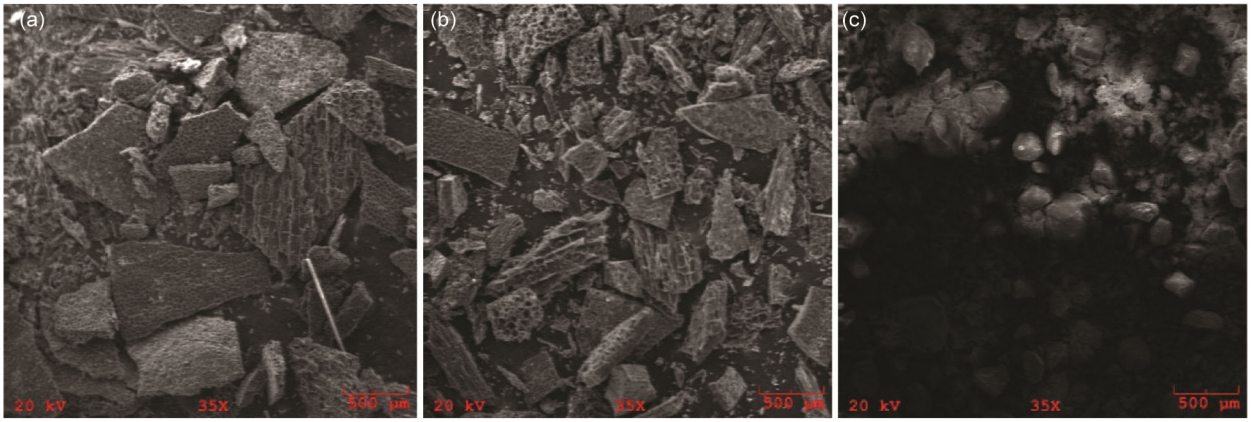

Fig. 3 — SEM images of the adsorbent (a) raw, (b) carbonized and (c) carbonized plus adsorbed magnification (350X)

3.5 mg/L to 9.5 mg/L and attained equilibrium in 75 mins for the more concentrated solutions. The concentration increase is rather an important driving force to overcome the mass transfer resistances of pesticides between liquid and solid phases.

The results demonstrated that pesticide solutions with low concentrations of 1 and 3.5 mg/L attained equilibrium faster than the high concentration of 6.5 and 9.5 mg/L PCNB solutions. The equilibrium was reached in approximately 30 min for 3.5 mg/L, while at higher initial concentrations of 6.5-9.5 mg/L, they come to equilibrium in 75 mins. This behaviour is similar to the paper of Hameed *et al*., for the adsorption of cationic dye from an aqueous solution[21].

Although the sorption capacity increased with the increase in initial pesticide concentration, the removal % of PCNB behaved the opposite way. With the increase in PCNB concentration from 1 to 9.5 mg/L, the loading capacity increased from 0.311 mg/g to 2.28 mg/g and the percentage removal decreased from 77 to 51% (Figs 5 & 6). This behaviour is seen in the adsorption of DDD and DDE onto bagasse fly ash[22].

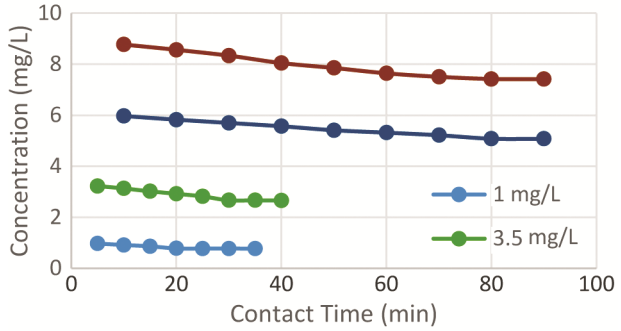

Fig. 4 — Effect of contact time on the removal of PCNB concentration (T=20°C, Agitation speed 120 rpm)

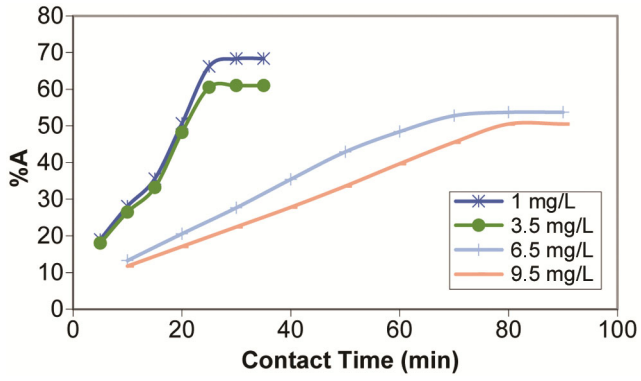

Fig. 5 — Effect of contact time on the removal of PCNB at 20°C

Effect of solution pH on PCNB adsorption

The removal of pesticide was studied at 20°C in the pH ranges of 2 to 8. The experiment was conducted by adding a 0.1 g carbonized adsorbent of 6.5 mg/L PCNB solutions (100 mL). As shown in Fig. 7, the amount of pesticide at equilibrium decreases from 77.20 to 38 % with the increase in pHs from 2 to 8. The experiments done at pH 10 has no effect on the removal of pesticide. The surface functional groups of pesticides make the adsorption process complex due to the charge characteristics of the adsorbent surface. In the acidic medium, the pesticide uptake increases faster than in the basic medium. This behaviour was seen in the adsorption of 2,4-D and carbofuran by granular activated carbon[23]. The surface funtional groups were determined from Boehm's titration and shown in Table 1. The acidic ones have a positive effect from the point of adsorption[24].

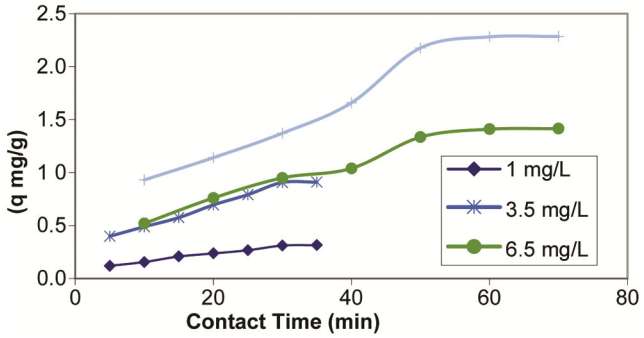

Fig. 6 — Effect of phase contact time on PCNB adsorption by activated carbon (Experimental conditions: pH=6, adsorbent dose=0.1 g/100 mL, T=20°C)

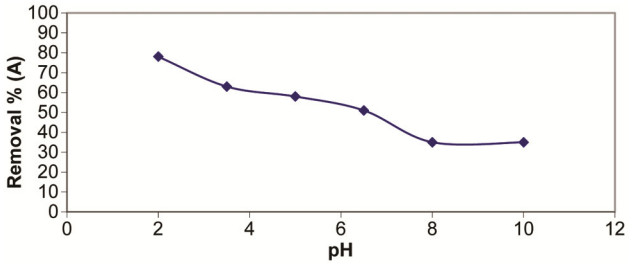

Fig. 7 — Effect of initial solution pH on the adsorption of PCNB by activated carbon (Experimental conditions: adsorbent dose = 0.1 g/100 mL, T: 20°C, contact time: 75 min, C= 6.5 mg/L)

Table 1 —The surface functional groups of the active carbon

Acidic functioal groups (mequiv./g)			All acidic groups (mequiv./g)	Basic surface groups (mequiv./g)
-COOH (Carboxylic)	-COO-/ =CO (Lactonic)	-OH (Phenolic)		
0.01	0.04	0.64	0.69	0.31

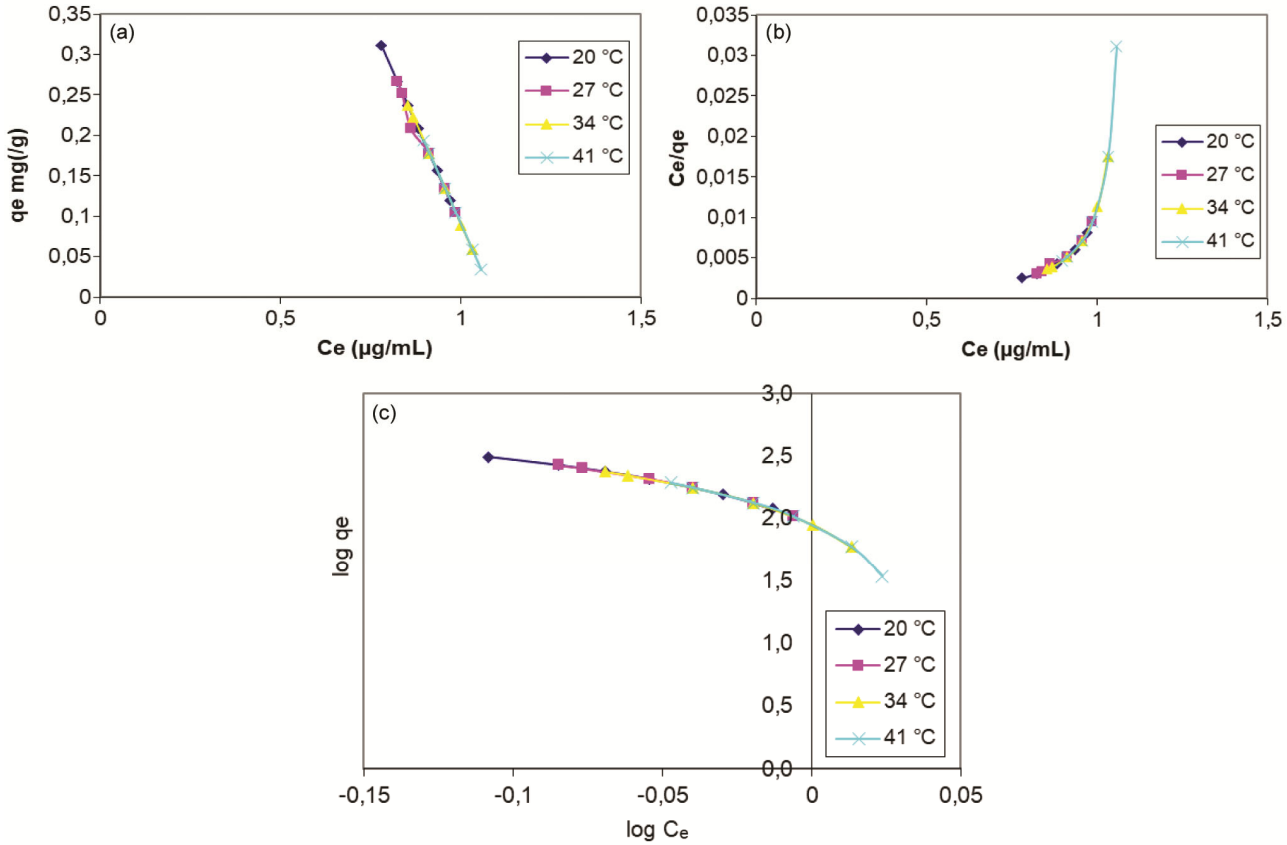

Fig. 8 — (a) Linear, (b) Langmuir and (c) Freundlich isotherm plots for PCNB adsorption onto activated carbon

Table 2 — Parameters of adsorption isotherms of PCNB onto carbonized adsorbent at different temperatures

Freundlich	20°C	27°C	34°C	41°C
k_F(mg/g)	113.608	104.256	80.448	74.131
N	0.232	0.197	0.143	0.099
R^2	0.9714	0.9767	0.9583	0.9393
Langmuir				
q_m (mg/g)	349.65	273.37	142.05	68.92
k_L	-1.403	-1.340	-1.217	-1.127
R^2	0.9298	0.9441	0.8721	0.8106
Linear				
K_{Li}	-1	-1.0113	-1.0131	-1..0331
R^2	1	0.9987	0.9987	0.9997

PCNB adsorption isotherms

The correlation of equilibrium data employing either theoretical or empirical equations is essential for the design of adsorption. The adsorption isotherms of PCNB onto carbonized adsorbent at temperatures 20, 27, 34 and 41°C, fitted with the linear, Langmuir and Freundlich isotherms are shown in Fig. 8. The isotherm parameters and linear regression coefficients obtained from the fitting of the sorption data to those mentioned isotherms by activated carbon are also given in Table 2.

Most R^2 values for carbonized adsorbent are rather high for the Linear and Freundlich models, suggesting that both models describe the experimental results well. Comparing the Langmuir and Freundlich models, the Freundlich model was more suitable than Langmuir. Similar behavior was seen in Wen *et al.*[25]. The monolayer adsorption capacity for carbonized adsorbent was found as 349.65 mg/g at 20°C.

The adsorption of zinc and dyes by various adsorbents was best represented by the Langmuir model[26,27] while chlorophenol removal via red mud obeyed the Freundlich model[28]. Table 3 shows the maximum monolayer adsorption capacity for different pesticides by various adsorbents.

Adsorption kinetic study

Pseudo first and second-order kinetic model

The plot of log (q_e-q_t) versus t at 20°C is shown in Fig. 9. The kinetic parameters of PCNB adsorption onto carbonized adsorbent according to the pseudo first-order model at different concentrations are given in Table 3. The results showed that the pseudo-first-order model fits the experimental data rather well, as most of the R^2 values are greater than 0.97 and

Table 3 — Adsorption capacity of various adsorbents

Adsorbent	Q_{max}(mg/g)	Pollutant	Reference
Carbonized ponderosa	349.65	PCNB	This study
Microwave activated carbonized coconut shells	256.41	Urea	29
Banana stalks activated carbon	156.30-164	Carbofuran	30
AC Cloth	115	Bentazon	31
Rice husk	68	Phosphate	32
Wood derived biochar	44.64	Ammonium nitrogen	33

Table 4 — Comparison of the pseudo-first-order, pseudo-second-order adsorption rate constant and calculated and experimental q_e values obtained at different initial PCNB concentrations

Co (mg/L)	Pseudo-first-order kinetic model				Pseudo-second-order kinetic model		
	q_e,exp (mg/g)	q_e,cal (mg/g)	k_1 (1/min)	R^2	q_e,cal (mg/g)	k_2 (g/mg min)	R^2
1	0.311	0.370	1.447	0.974	0.214	4.902	0.986
3.5	0.957	0.989	1.211	0.985	0.622	0.885	0.965
6.5	1.410	1.489	1.209	0.992	0.677	1.144	0.975
9.5	2.282	2.553	1.237	0.999	0.739	0.942	0.956

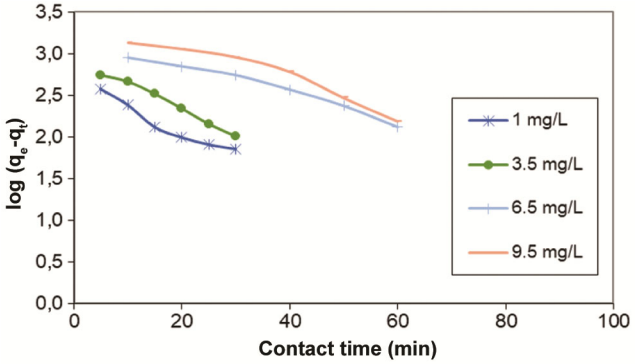

Fig. 9 — Kinetic plots of various PCNB concentrations on carbonized adsorbent (pseudo-first-order)

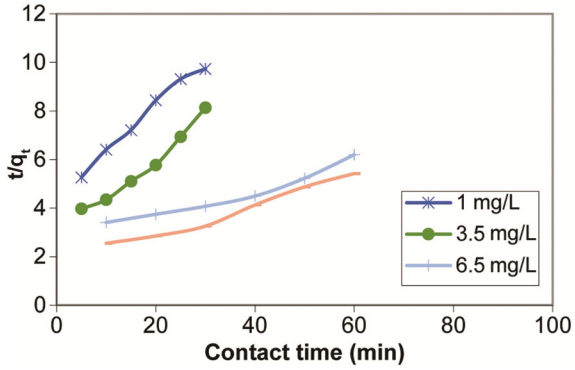

Fig. 10 — Kinetic plots of various PCNB concentrations on carbonized adsorbent (pseudo-second-order)

closeness of experimental and calculated adsorption capacity values. This indicates the applicability of the first-order kinetic model to describe the adsorption of PCNB onto carbonized adsorbent [25].

The second kinetic plot and the kinetic parameters are given in Fig. 10 and Table 4, respectively. The relatively low R^2 and rather high variables between

Table 5 — Thermodynamic parameters

Temperature (K)	ΔH° (J/g)	ΔS° (J/gK)	ΔG° (J/g)
293	-5960.900	9.769	-3098.583
300			-3030.300
307			-2961.817
314			-2893.434

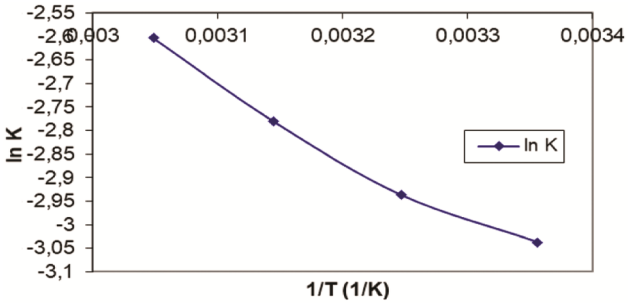

Fig. 11 — ln K versus 1/T relationship at different temperatures for determining thermodynamic parameters

the experimental and theoretical uptakes shown in Table 4 indicate the poor fitting of the pseudo-second-order model. Thus, a second-order adsorption is not considered. The adsorption of nitrobenzene from an aqueous solution by a novel lipoid adsorption material (LAM) was a similar behaviour [25].

Thermodynamic analysis

The thermodynamic analysis of PCNB adsorption on the carbonized biomass was conducted according to Eq. 12-14. The plot and the calculated parameters are shown in Fig. 11 and Table 5, respectively.

The Gibbs energy changes (ΔG°) were negative which indicated of spontaneous adsorption. A negative value of the standard enthalpy change (ΔH°) indicated that the PCNB adsorption was an

exothermic reaction. The positive entropy changes (ΔS°) supported the limited activity of the pesticide between the solution and the carbonized adsorbent. Removal of lindane and malathion from wastewater by a bagasse fly ash was given negative Gibbs energy but positive ΔH° and ΔS° values[34].

Those values are the normal consequence of the physical adsorption phenomenon which governs through electrostatic interactions. To further support that physical adsorption is the predominant mechanism, the values of activation energy (Ea) and sticking probability (S*) were estimated from the experimental data. They were calculated using a modified Arrhenius type equation related to surface coverage (θ) as follows [35].

$$S^* = (1-\theta)e^{-(E_{a/RT})} \quad \ldots (17)$$

The sticking probability, S*, is a function of the adsorbate/adsorbent system under observation, its value lies in the range 0 < S*< 1 and is dependent on the temperature of the system. The parameter S* shows the indicator of the potential of an adsorbate to remain on the adsorbent surface. The surface coverage, θ, can be calculated from the following equation:

$$\theta = \left(1 - \frac{C_e}{C_o}\right) \quad \ldots (18)$$

E_a and S values were found as -5.0413 J/g and 0.1 (0<S*<1), respectively. The negative values of E_a indicate the exothermic nature of the adsorption process and these values confirm that the sorption process is a physisorption phenomenon.

Conclusion

A novel adsorbent prepared from ponderosa was employed for the adsorption of PCNB from waste effluents. The average removal efficiency of pesticide by activated carbon was reached 77% after 20 min contact time for 1 mg/L initial concentration of PCNB. The adsorption isotherm data fitted the linear and Freundlich isotherms and was described by the pseudo-first-order kinetic model which displayed that the factors that affected the adsorption rate would be either the PCNB concentration or the characteristic of the activated adsorbent. Based on the negative Gibbs energy changes, the adsorption of PCNB was spontaneous and occurred as an exothermic reaction.

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Conflict of interest

The authors declare no conflict of interest.

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