

## Activated Orange Meso-Carp Carbon (AOMC); An Acceptable Remediation Techniques for Crude Oil Pollution Effect

<sup>1</sup>Atulegwu Patrick Uzoije, <sup>2</sup>Luke O. Uzoigwe and <sup>3</sup>C.I.O. Kamalu

<sup>1</sup>Department of Environmental Engineering, Federal University of Technology Owerri  
PMB, 1526 Owerri, Nigeria

<sup>2</sup>Department of Agricultural Engineering, Imo state University, Owerri, Nigeria

<sup>3</sup>Department of Chemical Engineering, Federal University of Technology, Owerri, Nigeria

**Abstract:** Orange mesocarp with its potentials application to remove spilled crude oil was used to prepare an activated adsorbent. Therefore, adsorption of crude oil onto the activated orange meso-carp (AOMC) was investigated. Batch experiment was adopted for the equilibrium studies and the studies were conducted for various operational parameters such as varying crude oil concentration and temperature values. The crude oil samples of A, B, C, D and E, with concentrations 6045, 4393, 8508, 11583, and 5220, respectively and temperature values varied between 10-50°C were used for the experiment. The adsorption equilibrium was established at 40 min of adsorption time. Partition coefficients,  $k_d$  (L/kg) for various samples reacted inversely with temperature and were in the ranges of 0.37-0.69, 0.65-1.11, 1.28-2.04, 1.17-1.39 and 1.23-4.53 for samples A, B, C, D and E, respectively. percentage of crude oil samples on the AOMC decreased with low crude oil hydrocarbons and the trend was shown as follows  $D > C > A > E > B$ . Percentage ranges for samples A, B, C, D and E were given to be 86.8-88%, 60.5-72.4, 52.5-55.2, 47.9-55.4 and 45.3-49.2%, respectively. Sorption of the crude oil samples to AOMC was found to be spontaneous, exothermic, and physio-sorption controlled with G values being in the ranges of -71.49 to -21.29, -18.25 to -5.43, -30.24 to -21.11, -28.55 to -11.33 and -50.74 to -50.12 KJ/mol for samples A, B, C, D and E, respectively. Enthalpy(H) and Entropy changes(S) were also found to be -10.28, -12.24, -8.92, -6.9, -50.28 J/mol/K and -2.92, -0.32, -0.50, -0.44, -0.01 for samples A, B, C, D and E, respectively. Activated orange meso-carp provides a veritable environmentally remediation technique for crude oil spillage.

**Key words:** Crude oil, orange meso-carp, partition coefficient, sorption, thermodynamics

### INTRODUCTION

Crude oil pollution effects found in soil, water and air environments continued to be a major issue of scientific interest, political and public concern. Crude oil alters the physio-chemical properties of the soil (Gesinde *et al.*, 2008). Soils polluted with crude oil experience instant loss of fertility due changes in Ph and other physiochemical properties, with its concomitant influence on the solubility and abundance of micro-nutrients (Ogaji *et al.*, 2005; Onweremadu and Eshett, 2007). Jobson *et al.* (1974) observed glaring imbalance of carbon-nitrogen ratio in an oil-soaked soils. Further more, large concentration of crude oil on the soil surface depletes and slows down oxygen diffusion in the lower horizons limiting robust agricultural usage of the soil (Molnaa and Grubbs, 1989). In aquatic environment, different aquatic species have varying sensitivity to crude oil pollution in water. However, the crude oil spill in an aquatic environment floats and blocks out sunlight initiating the death of Phytoplankton and seaweed, sources of food and

indeed the chief sources of food chains, hence starving life under the spill (Fominyen, 2010). Johnson *et al.* (2000) observed that fishes whose growths are retarded by aggressive environment occasioned by crude oil spill pose serious health problems to consumers. In most cases death of large aquatic lives such whales occurs due to crude oil coverage of their blow holes. It attaches to the fur of otters and other animals with fur or feathers. Crude oil soaks the feather and fur of the animals, destroying their protective coats and subsequently causing their death due to severe cold (Fominyen, 2010). Al-Hashem and Brain (2009) also observed that lizards living in crude oil polluted environment experienced reduced body size due to lack of preys with high level of fat occasioned by the pollution. Crude oil has become one of the most frequently detected underground water pollutants caused by leakages from underground storage tanks, Pipelines and other components of crude oil distribution systems (Kharoune *et al.*, 2001): In view of the aforementioned, there has been a growing concern regarding the treatment of water and wastewater in recent years. Generally, the

conventional method of water and wastewater treatment technologies including flocculation, ultrafiltration, biological treatment and coagulation (Suidan *et al.*, 2005; Ayotamuno *et al.*, 2006; Guipeng *et al.*, 2006; Nilsson, 1971) do not seem to be economically viable. To forestall the glaring difficulties in applying remediation techniques mentioned above it is pertinent to apply sorption process as convenient remediation measure. Several research works have shown that removal of crude oil from the environment is predominantly through sorption onto solid (Uzoije, 2008a; Filho *et al.*, 2010; Buckley and Liu, 1998; Uzoije and Agunwamba, 2011). The sorption behavior of crude oil onto solids is to a large extent predicted by partition coefficient (Ifelebuegu *et al.*, 2010). Understanding of partitioning of crude oil between the solids and aqueous phase and the sorption techniques is important to know the fate and the route of this substance into the environment. (Bennett *et al.*, 2003; Rixey *et al.*, 1999; Bennett and Larter, 1997) examined the partition coefficient of a number of organics including hydrocarbons onto solids. Bennett and Larter (1997) observed that Partition behavior of alkylphenols in crude oil/brine systems under subsurface conditions was measured under three basic conditions; pressure (25-340 bar), temperature (25-150°C), and water salinity (0-100,000 mg/L sodium chloride) for a variety of oils. Their further findings showed that Partition coefficients for all compounds decreased with increasing temperature, increased with water salinity and crude oil bulk content, and showed little change with varying pressure. (Lafrance *et al.*, 1991) found that log  $K_a$  of aromatic hydrocarbons on powdered activated carbon of high acidic range is less than 4. Orange meso-carp is rich in cellulose and are used as binder and raw materials for several production processes (Ejikeme, 2008). Various research studies have shown that cellulose extracted from orange meso-carp has served as raw materials for pharmaceutical and food industries due to its good binding and sorption qualities (Cuevas *et al.*, 2010; Akaranta and Amadi, 2000; Soh *et al.*, 2008; Bhalekar *et al.*, 2010).

This study examined the partitioning of crude oil in the activated orange mesocarp and its sorption thermodynamics.

## MATERIALS AND METHODS

**Activation of orange meso-carp:** The study was carried out between the year 2009 and 2010 in the Laboratory of the Chemistry Department of Federal University of Technology Owerri. 2500 of fresh orange meso-carp collected from the waste bin of the orange vendors on the street of owerri was washed and pulverized at the temperature of between 80-1000°C for 2 h and 30 min. The pyrolysed orange meso-carp was carefully meshed into powder and sieved using sieve of 1.18 mm to get

particles of uniform size. 200 g of the well sieved and pyrolysed material was transferred into a beaker containing 150 mL of 0.1 M hydrochloric acid and mixed until the mixture formed a paste. The paste was then transferred into a dry crucible and oven dried for 1 hour at 150°C before it was heated in muffle furnace at 800°C for 2 h to increase its surface area. After which, it was washed with a distilled water to bring its PH range to 7 and oven dried for 3 h to get an Activated Orange Meso-Carp based carbon (AOMC).

**Characterization of the adsorbent:** PH of the activated adsorbent (AOMC) was determined by dissolving 2.5 g of the activated adsorbent into 50ml of distilled water. The mixture was heated and stirred adequately for some minutes to ensure proper dilution of the sample. The mixture was filtered and the pH of the clear solution was read using digital pH meter. The particle size of the adsorbent was determined by adopting ASTM D2862. Bulk density, moisture content, ash content, pore volume and porosity were determined using the methods adopted by (suidan *et al.*, 2005).

**Characterization of the adsorbate:** Five crude oil samples denoted as samples A, B, C, D and E were used as adsorbates. The crude oil samples were collected from five different oil wells in Niger-delta, south-south region of Nigeria name; Escravous, Bonny light, Que Iboe, Brass and Forcados crude oils, respectively. Their Total Hydrocarbon Concentrations (THC), density, specific gravity, viscosity and refractive index were analyzed using standard methods.

**Adsorption isotherm study:** Batch experiment was adopted for the isotherm study. Crude oil samples of five different initial concentrations were put in five sealed bottles containing fixed amount of the AOMC (1 g/L) (each bottle for one sample). The bottles were agitated by a rotary shaker at 180 rpm for five days to attain equilibrium. Each experiment was carried in three replicates. After the agitation, samples were withdrawn at fixed intervals of 10 min for 70 min and centrifuged. The solid phase crude oil concentrations were determined using mass balance. The experiment was repeated at varying temperatures of 10, 20, 30, 40 and 50°C. The amount adsorbed by the activated adsorbent which was aimed at assessing the equilibrium capacity of the adsorbent was determined by the difference between initial concentration and the equilibrium concentration of the THC in solution as shown in this relationship (Chen *et al.*, 2010):

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

The THC/aqueous partition coefficients (Kd L/kg) for each crude oil sample were calculated by this relationship (Ifelebuegu *et al.*, 2010):

$$Kd = \frac{C_s}{C_{aq}} \quad (2)$$

where,  $C_s$  (mg/g) represents the concentration of THC adsorbed by the activated adsorbent (AOMC) and  $C_{aq}$  (mg/L) Concentration of THC in aqueous phase.

Percentage of THC adsorbed onto the adsorbent was calculated with the following relationship:

$$\% \text{ of THC onto AOMC} = \frac{C_s}{C_{total}} \times \frac{100}{1} \quad (3)$$

where,  $C_s$  = concentration of THC on the adsorbent;  $C_{total}$  = concentration of the total THC in various crude oil samples i.e., in both the aqueous and solid phase.

**Thermodynamics of the sorption process:** It is important to determine the heat energy losses or gains in the adsorption process. It determines the extent of sorption and partitioning of the adsorbate onto the adsorbent at varying temperature ranges (Piatt *et al.*, 1996). Two basic variables mainly explain the thermodynamic properties of sorption processes. (Ifelebuegu *et al.*, 2010) enumerated such properties as enthalpy (H) and entropy (S). To determine these variables a plot of  $\ln Kd$  and  $1/T$  was made and its slope and intercept values explained the enthalpy and entropy respectively. The relationship is as expressed in Eq. (4):

$$\ln Kd = - \left[ \frac{\Delta H}{R} \right] \left[ \frac{1}{T} \right] + \frac{\Delta S}{R} \quad (4)$$

where, H and S are as explained above, T and R are the absolute temperature and gas constant 8.314 J/mol/k respectively. The Gibb free energy of the adsorption process was computed using the following relationships.

$$\Delta G = \Delta H - T\Delta S \quad (5)$$

$\Delta G$  represents the Gilbb free energy while other paramenters are as defined in Eq. (4).  $\Delta G$  expresses the driving force in a chemical reaction. At constant temperature and pressure,  $\Delta G$  represents the change in internal energy per unit mass and measures the reaction capability (Aneke, 2008). The Gibb free energy was determined at different sorption temperature ranges using

Table 1: Physiochemical properties of orange mesocarp

Parameters	Values
$\alpha$ -Cellulose	58.9%
$\mu$ -Cellulose	27.5%
Ash (g/g)	$2.7 \times 10^{-4}$
Density (g/cm)	0.9
Moisture Content (%)	0.511
pH	5.8
Pore vol. (mL)	1.044
Posrosity	0.45

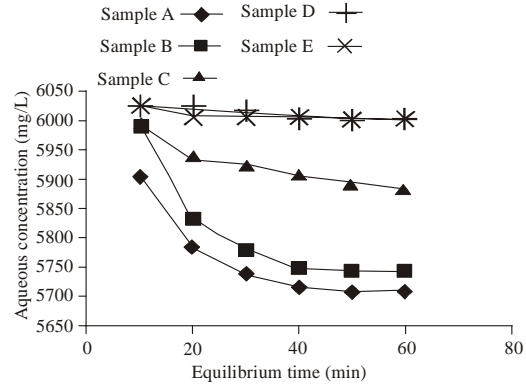


Fig 1: Determination of equilibrium time various crude oil concentrations

the enthalpy and entropy values derived from the slope and intercept respectively of the plot of the relationship in Eq. (4).

## RESULTS AND DISCUSSION

**Adsorbent and adsorbate characterization:** Table 1 shows the proximate analysis of the adsorbent (AOMC). The analysis shows that cellulose constitutes a large component of orange meso-carp with  $\alpha$ -cellulose having the highest value. However, the physiochemical results of orange meso-carp as presented in this study are in line with the observation of (Ejikeme, 2008). The high cellulose content of orange meso-carp provides its binding ability to other substances, making it a veritable raw materials to most pharmaceutical industries and perhaps a potential adsorbent (Cuevas *et al.*, 2010; Akaranta and Amadi, 2000; Soh *et al.*, 2008; Bhalekar *et al.*, 2010) . The high sorption ability of (AOMC) observed in this study corroborated this observation. For the characterization of the adsorbate, five crude oil samples which include; Escravous, Bonny light, Que Iboe, Brass and Forcados crude oils, were carried out with the following results; sample A= 6045 ppm, sample B= 4393 ppm, sample C= 8508 ppm, sample D = 11593 and sample E = 5220 ppm. From the results, sample D has the highest THC values followed by sample C with the least value ascribed to sample B. This implies that sample D has the highest amount of hydrocarbons perhaps highest

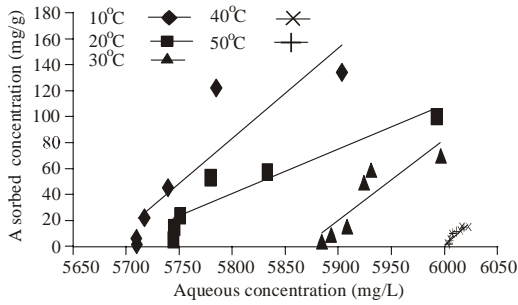


Fig. 2: Crude oil A Aisthem for activated orange meso-carpat varying temperature

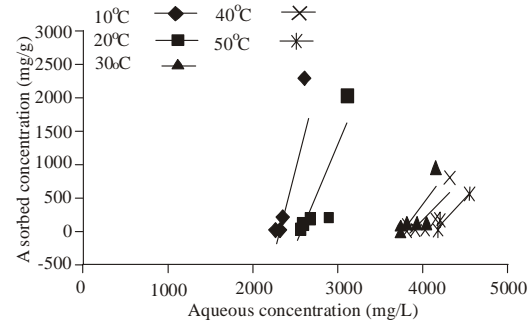


Fig. 6: Crude oil sorption isotherm for the active orange mesocarpat at varying temperature for the determination of partititon coefficient

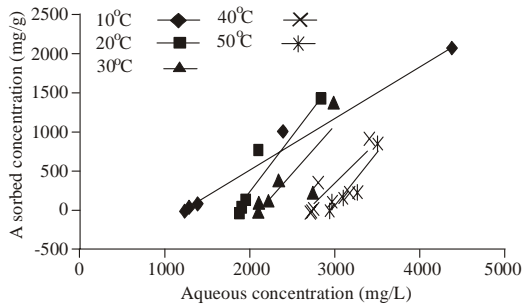


Fig. 3: Crude oil B Soption for the activated orange mensocarp at varying

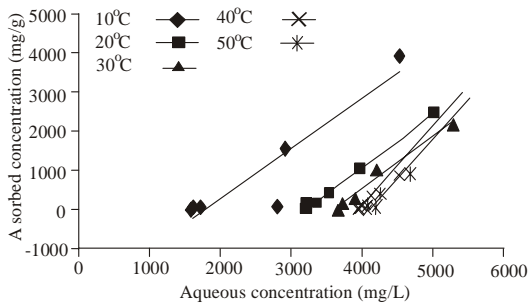


Fig. 4: Crude oil C isotherm for activated meno carp at varying temperature

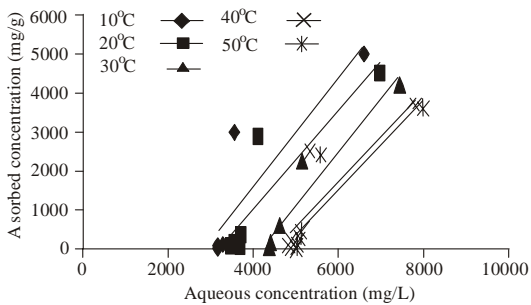


Fig. 5: Crude oil D isotherm orange mesocarp at varying temperature

viscosity and density values as equally observed in the studies of Uzoije (2008b), Uzoije *et al.* (2010a, b).

**Adsorption isotherm and partition coefficient:** Sorption equilibrium of various crude oil samples in aqueous phase was established at virtually 40minutes of sorption period after which adsorption of the adsorbates remained constant as shown in Fig. 1.

Figure (2-6) represent the crude oil sorption isotherm for the activated orange meso-carp at varying sorption temperature ranges. The Figures show the sorption isotherm for crude oil samples A, B, C, D and E, respectively. It can be deduced from the result that sorption isotherm curves for the samples tend to follow a linear curve.

This implies that the partitioning of the crude oil samples between aqueous and solid phase assumed linear isotherm as corroborated by the results of several studies ( Ifeiebuegu *et al.*, 2010; Chen *et al.*, 2010 and Ternes *et al.*, 2004). The results equally show that linear regression model similar to freundlich isotherm described the crude oil sorption onto the activated orange meso-carp . The Freunlich model description of Jackfruit peel Nanoporous adsorbent for removal of Rhadamine Dye as presented by Jayarajan *et al.* (2011) also corroborated this study. The partition coefficients and correlation coefficients of the linear regression model for different crude oil samples at varying sorption temperature values is presented on Table 2

Coefficient of correlations ( $R^2$ ) of the plots for crude oil samples A-E at the sorption temperature ranges of between 10-50°C were shown to be between 0.6-0.994. The coefficient of correlation ( $R^2$ ) at 5% significant level for various crude oil samples at varying sorption temperature seems to have the maximum values of between 0.85-0.99 for crude oil sample C followed by sample B with sample D recording the lowest value. Previous studies corroborated the observation (Kadirvelu *et al.*, 2001; Uzoije *et al.*, 2010a, b). Figure 2, 3, 4, 5 and 6 show the

Table 2: Partition coefficient for crude oil samples at varying sorption temperature value

0°C	A		B		C		D		E	
	Kd (L/kg)	R <sup>2</sup>	Kd (L/kg)	R <sup>2</sup>	Kd (L/kg)	R <sup>2</sup>	Kd (L/kg)	R <sup>2</sup>	Kd (L/kg)	R <sup>2</sup>
10	0.698	0.79	1.164	0.98	2.041	0.99	1.399	0.77	4.532	0.62
20	0.635	0.88	1.142	0.90	2.017	0.98	1.370	0.92	2.980	0.77
30	0.623	0.77	1.345	0.69	1.369	0.98	1.264	0.80	1.658	0.61
40	0.610	0.92	1.049	0.75	1.353	0.99	1.179	0.77	1.428	0.96
50	0.347	0.73	0.645	0.89	1.281	0.85	1.172	0.81	1.231	0.69

A: Escravovus; B: Bonny light; C: Que Iboe; D: Brass ; E: Forcados crude oils; Significant at 5% level

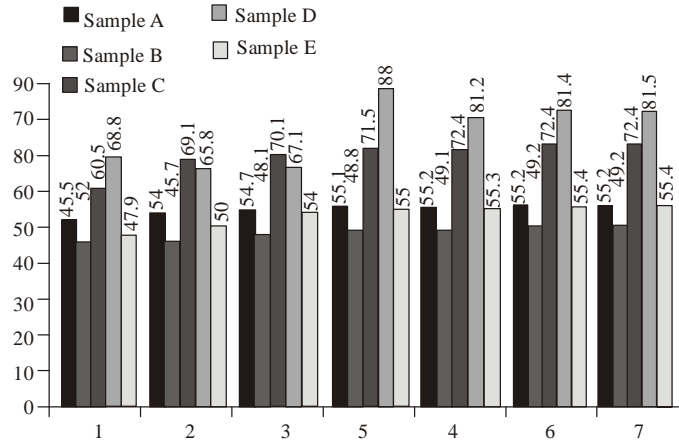


Fig. 7: % various crude oil adsorbed onto AOMC

sorption isotherm plots of crude oil samples A, B, C, D and E for activated orange meso-carp at varying temperature respectively. Slopes of Fig 2-6 represent the partition coefficient kd (L/kg) of various crude oil samples as presented in Table 2. Kd values of crude oil samples A, B, C, D and E were given as between 0.347-0.698, 0.45-1.164, 1.281-2.041, 1.172-1.399 and 1.231-4.532, respectively. From the table, Kd values varies significantly with temperature variation in all samples. It varies inversely with sorption temperature implying that Kd at temperature value of 10°C recorded the highest value and decreased to the lowest value at 50°C . These are presented in Fig. 2-6. Similar observations were made by (Felebuegu *et al.*, 2010; Wanchanthuek and Nunnung, 2011; Nwokoma and Anene, 2010; Hosseini and Qureshi, 2011; Syed and Mani, 2011). It was also observed that the optimum adsorption temperature range seemed to lie between 10-30°C. This is at variance with the observations of (Nwokoma and Anene, 2010). The variation could be attributed to differences in adsorbent type with respect to its texture, as the texture of adsorbent determines largely its sorption capacity (Kadirvelu *et al.*, 2001). Further similar observations on the phenomenon of increase in pollutant removal on a low particle size adsorbent with its attendant increase in surface area, was credited to Prasad and Sagar (2010), EL-Gaini *et al.* (2009) and Wanchanthuek and Nunnung (2011). Therefore, the differences in surface area occasioned by variations in particle sizes of the adsorbent can result to

differences in driving forces of sorption process and sorption temperature ranges.

**Percentage of crude oil adsorbed onto (AOMC):** Percentages of crude oil adsorbed on AOMC were computed to further study the capacity of the adsorbent (AOMC) to adsorb the adsorbate (crude oil). Adsorbents have varying degrees of capacity of adsorption of different adsorbate due to types and constituents of the solid and liquid phases involved (Dash and Murthy, 2010). In this study, percentage of crude oil adsorption on AOMC was highest for crude oil sample D followed by C with B having the least value. The trend of percentage adsorption of various crude oil samples on the adsorbent is shown as follows D>C >A>E>B. From Fig. 7 where the results of the percentage adsorption is shown, the % of adsorption for crude oil sample D is between 86.8-88% at different adsorption periods, and other samples subsequently followed as 60.5-72.4, 52.5-55.2, 47.9-55.4 and 45.3-49.2% for samples C, A, E and B, respectively. A similar study by Nwokoma and Anene (2010) reported >93% crude oil adsorption rate onto the solid phase. The pattern of sorption rate of the crude oil samples seems to have direct relationship with their constituent hydrocarbon concentrations. Crude oil sample D with the highest hydrocarbon values, has the highest adsorption rate of 86.8-88% and it decreased with other samples according to their hydrocarbon concentrations values. Therefore, the high sorption rate of crude oil sample D can be attributed

Table 3: Enthalpy and Entropy values at different samples.

Crude oil samples	Enthalpy change (KJ/mol)(H)	Entropy change (KJ/mol)(S)
A	-10.28	-2.92
B	-12.24	-0.32
C	-8.92	-0.50
D	-6.90	-0.44
E	-50.28	-0.01

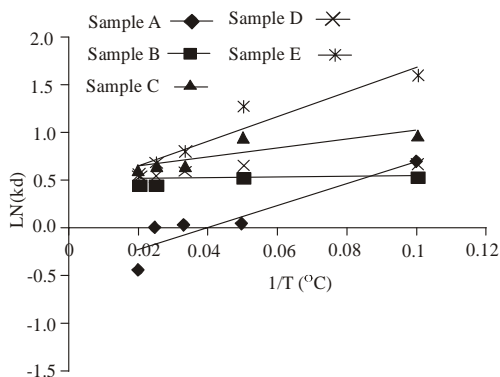


Fig. 8: Plot of  $\ln(kd)$  versus  $1/T$  for determination of change in enthalpy and entropy

to its high hydrocarbon concentration, adequate dryness and homogenized blend of AOMC particles as equally observed by (Dash, 2008; Rios *et al.*, 2004; Nwokoma and Anene, 2010). This explains why crude oil of high hydrocarbon concentration is more recalcitrant to soil particles in the face of crude oil spill on land (Nudelman *et al.*, 2002). The recalcitrant nature of such hydrocarbons values to solid phase enhances its easy remediation by sorption process in a case of crude oil spill on land and also limits the possibility of ground water hydrocarbon pollution through infiltration.

**Thermodynamics of crude oil sorption onto AOMC:**

The Thermodynamic properties determined using Eq. (4) and (5) included; enthalpy (H), entropy (S) and Gibbs free energy (G). The natural logarithm of the partition coefficient ( $\ln K_d$ ) obtained in Eq. (2) was plotted against the inverse of temperature ( $1/T$ ) for various crude oil samples to get linear curves.

$\Delta H$  and  $\Delta S$  were computed from the slopes and intercepts of the curves (Fig. 8). With enthalpy and entropy determined, the change in Gibbs free energy (G) was computed using Eq. (5). Table 3 shows the enthalpy

and entropy of sorption of crude oil samples at various hydrocarbon values. The enthalpy changes for different crude oil samples A, B, C, D, and E were: -10.28, -12.24, -8.92, -6.9 and -50.28 KJ/mol, respectively. This implies that the sorption process of crude oil onto the solid phase of AOMC is simply exothermic.

Again, the enthalpy changes seemed to be in tune with the varying hydrocarbon concentrations of crude oil samples. The direct relationships of the two variables can be attributed to level of affinity of high molecular hydrocarbons to adsorption to solid phase as the high molecular hydrocarbons cling to AOMC which was equally enhanced by its high sorption ability, more exothermic reactions take place resulting to less energy left within the system as enthalpy. The studies of Chen *et al.* (2010) and Ifelebuegu *et al.* (2010) showed that the heat of adsorption for processes observing physisorption and chemisorptions were in the ranges of 5-20 and 100-400 KJ/mol, respectively. From these observations, it is apparent that the enthalpy of sorption for crude oil adsorption onto AOMC is physisorption controlled.

Also the comparisons of entropy of sorption values of this study with the stated values of physisorptions and chemisorptions equally demonstrated limited effects of entropic process. Gibbs free energy ( $\Delta G$ ) of the sorption process seemed to be temperature sensitive. The Gibbs free energy ( $\Delta G$ ) value increased with temperature values. This trend is similar to the observation of (McMinn and Magee, 2003). With the values of G for different samples at various sorption temperatures, sorption of the crude oil samples onto AOMC is spontaneous.

Values from Table 4 show that sorption process is most spontaneous at temperature of 10°C and level of spontaneity decreased with increase in sorption temperature. This implies that the adsorption reaction is more spontaneous at low Gibbs free value. For instance, the spontaneity of adsorption reaction between the crude oil samples A and the AOMC at temperatures of 10, 20, 30, 40 and 50°C was expressed by the Gibbs free values at -71.49 < -66.79 < -58.02 < -49.32 < -21.29, respectively. That of crude oil samples B, C, D, and E were also expressed as -18.25 < -11.84 < -8.65 < -7.01 < -5.43, -30.24 < -27.33 < -24.90 < -22.99 < -21.11, -28.55 < -19.89 < -15.50 < -13.91 < -11.33, -50.74 < -50.30 < -50.13 < -40.40 < -50.12, respectively. This observation was given credence by the following observations credited to Aneke (2008) in respect of spontaneity and Gibbs free energy; for a

Table 4: Gibbs free energy ( $\Delta G$ ) for different crude oil samples.

Temp (°C)	( $\Delta G$ ) A	( $\Delta G$ ) B	( $\Delta G$ ) C	( $\Delta G$ ) D	( $\Delta G$ ) E
10	-71.49	-18.25	-30.24	-28.55	-50.74
20	-66.79	-11.84	-27.33	-19.89	-50.30
30	-58.02	-8.65	-24.90	-15.50	-50.13
40	-49.32	-7.01	-22.99	-13.91	-40.40
50	-21.29	-5.43	-21.11	-11.33	-50.12

\*:negative signs showed the level of spontaneity

chemical reaction to proceed spontaneously,  $\Delta G$  must be less than zero ( $\Delta G < 0$ ), and not spontaneous if  $\Delta G > 0$ . With  $\Delta G = 0$  equilibrium condition is established implying that the reaction will not proceed in either direction.

## CONCLUSION

Sorption of crude oil on the Activated Orange Meso-Carp (AOMC) has been studied. The driving force exerted by the activated orange meso-carp to influence sorption got to its equilibrium after 40 min. the sorption isotherm curves which described the partition coefficients followed the linear curve with coefficients of correlation ranging between 0.6-0.99 for various crude oil samples at different sorption temperatures values. The partition coefficients varied inversely with temperature and the optimum adsorption temperature range seemed to lie between 10-30°C. percentage of adsorption of the crude oil samples increased with hydrocarbon concentration values of respective samples. the reaction of the sorption process is exothermic and spontaneous, its enthalpy and entropy are physisorption controlled.

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