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POTENTIAL OF FLUTED PUMPKIN AND COMMERCIAL ACTIVATED CARBONS FOR PHENOL REMOVAL IN AQUEOUS SYSTEMS

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ABSTRACT

The potential of fluted pumpkin activated carbon made from fluted pumpkin (*Telfairia occidentalis Hook F*) stem waste and commercial activated carbon was studied. The fluted pumpkin stem waste was carbonized and activated with 0.3M ortho-phosphoric acid. Batch kinetic and isotherm studies were carried out under varying experimental conditions of contact time, phenol concentration, temperature, adsorbent dosage and pH. The results showed that the amount of phenol adsorbed increased with increasing initial phenol concentration and decreased with increasing pH. Langmuir and Freundlich adsorption models were used for the mathematical description of adsorption equilibrium. It was found that the experimental data fitted Langmuir better than Freundlich models. The result illustrated how fluted pumpkin stem waste, an environmental nuisance in Nigeria can be converted to a useful activated carbon for phenol removal in aqueous solution.

Keywords: model, activated carbon, phenol removal, aqueous system, adsorption, fluted pumpkin.

INTRODUCTION

Pollution by phenols (Figure-1) is an important environmental issue. Phenols are common contaminants in wastewater generated from oil, gasoline, coal, paper, petrochemicals, pharmaceuticals, pesticides and dye manufacturing industries (Kujawski *et al*., 2004; Entezari *et al*., 2005). They are considered one of the priority pollutants in wastewater, because they are harmful to organisms even at low concentrations and many of them have been classified as hazardous pollutants because of their potential harm to human health. The low volatility of phenol and its affinity to water make oral consumption of contaminated water the greatest risk to humans (USEPA, 1987; Mostafa *et al*., 1989; Prpich and Daugulis, 2005). This aromatic compound is water soluble and highly mobile and as such, waste waters generated from these industrial activities contain high concentrations of phenolic compound (Dursun and Kalayei, 2005) which eventually may reach down to streams, rivers, lakes and soil, representing a serious ecological problem due to their widespread use and occurrence throughout the environment (Sofia *et al*., 2005). In the past several decades, extensive research has been conducted to develop innovative and promising adsorbent material for dealing with the treatment problem of contaminated industrial effluents. There is a growing interest in cost effective innovative materials and method that will be useful in the treatment of industrial waste streams. Many agricultural by-products that are available at little or no cost have been reported to be capable of removing substantial amounts of phenols and phenolic derivatives from aqueous solutions (Okieimen, *et al*., 2007). 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. *T. occidentalis* is grown in some parts of West Africa for its nutritional uses (Horsfall and Spiff, 2005). Several workers have reported the nutritional composition of fluted pumpkin seeds (Fagbemi *et al*.,

2005; Ganiyu, 2005; Agatemor, 2006; Fasuyi 2008). A single stem with leaves weighing 1.2kg produces less than 200g of leaves, leaving over 1kg stem as waste. Fluted pumpkin is the largest consumed vegetable in the West African sub region and therefore, creates one of the major agro waste problems in Nigeria. Preliminary investigations showed that several tons of these waste are produced daily in market places around the country but scarcely useful and therefore create environmental nuisance. The objectives of this study were: (1) to explore the possibility of producing fluted pumpkin activated carbon (FPAC) and comparing it with a commercial activated carbon (CAC) as an adsorbent for adsorption of phenol. (2) To investigate the effects of initial phenol concentration, contact time, pH and adsorbent dosage on the adsorption of phenol from aqueous solution. Equilibrium isotherm data were fitted to Langmuir and Freundlich equations and constants of isotherm equations were determined.
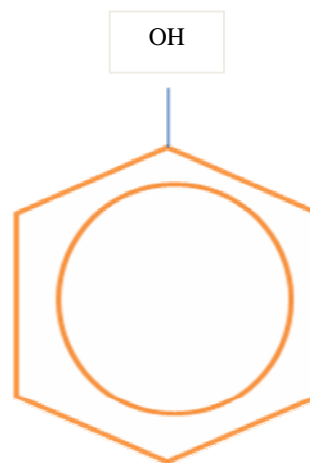

Figure-1. Structure of phenol (C_5H_6OH)

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MATERIALS AND METHODS

The fluted pumpkin stem waste (*Telfairia occidentalis Hook F*) used for this study was obtained from Iwofe market Rumuolumeni Port Harcourt. The stems collected were washed thoroughly with water, cut into smaller bits rinsed with distilled water, air dried, and later oven dried at 105°C for 10h. The oven-dried fluted pumpkin was carbonized to obtain the carbonized biomass.

Carbonization

Carbonization was carried out in the Plant Physiology and Anatomy Laboratory of the University of Port Harcourt, using a muffle furnace (Carbolite Sheffield England LMF4) which allows limited supply of air. Carbonization was done at 350°C for two hours and allowed to cool at room temperature for three hours before activation.

Acid activation of the biomass

A carefully weighed 25.00 ± 0.01g carbonized fluted pumpkin was placed in a beaker containing 500cm^3 of 0.3M H$_3$PO$_4$. The content of the beaker was thoroughly mixed, heated until it formed a paste. The paste was put in a crucible and placed in a furnace which heated to 300°C for thirty minutes. The fluted activated carbon was washed free of acid to get a pH of 6.7 ± 0.12. This was allowed to cool, washed with distilled water, oven dried at 105°C to constant weight and ground. It was sieved with a 106µm mesh to obtain a fine powdered activated carbon which was used for the various experiments. The treatment of the adsorbent with 0.3M H$_3$PO$_4$ solution aids the removal of any debris or soluble bio- molecules that might interact with phenol during sorption. The commercial activated carbon (Sifico Ltd. Surrey England) was bought from a scientific store in Port Harcourt and used without treatment.

Chemicals

The test solutions were prepared by diluting a stock solution of phenol to the desired concentrations. A stock solution was obtained by dissolving 1.0g of phenol (obtained from Merck India), in distilled water and diluted to 1000ml. Desired solutions of phenol were prepared using appropriate subsequent dilutions of the stock solution. The range in concentrations of phenol prepared from standard solution varied between 40mg/l to 100mg/l. Before mixing the adsorbents, the pH of each test solution was adjusted to the required value with dilute 0.1M H$_2$SO$_4$ and 0.1M NaOH solutions.

Adsorption equilibrium

Equilibrium studies were carried out by contacting 0.2g of fluted and commercial activated carbon with 100ml of phenol solution of different initial concentrations (100, 120, 140, 160, 180, and 200mg/l) in 250ml stopper conical flasks. The samples were agitated on a shaker at 200rpm constant shaking rate for 1h. This was immediately centrifuged at 150rpm for 5minutes and

then decanted. The supernatants' were analyzed using a UV/VIS spectrometry at 269nm with distilled water (H$_2$O) as blank. The percent phenol removed by the fluted pumpkin activated carbon (FPAC) and commercial activated carbon (CAC) was calculated from the difference between the initial (C_o) and equilibrium (C_e) adsorbate (Phenol) concentrations, which is given in equation 1.

$$\%R = \frac{C_o - C_e}{C_o} \times 100 \qquad \text{............................... (1)}$$

3 EFFECT OF pH AT 30°C.

0.2g each of activated carbons of 106µm mesh particle size were weighed and introduced into several 250ml conical flasks. 50ml of 100 mgL^{-1} solutions of phenol was added 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, 10.0 and 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 analyzed using UV-visible spectrophotometer.

Effect of temperature

50ml of phenol solution with an initial concentration of 100mgL^{-1} was placed in several 250ml conical flasks. 0.2g each of activated carbon was added to these solutions. The conical flasks were labeled at temperatures of 30, 40, 50, 60, 70, 80 and 90 ^{0}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 analyzed using UV-visible spectrophotometer

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Effect of contact time

The effect of contact time on the amount of phenol removal was analyzed over the contact time range from 20 to 120mins. In this study, 50ml phenol solution of 100mg/l was taken in stopper conical flask and was agitated at 200rpm with 0.2g of FPAC and CAC for the various contact time of 20 - 120mins. The suspensions were filtered through Whatman No 40 filter paper, centrifuged at 150rpm for 5 minutes decanted and analyzed.

Effect of adsorbent dosage

Different masses of activated carbon ranging from 0.1 to 0.7g were accurately weighed and transferred into several 250ml conical flasks. 50ml of phenol was added 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

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Whatman No 40 filter paper and centrifuged for 5 minutes. The concentration in the supernatant phenol solution was analyzed using UV- visible spectrophotometer.

RESULTS

The data for Physico chemical properties are presented in Table-1. The proximate analyses showed a low amount of moisture, ash and volatile matter, indicating that the particle density is relatively small and that the biomaterial should be an excellent raw material for use as adsorbents in column or fixed -bed reactors. Most activated carbons are specified for a pH of 6-8, from the above, the pH of the activated carbon samples were in the order FPAC (6.7±0.02) < CAC (7.0 ±0.01). The pH values of the activated carbon samples are therefore within the specified range of activated carbon. The porosity and iodine number is comparable to other materials used for batch analysis (Horsfall and Abia, 2003; Tarawou *et al*., 2007).

Table-1. Physico chemical characterization of FPAC and CAC.

Property	FPAC	CAC
pH	6.70 ±00	7.00 ±0.01
Moisture (%)	19.50 ±0.02	16.67 ±0.03
Porosity (%)	0.77 ±0.05	0.89 ±0.01
Iodine	200.90 ±0.30	224.46 ±0.50
Ash (%)	22.38 ±0.02	17.10 ±0.10
Volatile matter (%)	40.06 ±3.22	37.32 ±2.02
Particle density (g/cm^3)	3.50 ± 3.22	3.12 ±2.52

Effect of initial concentration

The percentage removal of phenol was examined by plotting the percentage of phenol removed against different initial concentrations as presented in Figure-1.

Figure-2 shows that percentage of phenol removal decreases with increasing concentration. Similar results were obtained by Singh *et al*., (1994); Uddin et al., (2007).This is because at lower concentrations the activated carbon is able to remove large amounts of phenol from solution. These removal characteristics indicate that surface saturation is dependent on the initial concentration and that the active sites take up the available phenol at lower concentration of 100mg/l.

It was observed that the percentage of phenol removed favored concentrations of 100mg/l for both FPAC and CAC. It was observed that more than 90% of phenol was removed in lower concentration of the activated carbon studied. Phenol removal for CAC was higher at 100mg/l with 92.30% and 91.54% for CAC. The high sorption at the initial concentration may be due to an increased number of vacant sites on the adsorbents available at the initial stage. As concentration is increased there is a decrease in percentage removal of phenol due to the accumulation of phenol particles on the surface. This observation is in line with the findings of Mahvi *et al*., 2004.
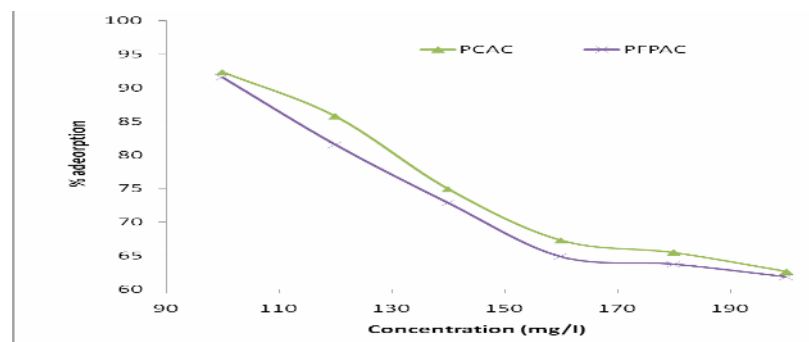

Figure-2 Effect of initial concentration on % removal of phenol from solution.

Effect of pH on phenol adsorption

The initial pH of an adsorption medium is one of the most important parameters affecting the adsorption process. The effect of pH on the adsorption of phenol was studied varying the pH from 2 to 12 as shown in Figure-3. It was observed that the relative amount of phenol adsorbed was significantly affected by pH. The maximum adsorption occurs at pH 6 for both FPAC and CAC. Significant decline in removal efficiency was observed for further increase in pH, which may be attributed to formation of phenolate anions and at the same time the presence of hydroxyl ions on the adsorbent which prevent the uptake of phenolate ions (Banat *et al.*, 2000; Nagda *et al*., 2007; Uddin *et al*., 2007; Hamdaoui and Naffrechoux, 2007).

The maximum uptake of adsorbate was highest at optimum pH of 6.0, with the following trend established PCAC (24.38mg/g) > PFPAC (23.91mg/g). Phenol considered in this study, have pKa value of 10; when the pH of a solution goes beyond the pKa, phenol chiefly exist as negative phenolate ions, whereas they exist as neutral molecules below the pKa. Due to the electron rich nature of the hydrogen atom in phenolate ions, the hydrogen bonding efficiency decreases. Therefore, phenols effectively adsorbed on to the adsorbent as molecules but not phenolate ions. At high pH value greater than 6 for fluted pumpkin and commercial activated carbon, OH$^-$ ions would compete with the phenol molecules for sorption sites. Sorption of excess OH$^-$ ions could populate an initial positively charged surface resulting in repulsion of negatively charged phenoxide ions and thereby causing decreased adsorption. From the experimental results pH 6 was selected as an optimum pH. The trend established on both activated carbons showed that phenol on CAC was slightly better adsorbed than phenol on FPAC.
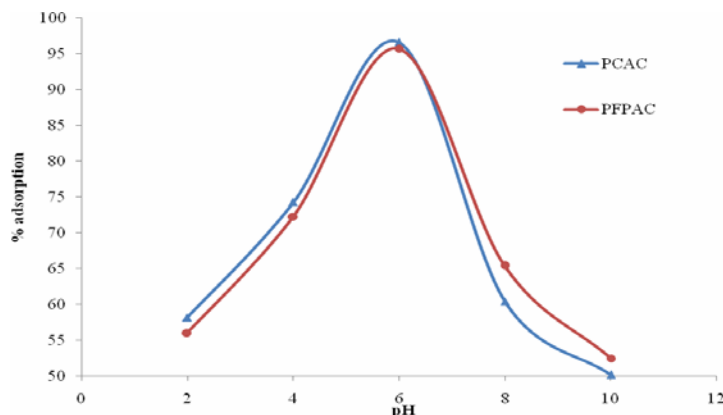

Figure-3. Effect of pH on the percentage removal of phenol from solution on
FPAC and CAC.

Effect of temperature on phenol adsorption

Figure-4 is a plot depicting the effect of temperature on percentage adsorption of phenol on FPAC and CAC. The percentage removal of phenol was optimum at 30°C for FPAC and CAC. Adsorption decreased with increasing temperature. The amount of phenol adsorbed decreases at temperature of 30°C to 90°C as follows PFPAC (24.88 to 13.92mg/g), PCAC (24.52 -14.05mg/g) Hence the lower temperature is favorable for the adsorption of phenol. 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 phenol further indicates that both physisorption and chemisorption 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% phenol removal was achieved at 30oC on FPAC and CAC, respectively. The overall high percentage of phenol removal from solution could possibly be due to adsorption through ion exchange, hydrogen bonding, van-der Waal's forces as well as intraparticle diffusion.
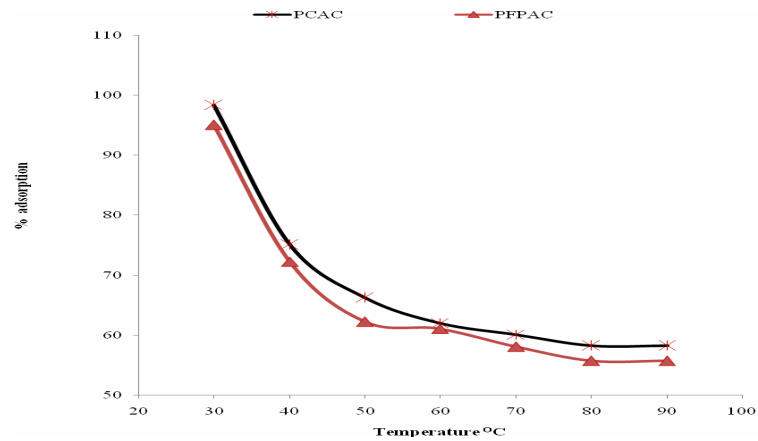
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Figure-4. Effect of temperature on the adsorption of phenol on FPAC and CAC.

Effect of contact time

The effect of contact time on the percentage removal of phenol from solution by FPAC and CAC was studied at varying contact time of 20 to 120 minutes as presented in Figure-5. The optimum percentage removal of phenol was achieved at 60 minutes, showing the following trend PCAC (98.50%) > PFPAC (98.40%).

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 , Nagda et al., 2000; Vadivelan and Kumar, 2005; Uddin et al., 2007. Figure 4, shows that the contact time needed for phenol 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 60minutes is sufficient to attain equilibrium for the maximum removal of phenol from aqueous solutions by FPAC and CAC, respectively. It is a proven fact that adsorption equilibrium time is dependent on the type of adsorbent. Kannam and kruppasmy (1998) using tea powder and saw dust obtained adsorption equilibrium within (180min) contact time. Singh et al., (1994) reported that for impregnated fly ash, the maximum removal of phenols was found at a contact time of 120min. Qadeer and Rehan 2002 worked on a commercial activated carbon and observed that the adsorption of phenol was instantaneous attaining equilibrium within five minutes. The decrease in the adsorption of phenol with time could be due to the accumulation of phenol particles in the vacant sites leading to a decrease in sorption percent from time 70 to120 minutes.
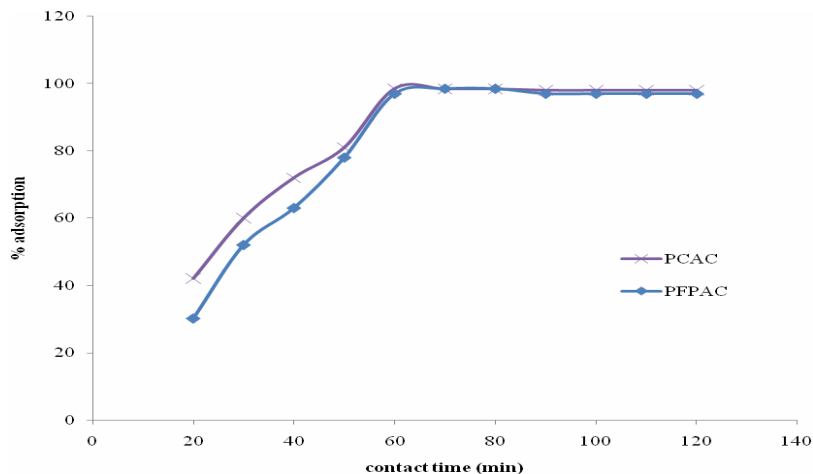

Figure-5. Effect of contact time on the percentage sorption of phenol on fluted activated carbon (FPAC) and commercial activated carbon (CAC).
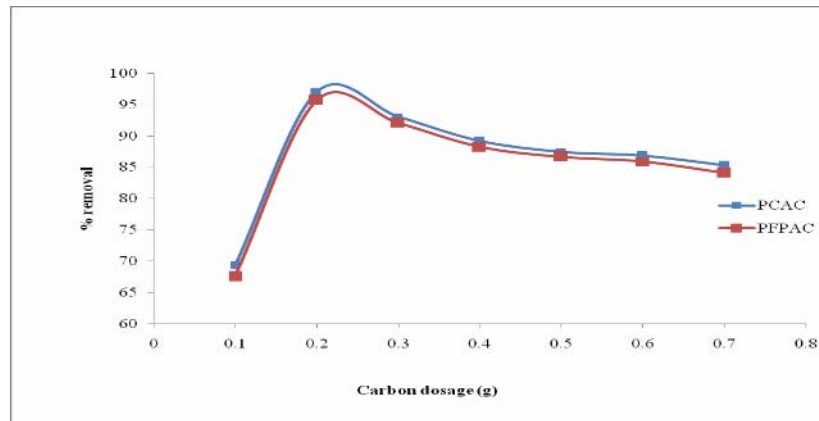
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Figure-6. Effect of adsorbent dosage on % removal of phenol on FPAC and CAC.

Effect of adsorbent dosage

The adsorption of phenol was carried out at different adsorbent dose by keeping other parameters constant. The relationship between adsorbent dose and substrate removal for same initial concentrations of phenolic compounds is presented in Figure-6.

Figure-6 shows that percentage removal of phenol 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 Uddien et al., 2007. The uptake of solute markedly increased up to adsorbent dose of 0.2g and thereafter no significant increase was observed. It can be concluded that the rate of phenolic compounds binding with adsorbents increases more rapidly in the initial stages.

Maximum percentage removal of 97.00% and 95.67% was obtained with 0.2g of FPAC and CAC respectively. Both activated carbons removed over 95 percent of phenol. The results clearly indicate that the removal efficiency increases up to the optimum dosage of 0.2g beyond which the removal efficiency is negligible for both biomass becomes almost a constant.

ADSORPTION EQUILIBRIUM

The Langmuir isotherm treatment

The basic assumption of the Langmuir theory is that adsorption takes place at specific homogenous sites within the adsorbents. It has been assumed that once a sorbate occupies a site, no further sorption can take place at that site until monolayer saturation occurs. 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 modeled using the equation below.

$$\frac{c_e}{q_e} = \frac{1}{q_m k_L} + \frac{c_e}{q_m} \quad\text{.................................(2)}$$

Where q_m and K_L are the Langmuir constants. The plots of specific sorption (C_e/q_e) against equilibrium concentration (C_e) gave the linear isotherm parameters of q_m, K_L and the coefficient of determination and these are presented in Table-2.

The sorption capacity, q_m which is a measure of the maximum sorption capacity corresponding to complete monolayer coverage showed that the phenol on commercial activated carbon had a mass capacity of 31.25mg/g higher than phenol on fluted pumpkin activated carbon 30.30mg/g The adsorption coefficient, K_L that is related to the apparent energy of sorption for phenol (CAC) is (0.220 dm^3/g) is higher than phenol on FPAC (0.264dm^3/g).

The adsorption data for phenol onto activated carbon were analyzed by a regression analysis of Langmuir isotherm model. From Figure-7 the graphs obtained were linear with high regression values for phenol on FPAC 0.985 and phenol on CAC 0.982. The favourable nature of adsorption can be expressed in terms of dimensionless equilibrium parameter of Horsfall and Spiff, 2004. $S_F = 1/(1+k_L C_o)$, where K_L is the Langmuir constant and C_o is the initial concentration of the adsorbate in solution. The dimensionless separation factors calculated for phenol on FPAC and CAC were 0.036 and 0.043, respectively. The values of S_F indicates the type of isotherm to be favorable ($0 < S_F < 1$).$_F$ values were less than 1.

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Table-2. Equilibrium constants obtained from Langmuir, Elovich and Dubinin-
Radushkevich isotherm.

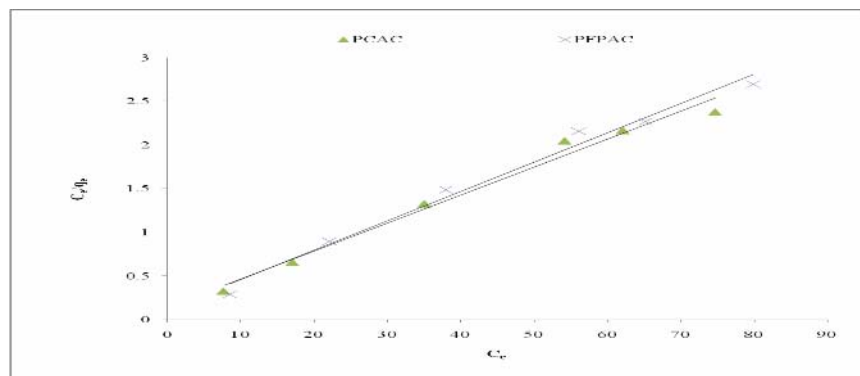| LANGMUIR CONSTANT					
Adsorbate	Adsorbent	q_m(mg/g)	K_L dm^3/g	R^2	S_F
Phenol	FPAC	30.30	0.264	0.985	0.036
Phenol	CAC	31.25	0.220	0.982	0.043
FREUNDLICH					
Adsorbate	Adsorbent	n(L/mg)	K_F (mg/g)	R^2	
Phenol	FPAC	8.77	17.17	0.834	
Phenol	CAC	8.92	17.86	0.846	

Figure-7. Langmuir isotherm plot for phenol removal from aqueous solution by fluted
activated carbon (FAC) and commercial activated carbon (CAC).

Freundlich isotherm

The linearised form of the Freundlich equation is represented by equation (3).

$$logq_e = logK_F + \frac{1}{n}logC_e \quad ......(3)$$

A plot of $logq_e$ against $logC_e$ yeilding a straight line was made to confirm the model. Thevalues of K_F and 1/n were obtained from the intercept and slope of the straight line
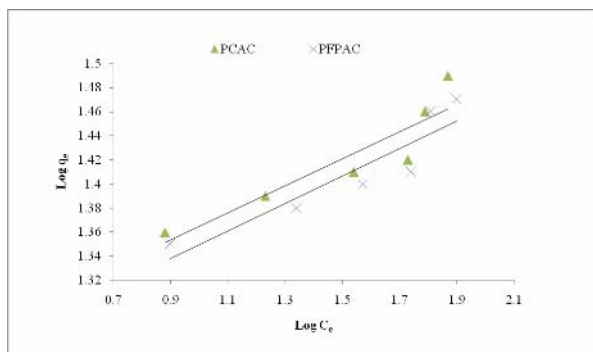

Figure-8. Freundlich equibrium isotherm model for the sorption of phenol onto FPAC and CAC.

From Table-2, it was observed that the intensity of adsorption (n) was higher for phenol on CAC (8.92) than for phenol on FPAC (8.77). The intensity of adsorption values obtained for phenol on FPAC (8.92) and CAC (8.77) are higher than the result of Mahvi et al., 2004 on rice husk (5.13). A favorable adsorption corresponds to a value of 1<n<10. The intensity of adsorption (n) on FPAC and CAC for the sorption of phenol was found to be greater than 1 and less than 10. Therefore the adsorption process as obtained from the Freundlich isotherm model is favorable. The adsorption capacity, K_F, phenol on CAC is (17.86mg/g) and slightly higher for phenol on FPAC (17.17mg/g). The maximum adsorption capacity obtained by Aksu and Yener (2001) on phenol using fly ash was 27.9mg/g at an initial concentration of 100mg/l which is higher than the values obtained in this study for phenol on FPAC (17.86mg/g) and on CAC (17.17mg/g), respectively.

The R^2 values (0.834-0.846) show that the equilibrium data for the adsorption of phenol slightly fitted the Freundlich isotherm in the concentration range (100 – 200mg/l) studied. The regression coefficients (R^2) values as shown in Table-2 indicate that Langmuir isotherm is better fitted for describing the equilibrium adsorption data for phenol than Freundlich isotherm. Similar observations were reported by Aktas and Cecen, 2007.

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CONCLUSIONS

In this study, the adsorption of phenol from aqueous solution was investigated using fluted pumpkin activated carbon and commercial activated carbon as adsorbent. The results indicated that adsorption capacity of the adsorbents was considerably affected by initial pH, initial concentration, contact time, temperature and adsorbent dosage. The results showed that the amount of phenol adsorbed increased with increasing initial phenol concentration. The result also indicated that the uptake of phenol optimized at pH 6 for FAC and CAC. Lower temperature of 30^OC favoured phenol removal. Equilibrium data fitted into the Langmuir isotherm better than Freundlich.

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