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RESEARCH ARTICLE

COMPARATIVE STUDY ON THE ADSORPTION CAPACITY AND KINETICS OF XYLENE ONTO RICE HUSK AND CASSAVA PEEL ACTIVATED CARBON

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ABSTRACT

This study focuses on the comparative adsorption of xylene from aqueous media onto cassava peel activated carbon (CPAC) and rice husk activated carbon (RHAC). Ash content, bulk density, iodine number, moisture content, pH, point of zero charge, and % carbon yield of the prepared CPAC and RHAC were reported. To evaluate the removal of xylene, adsorption activities of CPAC and RHAC were monitored under different experimental conditions: adsorbent dose, contact time, pH as well as initial xylene concentration. The adsorption capacity and kinetics depended on the properties of the activated carbons, the adsorbate and the prevailing adsorption milieu while the equilibrium and rate of adsorption were adequately interpreted by Langmuir and pseudo-first-order models respectively. Batch adsorption kinetic experiments revealed that the adsorption of xylene onto CPAC and RHAC involved fast and slow processes. At optimal conditions of 8 mg/L xylene in contact with 0.1 g of adsorbents for 150 min at pH 7.5, the quantity of xylene adsorbed onto RHAC and CPAC were 46.7 mg/g and 53.3 mg/g in that order. Thermodynamic study showed the adsorption process of xylene onto CPAC and RHAC to be non-spontaneous but ΔH was negative.

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INTRODUCTION

The volume of organic wastes generated in our society and the cost of their disposal has continued to increase, and these pose a challenge to researchers to develop improved, cost effective and environmentally safe disposal and treatment methods (Srivastava *et al.* 2006). Pollution by volatile organic compounds (VOCs) is a major challenge around human habitation in big cities. The toxicity of these organic compounds e.g. (benzene, toluene, xylene, ethylbenzene, formaldehyde and methylene chloride) makes their removal necessary. They have been classified as high priority pollutants by the U.S environmental protection Agency due to their extensive impact on water quality (IARC 1999b). A number of studies have been carried out on the adsorption of xylene and other organics from aqueous media with different submissions. Some authors Chikwe *et al.* 2018; Bedin *et al.* 2013 and Nourmaradi *et al.* 2012 have attempted to sorb xylene and toluene onto modified and unmodified clay minerals. They independently reported a higher percentage removal and adsorption capacity for the modified clays than the raw minerals. This study is aimed at the preparation of adsorbents from cassava peel and rice husk for the comparative removal of xylene from aqueous media by adsorption process. The results of the study will be applied in the removal of xylene from contaminated water bodies.

MATERIALS AND METHODS

Chemicals and Reagents: Orthophosphoric acid, xylene, potassium hydroxide, sodium hydroxide, nitric acid and hydrochloric acid were used as purchased from Sigma Aldrich, USA. The adsorbate solution (xylene) was prepared from stock solution by serial dilution using deionized water. Different concentrations of xylene were prepared for the adsorption studies.

Sample collection and Preparation of activated carbon: The cassava peel and rice husk were collected from a cassava processing unit at Oja-Iya Street, off Taiwo road and Ojagboro, Ilorin, Kwara State, respectively. The washed and dried cassava peels and rice husk samples were placed in crucibles in horizontal tabular carbonizer for carbonization in a

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furnace. At the attainment of the preset temperature, 5 g of prepared cassava peel and rice husk samples were weighed into pre-weighed empty crucibles, at varying temperatures from 50 - 500°C for 20 mins. The pyrolysed samples were cooled and ground into a granular size and then activated (*Nourmoradiet al.* 2012; *Odebunmi* and Okeola, 2001). The charred samples were mixed with 75% concentrated orthophosphoric acid in a beaker with the weight ratio of H_3PO_4 to char sample to be 0.1,evaporated at 60°C for 24 h to get a powdered activated char (Viboon *et al.*, 2008 and Lillo-Rodenas *et al.*, 2005). A 50 mL 1 M KOH was added to the sample for neutralization and stirred for 4 h (Odebunmi and Okeola, 2001). Subsequently, the mixture was washed several times with deionised water and dried at 105 °C in an oven. The carbonized samples were poured into a beaker containing 250 mL of 0.1 M HCl for purification and stirred for an hour according to Li and Wang 2009.

Adsorption studies: The adsorption experiment was carried out by batch test in 50 mL flasks under vigorous agitation at 80 rpm. In each of the experimental set up, 25 mL of xylene solution of known initial concentration (C_0), desired pH adjusted using .0.1 M HCL and 0.1 M NaOH solutions by a pocket pH meter. A 0.5 g of CPAC and RHAC (adsorbent dosage) was added to the 250 mL conical flask with a stopper was agitated in a temperature-controlled shaker for a predefined time of 2 hrs. After the contact time, samples were centrifuged and a quantity was taken and analyzed for the residual concentrations by Du'730 life science UV/Vis spectrophotometer at λ_{max} of 290 nm (Lillo-Rodenas *et al.* 2005).

The quantity adsorbed was obtained from equation 1

$$q_e = \frac{V(C_o - C_e)}{w} \tag{1}$$

Where C_o and C_e are the initial and equilibrium liquid phase concentrations (mol/dm^3) respectively, V is the volume of solution (ml), and w is the weight of dry sorbents (g) (Li and Wang 2009).

Adsorption Isotherm: The results of the effect of the initial xylene concentration study were used to elucidate the relationship between concentration and the amount of adsorbate adsorbed per unit mass by means of Langmiur and Freundlich isotherm models. Langmuir isotherm (eqn 2) assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface while Freundlich isotherm (eqn 3) predicts a homogeneous distribution of sorption energies and the heterogeneity of adsorption(Low *et al.* 1995 and (El-Shafey 2005).

$$\frac{C_e}{q_e} = \frac{1}{kq_m} + \frac{C_e}{q_m} \tag{2}$$

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \tag{3}$$

Where q_e is the amount of adsorbate adsorbed at equilibrium (mg/g), k is the Langmuir constant related to the energy of adsorption (l/mg), q_m is the maximum sorption capacity corresponding to complete monolayer coverage (mg/g), and Ce is the equilibrium solute concentration (mg/l). K_f represents the sorption capacity when equilibrium concentration equals to 1, and n represents the degree of dependence of sorption with equilibrium concentration.

Kinetic Models: The dependence of time on the adsorption experiment was studied by the analysis of the adsorption data with pseudo first and pseudo second models (4 and 5) respectively(El-Shafey 2005).

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{4}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{5}$$

Where q_e and q_t are the amounts of adsorbate (xylene) adsorbed (mg/g) at equilibrium and any time t, respectively, and k_1 (min^{-1}) is the rate constant of the pseudo first-order adsorption operation. Similarly, k_2 is the pseudo second order rate constant.

RESULTS AND DISCUSSION

Optimization of Physico-chemical Parameters: The percent yield of carbonization for RHAC and CPAC at 400 °C were 22.600±0.100 and 2.440±0.100 % respectively. The moisture content of the samples RHAC and CPAC ranged between 6.390±0.030 and 7.740±0.055% (Table 1). It has been observed that a high moisture content leads to low adsorption capacity which could necessitate the utilization of extra load of the adsorbent (Cheng and Tsai 2007). The moisture content of CPAC and RHAC were 2.790 % and 6.620 % respectively, similar to reports of El-Shafey2005 and Eliagwu *et al*, 2009 and it indicated that the adsorptive capacity of the adsorbents is fairly good. It can be denoted from table 1that high moisture content observed in CPAC (7.740 ±0.055 %) makes it less effective for adsorption compared to that of RHAC (6.390±0.030 %).

The iodine number for RHAC and CPAC were 241.230±0.120 and 63.530±0.080 mg/g respectively. It is the relative indicator of porosity in a carbonaceous material and may be used as an approximation of surface area for some types of carbons (Eliagwu *et al*, 2009; Adowel *et al.* 2012). The results indicate that CPAC with iodine number of (63.530±0.120 mg/g) has higher activity level and higher degree of micropore than that from RHAC (241.230±0.080 mg/g) which informed the consequent higher adsorption capacity of the CPAC and its in consonance with the reports of Malik 2002 and Aziz*et al.* 2009 (Malik 2002; Aziz 2009). The pH of adsorbent is the degree of acidity or basicity of that adsorbent and this depends on the number of factors which include preparation methodology, inorganic matter content, as well as the surface properties of the adsorbents (Baccar *et al.* 2009). The pH of solution after mixing RHAC and CPAC with de-ionized water was 7.1 ±0.1and 7.7±0.1, respectively. As shown in Table 1, the points of zero charge values were 8.9 and 9.2 for RHAC and CPAC respectively. According to Mapoungand Nogklai2008, anions adsorption will be favoured at pH value lower than the adsorbent's point of zero charge and this supports the high adsorption of the xylene onto RHAC and CPAC (Mapoung and Nogklai 2008). Ash reduces the overall activity of the adsorbent, and the efficiency of reactivation. The ash content of adsorbents increases with decrease in percentage of volatile matter as reported by Soleimani and

Table 1. Physico-chemical properties of CPACand RHAC

Parameters	CPAC	RHAC
Ash content (%)	9.650±0.255	12.590±0.460
Bulk density (g/m$^{3)}$	0.254±0.012	0.476±0.006
Iodine number(mg/g)	241.2300±0.008	63.53±0.120
Moisture content (%)	7.740±0.055	6.390±0.030
Ph	7.700±0.100	7.100±0.100
Particle size(mm)	0.250	0.250
Point of zero charge	8.900	9.200
% Carbon yield	2.440±0.100	22.600±0.100

Table 2. Elemental composition of RHAC and CPAC

Elements	RHAC (mg/L)	CPAC (mg/L)
K	1.811±0.81	3928±67.4
Ca	0.861±0.22	2811±14
Ti	14.2±1.1	101.8±10.2
Cr	0.003±0.002	11.11±2
Mn	0.199±0.06	71.1±8
Fe	0.7011±0.0143	2.2011±0.2918
Ni	3.11±3	52.1±9.2
Cu	231.8±5 . 7	102.2±11
Zn	0.411±0.003	77.2±8
Na	0.021±0.005	Nd
Sr	0.005±0.001	1.11±0 . 7
Se	0.0023±0.001	3.12±0 . 2
Zr	0.021±0.01	ND
As	ND	2.11 ±0 . 1

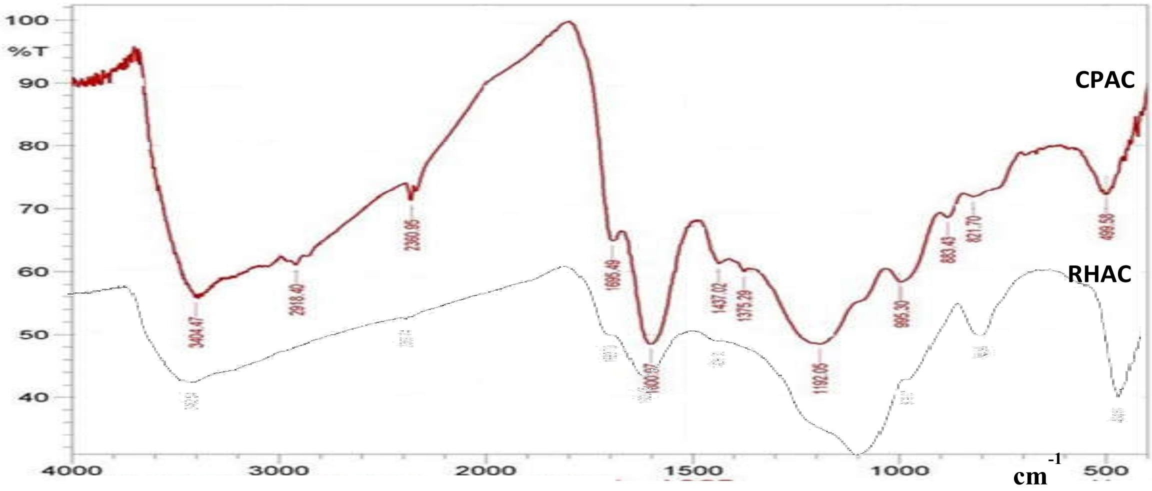

Fig 1. FTIR spectrum for RHAC and CPAC

Table 3. Equilibrium constants obtained for xylene adsorption Langmuir and FreundlichIsotherm

	Langmiur Constant				*Freundlich Constant*		
Adsorbent	*q_m (mg/g)*	*K_L (L/g)*	*R^2*	*R_L*	*1/n (L/mg)*	*K_f (mg/g)*	*R^2*
RHAC	*0.146*	*-3.32*	*0.854*	*-0.039*	*-0.704*	*1.127*	*0.653*
CPAC	*0.086*	*-4*	*0.886*	*-0.032*	*-0.784*	*1.584*	*0.645*

Table 4. Kinetic Parameters for the sorption of xylene

	Kinetic Constant of Pseudo-first Order				Kinetic Constant of Pseudo-second Order		
Adsorbent	q_e (mg/g)	k_1(min^{-1})	R^2	q_e (mg/g)	k_2 (g/mg)	h (mg.g^{-1}. Min^{-1}	R^2
RHAC	2	0.0032	0.9952	2	0.0023	0.0092	0.972
CPAC	1.584	0.0029	0.996	1.593	0.0025	0.0063	0.874

Table 5: Values of thermodynamic parameters for the equilibrium adsorption of xylene on RHAC and CPAC

Adsorbent	ΔH° (kJ/mol)	ΔS° (kJ/molK)	ΔG° (kJ/mol)				
			303K	313K	323K	333K	343K
CPAC	-76821.36	-267.96	4370.52	7050.12	9729.72	12409.32	15088.92
RHAC	-98271.48	-331.2	2082.12	5394.12	8706.12	12018.12	15330.12

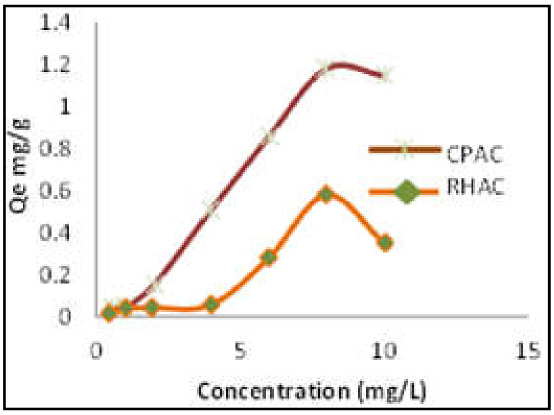

Fig. 2. Effect of initial conc. of xylene onto CPAC and RHAC

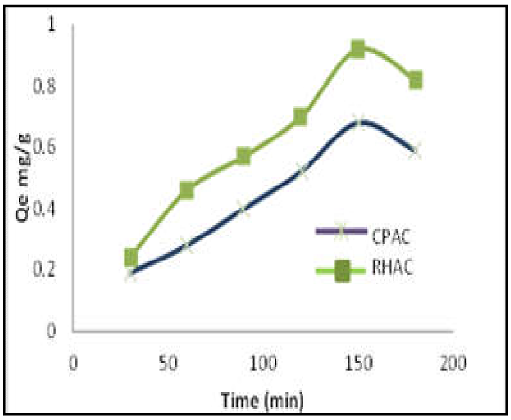

Fig. 3. Effect of contact time on xylene adsorption onto CPAC and RHAC

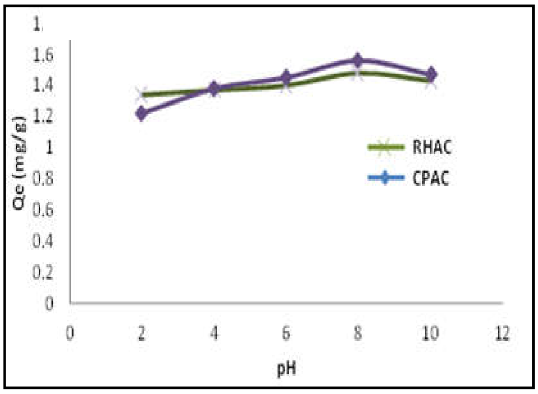

Fig. 4. Effect of pH adsorption of xylene onto RHAC and CPAC

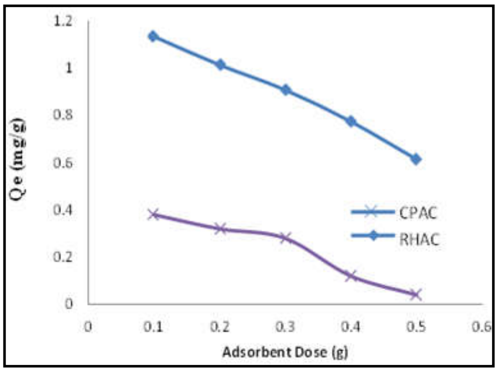

Fig. 5. Effect of adsorbent dosage on xylene adsorption onto CPAC and RHAC

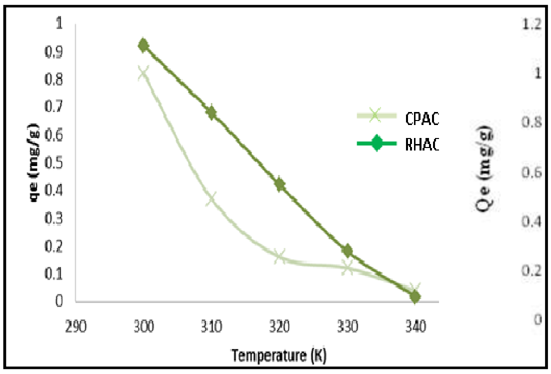

Fig. 6. Effect of temperature on the adsorption of Xylene on RHAC and CPAC

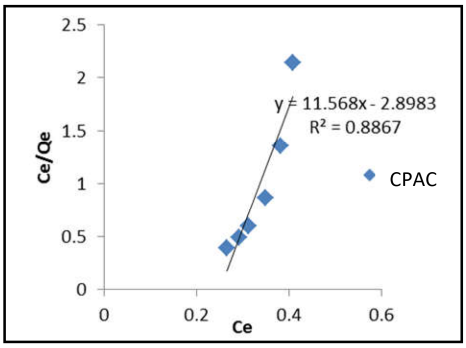

Fig 7: Langmuir isotherm for xylene adsorption onto CPAC

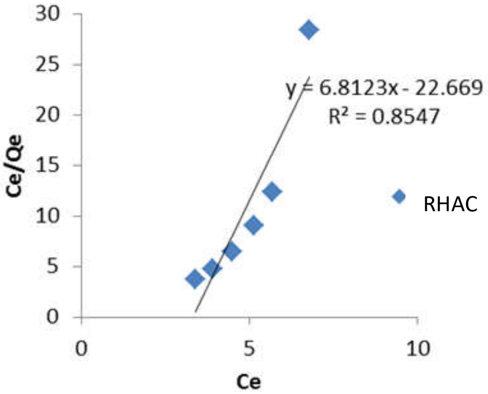

Fig 8. Langmuir isotherm for xylene adsorption onto RHAC

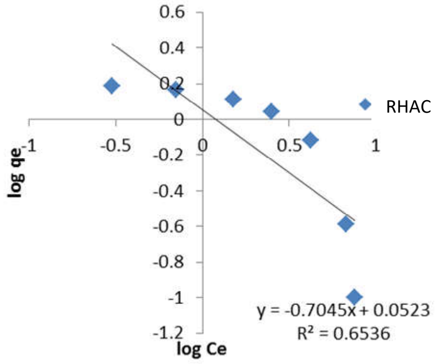

Fig. 10. Freundlichisotherm for xylene onCPAC

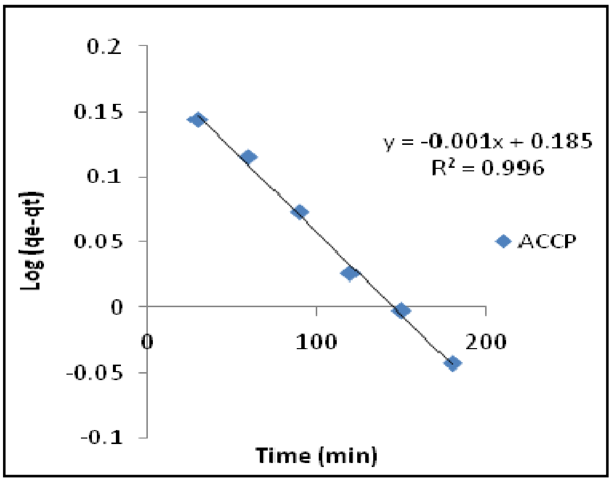

Fig. 11. Pseudo first -order plot for Xylene on CPAC

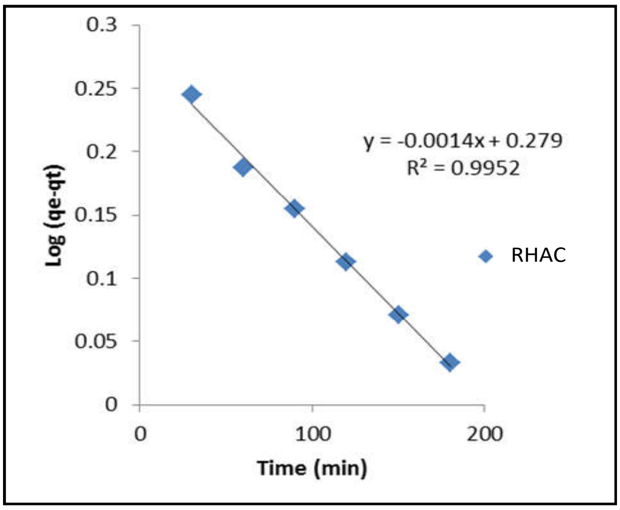

Fig. 12. Pseudo first -order plot for Xylene on RHAC

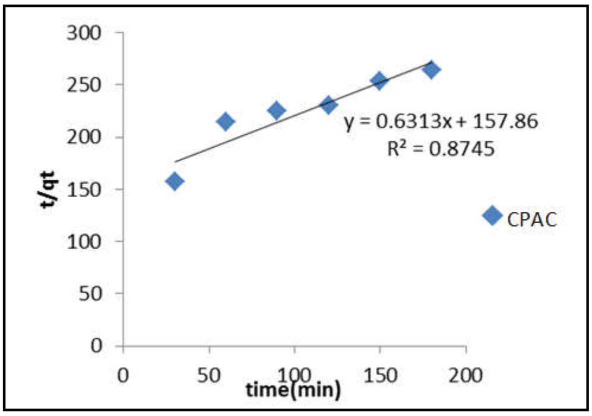

Fig 13. Pseudo second-order plot for Xylene onCPAC

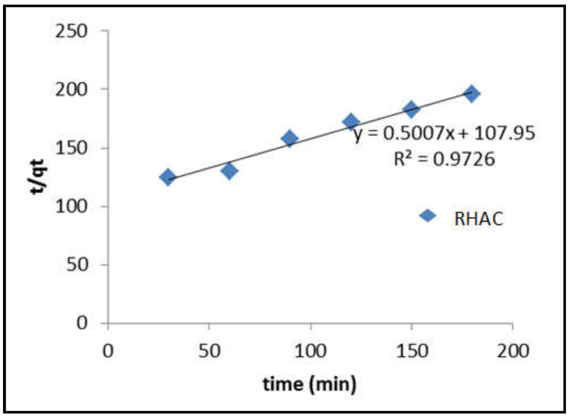

Fig. 14. Pseudo second-order plot for Xylene onRHAC

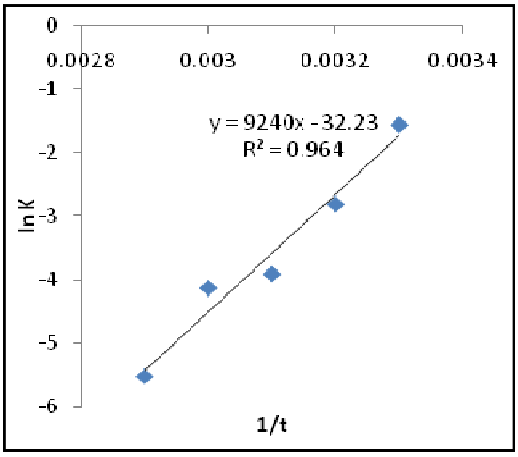

Fig 15. Thermodynamic plot for xylene onto CPAC

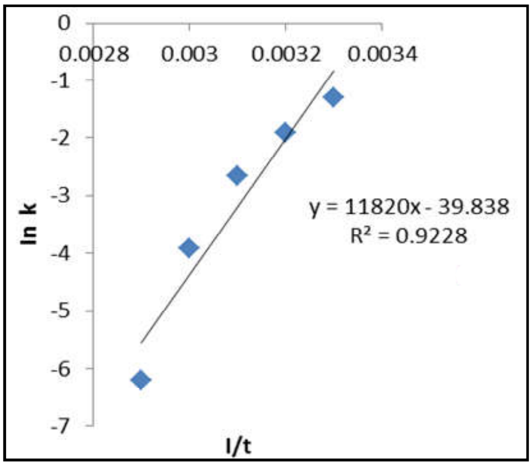

Fig 16. Thermodynamic plot for xylene onto RHAC

Kaghazchi, 2008 and this trend was observed in this study. The percentage ash contents of RHAC and CPAC were 12.590±0.460 and 9.650±0.255, respectively as shown in Table 1 and the lower ash content of CPAC further buttresses the higher adsorption capacity of CPAC over RHAC. Bulk density is an important parameter when carbon is removed by filtration because carbons with adequate density help to improve the filtration rate by forming an even cake on the filter surface. The bulk densities of RHAC and CPAC were 0.476±0.006 and 0.254±0.012 g/mL, respectively and these results were in accordance with the American Water Work Association (AWWA) limitations values (AWWA, 1991). The elemental analysis of the samples by XRF as shown in table 2, the major elements present in both adsorbents are K, Ca, Fe, Ti, Ni, Cu and Zn. The higher concentration observed in CPAC is likely due to the nature of the soil the cassava was grown which receives sewage sludge as soil conditioner.

This is not the case with the rice which was grown on a different soil type (Aksu and Yener2001).

Fourier Transform Infra-Red Spectroscopy (FTIR): The chemical structure of the adsorbent is of vital importance in understanding the adsorption process. The adsorption peak at 3417.98 cm^{-1} indicates the existence of free hydroxyl groups in CPAC and RHAC. The C-H stretching vibration at 2928.04 cm^{-1} shows the presence of alkanes. The peaks at the range 1653.05-1689.70 cm^{-1} corresponds to the C=O stretching that was attributed to the hemicelluloses and lignin aromatic group (Baccar *et al.* 2009).The C=O stretching vibrations between 1743.71-1737 $cm^{-1}$is indicative of ester functional groups. The peaks at the range 1456.3 -1373.36 cm^{-1} indicate the presence of CH_2 and CH_3 groups while the peak at 1380 cm^{-1} are indicative of CH_3 (AWWA) (1990). These peaks 1240.27, 1192.05, 1080.17 and 1093.67 cm^{-1} correspond to CHOH stretching, Si-O-Si stretching and Si-H groups respectively. The presence of polar groups on the surface is likely to give considerable cation exchange capacity to the adsorbent as reported by Inamullah *et al.* 2008 (Inamullah *et al.* 2008).

Effect of Initial Concentration on the Adsorption of xylene: It was observed that as the concentration increases there was a corresponding increase in the quantity of xylene adsorbed up to 8 mg/L. The sorption starts to reduce due to the accumulation of xylene particle on the surface of the adsorbents. The quantity of xylene adsorbed favours CPAC compared to the RHAC. This observation is in line with the findings of Mahvi *et al.* 2004 (Mahvi *et al.*, 2004).

Effect of Contact Time on the Adsorption of xylene: The adsorption of xylene by CPAC and RHAC sample was rapid for 30minas a result of the available binding sites on the sample. The adsorption at 150min was characterized by fast adsorption; the portion between 150-180 min was characterized by slow adsorption which may be due to saturation of the pores as reported by Ho *et al.* 2002 (Ho *et al.* 2002).

Effect of pH on the Adsorption of xylene: The rate at which the adsorption takes place is most important, especially when designing batch sorption system. After pH 8, there was a decrease in adsorption, this decrease may be due to formation of insoluble hydroxyl complexes. Shukla *et al.*2001 reported that at low pH values the surface of adsorbent would be closely associated with hydroxonium ions (H_3O^+), by repulsive forces, to the surface functional groups, consequently decreasing the percentage removal of volatile organic compounds. CPAC has the highest quantity adsorbed (1.56 mg/g) for xylene at a pH value of 7.7 compared to RHAC has (1.48 mg/g) pH of 7.1

Effect of Adsorbent Dose for xylene: As evident from fig 5, the amount of xylene adsorbed decreased with increase in adsorbent dose. This could be due to aggregation, resulting from high adsorbent dose. Such aggregation would lead to decrease in surface area of the adsorbent and an increase in diffusion path length as reported by Sanchez *et al.* 1999. RHAC has the highest quantity adsorbed for xylene (1.13mg/g) at adsorbent dose of 0.1gcompared to CPAC that has the quantity adsorbed for xylene to be 0.38mg/g.

Effect of Temperature on the Adsorption xylene: The result of effect of temperature on the adsorption of xylene decreased with increase in the temperature for RHAC and CPAC as shown in fig.5, this indicates the exothermic nature of the adsorption process (Shukla *et al.* 2001).

Adsorption and kinetic models: The Langmuir graphs depicting the adsorption behviour of xylene onto CPAC and RHAC are shown in Fig.7 and 8 while extrapolated parameters are shown in Table 3. Equilibrium constant (R_L) values from table 3 shows that the adsorption behavior of RHAC and CPAC were extremely favorable (R_L<1) due to the low values of R_L as strongly opined by Allen and Brown (Allen and Brown 1995). The Freundlich plots for the Xylene adsorption by RHAC and CPAC were given Figure 9 and 10. It can be deduced from Figs. 7, 8, 9 and 10 that Langmiur isotherm adequately explains the adsorption process better for RHAC and CPAC. It involved the formation of a monolayer adsorption surface containing a finite number of adsorption sites of uniform strategies. Evidently, the adsorption data fitted more to the pseudo-first order model with a regression coefficient of 0.9952 and 0.996 for RHAC and CPAC, respectively. This is depicted in Figs. 10, 11 and Table 3.

Thermodynamic Studies: The data show that the equilibrium adsorption decreases with increase in temperature (Table 5). It indicates that higher temperature counters the adsorption efficiency. The thermodynamic parameters: Gibbs free energy (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were calculated to evaluate the nature of the adsorption process. The magnitude of ΔG° (kJ/mol) was calculated using equation 8:

$$\Delta G^o = -RT\ln K_a \qquad (8)$$

$$\Delta H^0 = \Delta G^0 + T\Delta S^0 \qquad (9)$$

Xylene interaction with RHAC and CPAC is exothermic owing to the negative value of ΔH°. The values of ΔG were positive accounting for a range of 4370.52 - 15088.92 kJ/mol for CPAC and the ΔG value for RHAC is in the range of 2082.12 - 15330.12 kJ/mol for the adsorption of xylene. The positive value of ΔG shows that the reaction is non-spontaneous. The trend of the thermodynamic plot is in consonance with reports by Shukla *et al.*, 2001 and McKay 1996.

Conclusion

The adsorption capacity and kinetics depended on the properties of the activated carbon and the adsorbate as well as experimental conditions. Langmiur isotherm and pseudo-first order kinetics adequately explained the adsorption behavior and rates respectively of both adsorbents. The adsorption kinetic experiments revealed that the adsorption of xylene onto CPAC and RHAC involved fast and slow processes. The CPAC was a better adsorbent than RHAC based on the prevailing experimental conditions.

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