

# REMOVAL OF CHLOROPHENOL FROM AQUEOUS SOLUTION USING FLUTED PUMPKIN AND COMMERCIAL ACTIVATED CARBON

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## ABSTRACT

*This work aimed at exploring the potential use of fluted pumpkin stem waste as a biosorbent for the removal of chlorophenol from aqueous solutions. Batch kinetics and isotherm studies were performed to evaluate the effects of process parameters such as pH, temperature, initial chlorophenol concentration and adsorbent dosage. The adsorption of chlorophenol increased with increasing initial chlorophenol concentration, and solution pH. The adsorption equilibrium was well represented by Langmuir than Freundlich adsorption isotherm models.*

**Keywords:** Chlorophenol, adsorption, fluted pumpkin, aqueous systems, activated carbon

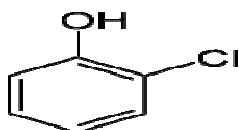
## INTRODUCTION

Phenolic compounds are pollutants of priority concern that enters water bodies through discharge from pharmaceuticals, petrochemicals and other chemical manufacturing processes (Mahvi et al., 2004; Denzeli et al., 2005). Chlorophenol (Figure 1) represent an important class of environmental water pollutants. Many of these compounds are present in our wastewaters from petrochemical, coal tar, plastics, pesticides and chemical industries, which produce them as chemical intermediates or generate them during chlorination of effluents containing compounds (Mostafa et al., 1989; Rengaraj et al., 2002). Industrial wastewaters are not only sources of chlorophenol; they could be present in domestic waters since they are widely used as pesticides, disinfectants and antiseptics. In addition, the common water treatment with chlorine generates chlorophenol since chlorine reacts quite fast with phenols Duarte-Davidson et al., (2004).

Chronic toxic effects due to phenolic compounds reported in humans include vomiting, difficulty in swallowing, anorexia, liver and kidney damage, headache, fainting and other mental disturbance. Among the different organic pollutions of aquatic ecosystems, phenols, especially the chlorinated ones, are toxic to animals and human even at low concentration (Asheh et al., 2003). The adsorbents used in this study are activated carbon produced from fluted pumpkin (*Telfairia occidentalis* Hook. F) stem waste impregnated with phosphoric acid and a powdered commercial activated carbon. Fluted pumpkin (*Telfairia occidentalis* Hook. F) is a creeping vegetative shrub that spreads low across the ground with large lobed leaves and long twisting tendrils. The genus *Telfairia* cucurbitaceae comprises two species, *T. pedata* and *T. occidentalis*. *T. pedata* is grown in East Africa for its oil and protein rich seeds while *T. occidentalis* is grown in some parts of West Africa for its nutritional leaves and seeds (Okoli, and Nyanayo, 1988).

Harvesting of fluted pumpkin takes place 120 - 150 days, after sowing. After harvesting, the leaves are carefully removed from the stems which are discarded as wastes. The seed contains 13% oil and is used for cooking (Horsfall and Spiff, 2005), marmalade manufacturing (Egbekun et al., 1998) and cookie formulations (Giami and Barber, 2004). Several workers have reported the nutritional composition, chemical characterization and functional properties of fluted pumpkin seeds (Fagbemi et al., 2005, Ganiyu, 2005, Agatemor, 2006 and Fasuyi, 2008). The utilization of fluted pumpkin stem waste in the wastewater treatment process would serve as an economically viable option to the increasing toxic threat to the environment. In this present study, the efficiency of fluted pumpkin activated carbon (FPAC) for chlorophenol removal as compared to a commercial activated carbon (CAC) was highlighted

The adsorbate used for the adsorption process of this study is chlorophenol.



**Chlorophenol C<sub>6</sub>H<sub>5</sub>ClO**

**Figure 1: Structure of chlorophenol**

## **MATERIALS AND METHODS**

### **Sample collection and preparation**

The fluted pumpkin stem waste (*Telfairia occidentalis Hook F*) used for this study, was obtained from Iwofe market Rumuolumeni Port Harcourt. The samples collected were washed thoroughly with water, rinsed with deionised water and air-dried. The air-dried samples were cut into smaller bits and carbonized. The powdered commercial activated carbon (Sifico Ltd.Surrey England) was bought from a scientific store in Port Harcourt.

### **Carbonization**

The carbonization of the air dried fluted pumpkin stem was carried out in the Plant Physiology and Anatomy Laboratory of the University Of Port Harcourt, Nigeria, using a muffle furnace (Carbolite Sheffield England, LMF4) which allows limited supply of air. To produce the carbonized sample of the fluted pumpkin carbon, the sample was heated at a rate of 5°C per minute to 350°C.

### **Acid activation of the carbon**

A carefully weighed 25.0 ± 0.01g carbonized carbon was placed in a beaker containing 500cm<sup>3</sup> of 0.3mol/dm<sup>3</sup> ortho-phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). The content of the beaker was thoroughly mixed, heated until it formed a paste. The paste was transferred to an evaporating dish, placed in a furnace and heated at 300°C for thirty minutes. This was allowed to cool and washed with distilled water to constant pH, and oven dried at 105°C for four hours to constant weight and ground. The produced carbon was sieved with a 106µm mesh which was kept in an air tight vial and used for the various experiments. Solutions were prepared by diluting of stock solution of chlorophenol to the desired concentrations. A stock solution was obtained by dissolving 1.0g of chlorophenol, in distilled water and diluted to 1000ml.

## **Adsorption equilibrium**

Equilibrium studies were carried out by contacting 0.2g of FPAC and CAC with 50ml chlorophenol solution of different initial concentrations (100,120,140,160,180 200mg/l) in twelve (250ml) conical flask. The pH of the solutions were adjusted to 6.0 by adding either HCl or NaOH solution. The suspensions were agitated at 150rpm on a shaker at room temperature (30°C) for 1 hour. At the end of the agitation period the samples were centrifuged for 5min and the supernatant 2CP solution, filtered through Whatman No 40 filter paper. 2-chlorophenol concentrations in the filtrate were analyzed using a UV/Vis spectrometry measuring absorbance at the wavelength of maximum absorption for 2- chlorophenol (280nm). Samples were run in duplicate.

### **Effect of contact time at 30°C**

0.2g each of FPAC and CAC of 106µm mesh particle size were weighed and put in twenty two (250ml) conical flasks. 50ml of 100mgL<sup>-1</sup> concentration of 2-chlorophenol solutions prepared separately in de-ionized water from the stock solution was added to the biomass. The pH values of these suspensions were adjusted to 6.0. The flasks were labelled for time interval of 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120 minutes. The flasks were tightly covered with cellophane and shaken at 150rpm for the appropriate time intervals on an electric shaker. The suspensions was filtered through Whatman No 40 filter paper and centrifuged for 5 minutes. The supernatants were analyzed using UV-Visible spectrophotometer. Determinations were run in duplicate.

### **Effect of pH AT 30°C.**

0.2g each of FPAC and CAC activated carbons of 106µm mesh particle size were weighed and introduced into twelve (250ml) conical flasks. 50ml of 100 mgL<sup>-1</sup> solutions of 2- chlorophenol were added separately to the activated carbons. This process was repeated for several other flasks. The pH value of these suspensions was adjusted to 2.0, 4.0, 6.0, 8.0, 10.0and 12.0 by adding a solution of either Conc HCl or NaOH. The flasks were tightly covered with cellophane and shaken for 1 hour at 150rpm. The suspension was filtered through Whatman No 40 filter paper and centrifuged for 5 minutes. The supernatants were analysed using UV-Visible spectrophotometer. Determinations were run in duplicate.

### **Effect of adsorbent dosage.**

Different masses of FPAC and CAC activated carbon ranging from 0.1 to 0.7g were accurately weighed and transferred into fourteen (250ml) conical flasks. 50ml of chlorophenol was added separately to each of the conical flasks. The flasks were tightly covered with cellophane and shaken for 1 hour at 150rpm. At the end of 1 hour, the suspensions were filtered using Whatman No 40 filter paper and centrifuged for 5 minutes. The supernatant 2- chlorophenol solution was analyzed using UV- Visible spectrophotometer. Determinations were run in duplicate.

### **Effect of temperature.**

50ml of 2-chlorophenol solutions separately with an initial concentration of 100mgL<sup>-1</sup> was placed in fourteen (250ml) conical flasks. 0.2g each of FPAC and CAC activated carbon was added to these solutions. The conical flasks were labelled at temperatures of 30, 40, 50, 60, 70, 80 and 90 °C respectively. The flasks were agitated at 150rpm and heated on a thermostat water bath to the appropriate temperatures for 1h. The suspensions were filtered using Whatman No 40

filter paper and centrifuged for 5 minutes. The supernatants were analysed using UV-Visible spectrophotometer. Determinations were run in duplicate.

The amount of adsorption at equilibrium,  $q_e$  (mg/g) and the percent adsorption (%) was computed as follows:

$$q_e = \frac{(c_o - c_e)V}{M} \dots \dots \dots (1)$$

$$\text{percent adsorption} = \frac{(c_o - c_e) \times 100}{c_o} \dots \dots \dots (2)$$

Where  $C_o$  and  $C_e$  are the initial and equilibrium concentrations of chlorophenol (mg/l), respectively.  $V$  is the volume of the aqueous solution (L) and  $M$  is the mass of the activated carbon used in the adsorption (g).

## RESULTS AND DISCUSSION

### Effect of carbon dosage

The adsorption of chlorophenol on FPAC and CAC were carried out at different adsorbent dose by keeping other parameters constant. The relationship between adsorbent dose and substrate removal for same initial concentrations of chlorophenol is presented in Fig 2, respectively. It can be seen from Figure 2 that percentage removal of chlorophenol on FPAC and CAC increased with increase in adsorbent dose. This can be attributed to increased adsorbent surface area and availability of more adsorption sites resulting from the increased adsorbent dosage as observed by Srivastava et al., 2006. The uptake of solute markedly increased up to adsorbent dose of 0.2g and thereafter no significant increase was observed. It has been shown in Fig 2 that the rate of chlorophenol binding with adsorbents increases more rapidly in the initial stage and after some point adsorption is marginal and becomes almost a constant.

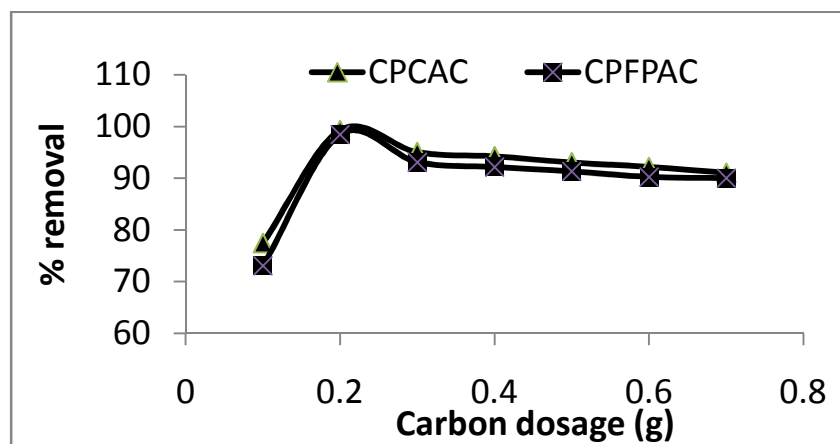


Fig 2: Effect of carbon dosage on the percentage removal of chlorophenol on FPAC and CAC

### Effect of carbon contact time

The effect of contact time on the percentage removal of chlorophenol from solution by FPAC and CAC was studied at varying contact time of 20 to 120 minutes as presented in Figure 3. The optimum percentage removal of 2-chlorophenol was achieved at 60 minutes, showing the following trend CPFPAC (99.00%) < CPCAC (99.90 %).

The saturation curves rises sharply in the initial stages, indicating that there are plenty of readily accessible sites. Eventually, a plateau is reached in all curves indicating that adsorbent is saturated at this level Vadivelan and Kumar, (2005). Figure 3 shows that the contact time needed for chlorophenol solutions of 100mg/l to reach equilibrium is 60 minutes. Almost, no remarkable improvement was observed after longer contact time. After this equilibrium period, the amount of solute adsorbed did not change significantly with time, indicating that 60 minutes is sufficient to attain equilibrium for the maximum removal of chlorophenol from aqueous solutions by FPAC and CAC, respectively. So the optimum contact time was selected as 60 minutes for further experiments.

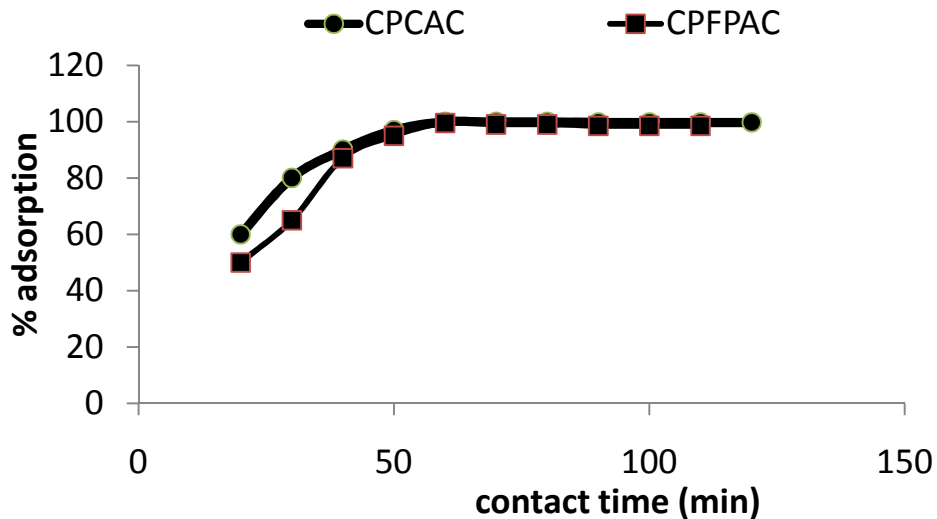


Fig 3: Effect of carbon contact time on the percentage removal of chlorophenol on FPAC and CAC

### Effect of initial concentration

Fig 4 shows that percentage of chlorophenol removal decreases with increasing concentration. Similar results were obtained by Singh et al., (1994). This is because at lower concentrations the activated carbon is able to remove large amounts of chlorophenol from solution. As the initial concentration increases the binding sites gradually becomes occupied, this reduces the percentage removal of chlorophenol removed. These removal characteristics indicate that surface saturation is dependent on the initial concentration and that the active sites take up the available chlorophenol at lower concentration of 100mg/l.

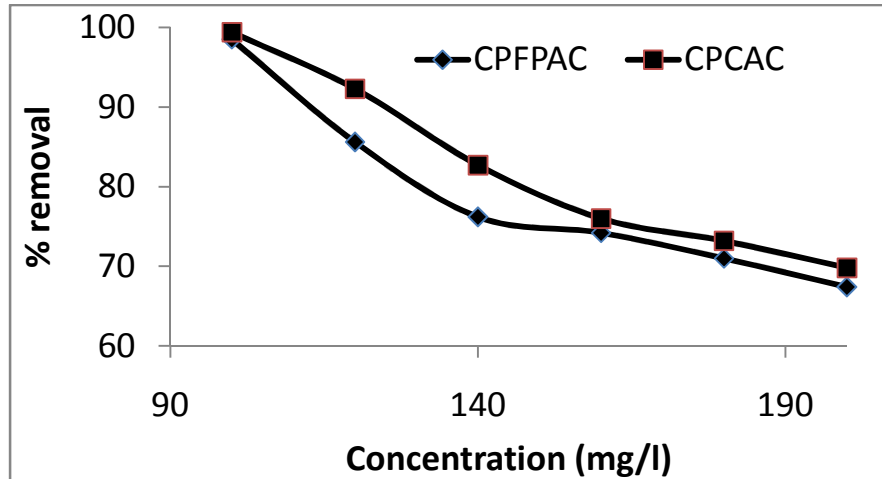


Fig 4: Effect of initial concentration on the percentage removal of chlorophenol on FPAC and CAC

### Effect of temperature

The effect of the solution temperature on the percent removal of chlorophenol by FPAC and CAC was studied at a constant initial chlorophenol concentration of 100mg/l, carbon dose of 0.2g at temperature range of 30 to 90°C. The effect of temperature and pH in this study is shown in Table 1

Table 1: Effect of temperature and pH on the percentage removal of chlorophenol on FPAC and CAC biomass.

TEMP °C	Chlorophenol		pH	Chlorophenol	
	CAC (%)	FPAC (%)		CAC (%)	FPAC (%)
30 <sup>0</sup> c	99.71	99.23	2	68.25	62.45
40 <sup>0</sup> C	85.34	80.02	4	85.12	78.26
50 <sup>0</sup> C	69.50	64.20	6	99.20	98.07
60 <sup>0</sup> C	66.38	62.05	8	70.54	72.21
70 <sup>0</sup> C	64.10	61.08	10	60.35	63.68
80 <sup>0</sup> C	63.00	60.03	12	50.25	51.05
90 <sup>0</sup> C	63.00	60.03			

\*Results are means of duplicate analyses

Table 1 show that the percentage removal of chlorophenol decreased as temperature increased from 30°C to 90°C as follows CFPAC (24.93 to 15.25mg/g) and CPCAC (24.84 to 15.08mg/g). Hence the lower temperature is favourable for the adsorption of chlorophenol. The decrease in adsorption with increase in temperature is partly due to the weakening of the attractive forces between the adsorbates and adsorbent. It could also be attributed to the enhancement of thermal energies of the adsorbate, thus making the force between adsorbent and adsorbate insufficient to retain the adsorbed molecules at the binding sites.

The rapid removal of chlorophenol further indicates that both physisorption and chemisorptions processes may be involved in the adsorption of the adsorbates by the adsorbents. The results of Nagda et al., (2007) using Tendu leaf biomass and Uddin et al., (2007) on water hyacinth biomass were similar. In general more than 95% chlorophenol removal was achieved at 30°C on FPAC and CAC, respectively. The overall high percentage of chlorophenol removal from solution could possibly be due to adsorption through ion exchange, hydrogen bonding, van-der Waal's forces as well as intraparticle diffusion.

**Effect of pH**

In any adsorbate - adsorbent system, pH of the solution affects the surface charge of the adsorbents, affects ionization and the extent and rate of adsorption (Nagda et al., 2007). Change in pH affects the adsorptive process through dissociation of functional groups. This subsequently leads to a shift in reaction kinetics and equilibrium characteristics of adsorption process. The effect of pH on the adsorption of chlorophenol was studied, varying the pH from 2 to 12. The maximum adsorption occurs at pH 6 as shown in Table 1 and thereafter a significant decline in removal efficiency was observed with further increase in pH. The maximum uptake of adsorbate established the following trend CPCAC (24.80mg/g) > CFPAC (24.51mg/g).

**Adsorption isotherm**

Sorption equilibria provide fundamental physiochemical data for evaluating the applicability of sorption process as a unit operation. In the present investigation, the equilibrium data were analysed using Langmuir and Freundlich isotherm models. To facilitate the estimation of the sorption capacities, experimental data from the initial concentrations were fitted to Langmuir and Freundlich equilibrium adsorption isotherms. In order to facilitate the estimation of the adsorption capacities at various conditions, the Langmuir adsorption isotherm, a typical model for monolayer adsorption was applied. The Langmuir equation was modelled using the equation below.

$$\frac{C_e}{q_e} = \frac{1}{q_m k_L} + \frac{C_e}{q_m} \dots \dots \dots (3)$$

Where  $C_e$  is the equilibrium concentration of solute in solution (mg/l),  $q_e$  is the amount of solute adsorbed at equilibrium (mg/g),  $q_m$  (mg/g) and  $k_L$  (L/mg) are the Langmuir constants, and  $q_m$  signifies adsorption capacity and  $k_L$  is related to energy of adsorption process. A plot of  $C_e/q_e$  against  $C_e$  yields a straight line, while  $q_m$  and  $k_L$  are obtained from the slope and intercept.

The experimental equilibrium data was also analysed using the Freundlich isotherm model. The Freundlich isotherm was adopted to characterize the adsorption intensity of chlorophenol by fitting the experimental data. The linearized form of the Freundlich equation is;

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \dots \dots \dots (4)$$

Where  $q_e$  is the adsorption intensity (mg/g),  $C_e$  is the concentration of the adsorbate in solution at equilibrium (mg/dm<sup>3</sup>),  $K_F$  and  $n$  are the Freundlich constants. If a plot of  $\log q_e$  against  $\log C_e$  yields a straight line, then the sorption process obeys a Freundlich adsorption isotherm. The  $K_F$  and  $\frac{1}{n}$  can be obtained from the intercept and slope of the straight line. The linearized Langmuir and Freundlich adsorption isotherms of chlorophenol on FPAC and CAC are shown in (Fig 4) and (Fig 5) respectively. The Langmuir and Freundlich constants are displayed in (Table 2).

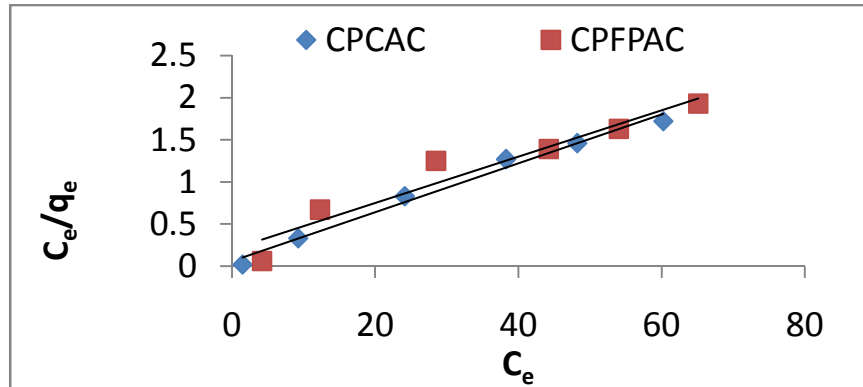


Fig 5. Langmuir plots for the sorption of various concentrations of chlorophenol from solution by FPAC and CAC

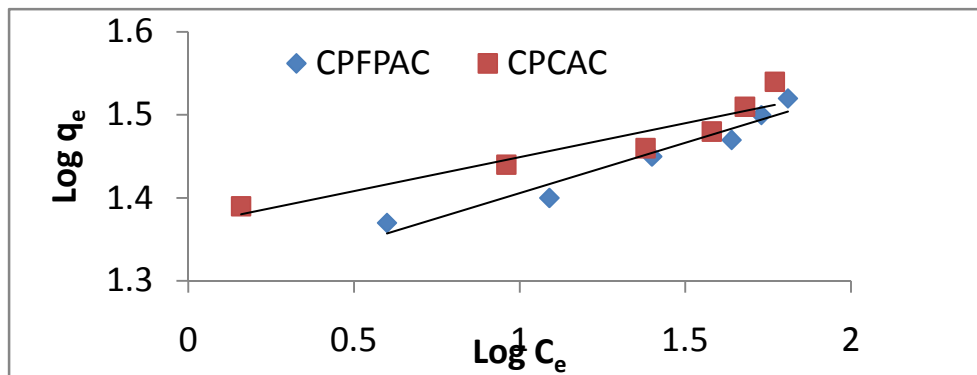


Fig 6. Freundlich plots for the sorption of various concentrations of chlorophenol from solution by FPAC and CAC

Table 2: Langmuir and Freundlich constants parameters for the sorption of chlorophenol onto FPAC and CAC.

LANGMUIR ISOTHERM				
Adsorbate/ Adsorbent	Maximum sorption capacity $q_m$ (mg/g)	Adsorption / Desorption energy $K_L$ (dm <sup>3</sup> /g)	Regression coefficient, $R^2$	Equilibrium parameter $R_L$
CPCAC	37.030	0.135	0.930	0.068
CPFAPAC	34.480	0.490	0.986	0.020
FREUNDLICH ISOTHERM				
Adsorbent/ Adsorbate	Intensity of adsorption, $n$ (L/mg)	Adsorption capacity, $K_F$ (mg/g)	$R^2$	
CPCAC	12.340	23.280	0.884	
CPFAPAC	8.260	19.230	0.944	

From the data in Table 2, the Langmuir constant,  $q_m$  which is a measure of the maximum sorption capacity corresponding to complete monolayer coverage shows that chlorophenol has a higher mass capacity (37.030mg/g) on CAC than on FPAC (34.480 mg/g). The adsorption



coefficient energy for chlorophenol on FPAC (0.490dm<sup>3</sup>/g) is higher than on CAC (0.135dm<sup>3</sup>/g), respectively.

The favourable nature of adsorption can be expressed in terms of a dimensionless equilibrium parameter reported in Horsfall and Spiff, (2004) equation 5.

$$R_L = \frac{1}{1+k_L C_o} \dots\dots\dots (5)$$

Where  $K_L$  is a Langmuir constant (L/g) and  $C_o$  is the initial concentration of chlorophenol. The values of  $R_L$  indicates if the type of isotherm is irreversible ( $R_L= 0$ ), favourable ( $0 < R_L<1$ ), linear ( $R_L=1$ ) or unfavourable ( $R_L>1$ ). The dimensionless separation factors calculated for chlorophenol on FPAC (0.068) is higher than on CAC (0.020), indicating favourable adsorption as values are less than 1. The regression values as shown in Table 2 depicts that the linear form of the Langmuir isotherm appears to produce a more reasonable model for the sorption of chlorophenol. Similar observations were reported by Aktas and Cecen, 2006; Dursun and Kalayei 2005. The  $K_F$  values suggest that chlorophenol has greater tendency towards CAC than FPAC (Rengaraj et al., 2002; Qadeer and Rehan, 2002).

## CONCLUSIONS

The following conclusions are drawn from the experimental results of this study:

1. Adsorbent prepared from fluted pumpkin stem waste compared favourably to the commercial activated carbon
2. Adsorption process of chlorophenol from aqueous solution favoured Langmuir better than Freundlich isotherm model.
3. The maximum adsorption of chlorophenol was at pH 6.0 for both activated carbons.

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