Adsorption of acenaphthene unto activated carbon produced from agricultural wastes

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ABSTRACT

The suitability and the performance of activated carbon produced from flamboyant pod back, milk bush kernel shell and rice husk for the effective removal of acenaphthene from simulated wastewater under the influence of carbonization temperature and initial concentration were investigated. The adsorption capacities of all the activated carbons obtained from the selected raw materials are influenced by increasing carbonization temperature. Activated carbons obtained from rice husk at carbonisation temperature of 600 ^{0}C had the maximum adsorption capacity (5.554 mg/g) while carbons produced from milk bush at carbonisation temperature of 300 ^{0}C had the minimum adsorption capacity (1.386 mg/g), for the adsorption of acenaphthene from the simulated wastewater. The removal efficiencies of the investigated adsorbents generally rank high and the highest value (80.56 %) was obtained for the adsorption of acenaphthene by rice husk carbonized at 600 ^{0}C. Furthermore, the removal efficiencies obtained in the study decreased as the initial concentrations of the adsorbate increased. The four selected isotherm models; Freundlich, Langmuir, Temkin and Dubinin-Radushkevich described well the equilibrium adsorption of acenaphthene unto activated carbon derived from Flamboyant pod bark, milk bush kernel shell and rice husk. Sequence of suitability of the selected isotherms in the study was Temkin ≈ Freundlich > Dubinin-Radushkevich > Langmuir for adsorption of acenaphthene. It therefore shows that Temkin isotherm is the most suitable model for fitting experimental data obtained from adsorption of acenaphthene from simulated wastewater unto activated carbon produced from Flamboyant pod bark, milk bush kernel shell and rice husk.

Introduction

The search for the removal of organic pollutants using alternative low cost adsorbents is now on the rise by many researchers (McKay, 1995; Mackay and Gschwend, 2000; Grupta *et al.,* 2002; Boving and Zhang, 2004; Zheng *et al.*, 2004; Gokturk and Kaluc, 2008). Few of these reasons are:

-Commercial activated carbons are expensive above $2.00/lb but agricultural by-products at low cost of $0.45/lb (Ahmedna *et al.*, 2000b).

- Agricultural wastes have low ash contents and high density (Johns *et al.*, 1999; Wartelle and Marshall, 2001).

-Activated carbon with a developed microporosity and wide micropore size distribution, which are characteristics of activated carbon produced from agriculture origin, can improve the adsorption of PAHs (Murilo *et al.*, 2004).

-Commercial activated carbons have recorded difficulty in regeneration and high cost of disposal (Darwish *et al.*, 1996; Gupta *et al.*, 1998; Flock *et al.*, 1999; Burlengh *et al.,* 2002; Singh *et al.,* 2003,).

-Some agricultural base activated carbon compare favourably well in physical and chemical properties with some commercial activated carbon (Ahmedna *et al.*, 1998; Johns *et al.*, 1999; Pendyal *et al.*, 1999; (Al-Barrak and Elsaid, 2005).

Wide varieties of high carbon content materials such as wood, coal, peat; nutshells, sawdust, bones, husk, petroleum coke and others have been utilized to produce activated carbon of varying efficiencies (Savova *et al.*, 2001; Ekinci *et al.*, 2002;

Aygun *et al.*, 2003; (Dutta and Basu, 2011)). These materials, usually in irregular and bulky shapes, are always adjusted to exhibit the desired final shapes, roughness and hardness. Generally, the production of activated carbon involves pyrolysis or carbonization and activation as the two main production processes (Vitidsant *et al.*, 1999).

Numerous carbonaceous materials, particularly, those of agricultural base, are being investigated to posses potential as activated carbon. The suitable ones have minimum amount of organic material and a long storage life. Similarly they consist of hard structure to maintain their properties under usage conditions. They can be obtained at a low cost. Some of the materials that meet the above conditions have been used, in past works, to produce activated carbons which were subsequently used for the treatment of wastewater and adsorption of hazardous gases. Agricultural by–products like rice straw, soybean hull, sugarcane baggasse, peanut shell, pecan shell and walnut shells were used by John *et al.,* (1998) to produce granulated activated carbons (GACs). Similarly, Atmani et al (2009) used acid, base and salt treatment process to activation of skin almond waste in order to improve its adsorption capacity for acid and basic dye from wastewater. Sea products such as crab shell have been used to produce activated carbon (Rana et al, 2009).

The choice of a particular material for the production of effective adsorbent (activated carbon) is based on low cost, high

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carbon and low inorganic content (Tsai *et al.,* 1997). Agricultural materials have attracted the interest of researchers for the production of adsorbents because of their availability in large amount and at a low cost (Ioannidou and Zabaniotou, 2007). The selected materials employed in this study were rice husk (*Oryza sativa*), flamboyant (*Delonix regia)* pod back and milk bush (*Thevetia peruviana*) kernel shell. Rice husk has been used in the production of activated carbon (Daifullah *et al.,* 2003; Subramanian *et al.*, 2004) but flamboyant pod bark and milk bush kernel shell are untapped agricultural produce that attracted the interest of this research. Use of agricultural by-product for the production of activated carbon is primarily for economic and ecological advantages (Itodo et al, 2011). Acenaphthene is also one of the common PAHs in the environment. It is otherwise called 1, 2- hydrocenbaphtylene and occurs in coal tar (Finar, 2005).

Materials and Methods

Agricultural raw materials

The natural precursor used in the production of the adsorbent include rice husk which were obtained from Arowomole Rice Mill, Ogbomosho. Others include flamboyant pod bark which was sourced from the campus yard of Ladoke Akintola University of Technology, Ogbomosho, while milk bush kernel shell was obtained from its trees scattered all over various secondary schools in Ogbomosho.

Reagents

The reagents used during the course of the experimental activities include sodium bicarbonate (NaHCO$_3$), phosphoric acid (H$_2$PO$_4$ 58 %), acetone (BDH Chemicals Ltd, mw 58.08g , spg 0.789 - 0.791 g/^{0}C), distilled water, polycyclic aromatic hydrocarbon (PAHs) of 2-rings, Naphthalene (100 g , Merck, Mw 128.18 g), and 3-ring, Acenaphthene (100 g, Merck, Mw 178.23). All reagents are analytical grade.

Materials processing

The materials were sorted to remove stones, shaft and debris, thereafter the backs of the flamboyant pod were removed mechanically and kept separately. Similarly, the shells of the milk bush kernel were mechanically cracked with nut cracker and the shells were carefully collected by hand for further treatment. The back and shell obtained were then washed with distilled water, to remove surface impurities (Bulut and Tez, 2007a; Jayrayan et al 2011) and later dried in the oven at a temperature of 105 ^{0}C overnight (Amuda and Ibrahim, 2006) to constant moisture level before crushing with milling machine so as to reduce their sizes to pellet-form and increase their surface area (Bulut and Tez, 2007a). They were then stored in dry containers prior to carbonization. Furthermore, since rice husk were in chaff form, they were washed with distilled water, before being dried in the oven at a temperature of 105°C to a constant weight (Amuda and Ibrahim, 2006).

Carbonization

1 kg of each agricultural material was charged into the furnace (Vecstra, Model 184A, Italy) which was then heated to the desired temperature (300^{0}C). The resulting charred material was collected and cooled at room temperature. The procedure was repeated for all the materials separately at carbonization temperatures of 300 - 600°C. The domain of variation of these factors is defined according to Bornemann *et al.,* (2007).

Activation

Samples of the carbonized material were weighed, soaked in excess phosphoric acid (H$_3$PO$_4$) for 3h (Kadirvelu *et al.*, 2001) and charged inside an oven at temperature of 200°C for 24

h (Kadirvelu *et al.*, 2001) to ensure proper adsorbate drying.. ZnCl$_2$ always produce wide pore surface area than base (Ahmadpour *et al.*, 1998), H$_3$PO$_4$ produces a better pore surface area and are relatively safer than ZnCl$_2$ (Johns *et al.*, 1999) and hence the choice for this study. The materials were then removed from the oven, cooled for 2 h and then washed with distilled water until leachable impurities due to free acid and adherent powder were removed (Amuda and Ibrahim, 2006). The samples were later soaked in 2% (w/v) NaHCO$_3$ to remove any residual acid left. The resulting mixture were further washed with distilled water to bring the pH to 7.0 and finally drained and dried overnight in an oven at 110°C (Amuda and Ibrahim, 2006).

Preparation of simulated water

Calculated amount (50 – 150 mg) of the desired PAH was weighed and added to 300 mL of acetone in 1dm^3 standard flask. The mixture was carefully swirled together for 10 min to allow proper dissolution. Then distilled water was added to the mixture to make up to the mark (Crisafully *et al.*, 2008), thus a solution of 50 mg/L of PAH was produced. The above procedure was repeated for the preparation of 75, 100, 125 and 150 mg/L of acenaphthene under this study.

Determination of adsorption capacity

Weighed amount (1g) of each activated carbon was added to 50 mL of the 50 mg/L stock solution in 250mL conical flask. The mixture in the flask was covered and placed on magnetic stirrer at 150 rpm for 2 h (Lemic *et al*, 2007) at ambient temperature (28$\pm$2 ^{0}C) and pH 7.5 (Crisafully *et al.*, 2008). After which the content was allowed to stand for 1 h and the supernatant solution was filtered with Whatman filter paper (15mm) into sample bottles (Crisafully *et al.*, 2008). Starting with 50 mg/L of the simulated wastewater prepared, the process was repeated for 75, 100, 125 and 150 mg/L of acenaphthene. Furthermore this procedure was repeated for same activated carbon produced at various temperatures. The resulting filtrates were subjected to analysis.

Analytical measurement

The unadsorbed concentration of either naphthalene or acenaphthene in the filtrate was quantified using gas chromatography coupled with flame ionization detection (GC – FID) (Crisafully *et al.*, 2008; Bornemann *et al.*, 2007). A HP-5 capillary of 30 m with internal diameter of 0.25 mm and film thickness of 0.25 µm was used. The column temperature was set to 60 ^{0}C for 2 min and then ramped to 320 ^{0}C programmed at 10 ^{0}C/min. Nitrogen was used as carrier gas at a constant pressure of 35 psi while hydrogen and air flow rate pressure were 22 psi and 28 psi, respectively. Injector port and detection temperature were 250 ^{0}C and 320 ^{0}C respectively while 1.0 µL of sample was injected, before analysis, calibration standard was run to check column performance peak height and resolution and the limits of detection of the compound was identified mainly by its retention time. The abundance of quantification of analyte with respect to authentic PAH standard detection limits was derived from replicate procedure.

Quantification of adsorption capacity

The adsorption capacities of the materials carbonized at different temperature were determined using equation 1 (Crisafully *et al.*, 2008)

$$q_e = \frac{(C_0 - C_e)}{w}V \qquad (1)$$

Where q$_e$ is the adsorption capacity of adsorbent (mg/g), C$_o$ is the initial concentration of the adsorbate in the solution

(mg/L); C_e is the final concentration of the adsorbate in the solution quantified with GC – FID (mg/L); V is the volume of the solution (mL) and w is the mass of the adsorbent (g). The removal efficiency RE (%) of each activated carbon at different concentration of selected adsorbate was calculated according to equation 2 (Kazemipour *et al.*, 2008; Amuda *et al*, 2009).

$$RE(\%) = \frac{(C_O - C_e)}{C_O} \times 100 \quad (2)$$

Where C_o and C_e are as defined above.

Adsorption isotherm models

Adsorption Isotherm equations applicable to single – solute were used to describe the experimental sorption data obtained in this study and the parameters of the isotherm models were obtained from graphical plotting of the experimental data. Four models used in this study include Freundlich, Langmuir, Temkin and Dubinin-Radushkevich isotherm models.

Langmuir Isotherm Model

The linearized form of the Langmuir Isotherm equation is expressed as:

$$\frac{C_e}{q_e} = \frac{1}{Q_L K_L} + \frac{1}{Q_L} C_e \quad (3)$$

Where C_e (mg/L) is the equilibrium concentration of the adsorbates, q_e (mg/g) is the amount of adsorbate adsorbed per unit mass of adsorbent. Q_L and K_L are related adsorption capacity and rate of adsorption respectively and were determined by plotting C_e/q_e against C_e. Importance of Langmuir isotherm is often investigated with dimensionless separation factor (R_L) (Hameed and Rahman, 2008; Amuda *et al*, 2009) which is expressed in the form (equation 4). Where K_L is the Langmuir constant and C_o is the highest concentration of adsorbate (mg/l) in the solution. The value of R_L obtained indicates Langmuir isotherm to be unfavourable ($R_L > 1$), linear ($R_L = 1$) and favourable ($0 < R_L < 1$) or irreversible ($R_L = 0$) (Hameed and Rahman, 2008)

$$R_L = \frac{1}{1 + K_L C_o} \quad (4)$$

Freundlich Isotherm Model

The Freundlich Isotherm equation is an empirical equation expressed in linear logarithmic form (Cheung *et al.*, 2009; Amuda *et al*, 2009) as (5). A plot of In q_e against In C_e is used to determine the Freundlich constants, K_f and $1/n$ respectively.

$$In q_e = In K_F + \frac{1}{n} In C_e \quad (5)$$

Temkin Isotherm Model

This equation focuses on the effects of indirect adsorbate - adsorbate interaction on adsorption (Olgun and Atar, 2009). It is expressed as:

$$q_e = B In A + B In C_e \quad (6)$$

Where A and B are the Temkin Constants which are evaluated by plotting q_e against In C_e. B is further determined from the expression

$$B = \frac{RT}{b} \quad (7)$$

Where b is related to the heat of adsorption, T is the adsorbate room temperature and R is the universal gas constant ($8.314 mol^{-1}K^{-1}$).

Dubinin-Radushkevich Isotherm (D-R) Model

D – R isotherm equation is usually use to estimate the porosity, apparent free energy and the characteristics of

adsorption (Nemr *et al*, 2009). It is commonly applied in its linear logarithm form as shown in equation 8

$$In q_e = In Q_{D-R} - K \varepsilon^2 \quad (8)$$

Where k is a constant related to adsorption energy and Q_{D-R} (mg/g) is the theoretical saturation capacity. The Polanyl potential is calculated from equation 9.

$$\varepsilon = RTIn\left(1 + \frac{1}{C_e}\right) \quad (9)$$

Where, R is the universal gas constant ($8.314 mol^{-1}K^{-1}$), T is the adsorbate room temperature and C_e (mg/L) is the equilibrium concentration of the adsorbates. D–R constants; k and Q_{D-R} (mgg^{-1}) were evaluated by plotting In q_e against ε^2.

Mean free energy of adsorption

The mean free energy of adsorption is defined as the free energy change when one mole of adsorbate is transferred to the surface of the adsorbent from infinity in the solution (Nemr *et al*, 2009). It is calculated from the values of K obtained from the plot of Dubinin-Radushkevic using the relation (Kundu and Gupta, 2006).

$$E = \frac{1}{\sqrt{2K}} \quad (10)$$

Where k is a constant related to the adsorption energy (J mol^{-1})

Adsorption isotherms error analysis

In order to determine the ability of the selected adsorption isotherm models, employed in linear method, the experimental data and parameter of the isotherms were subjected to statistical error analysis The selected error analysis are the Average Relative Error (ARE) and Average Relative Standard Error (ARS) Their values are expected to be very low for a given isotherm model to be favourable particularly under the linear method (Han *et al.*, 2009). The error analysis was employed in this study in order to find the best correlation between the non – linear isotherm equations and experimental data points (Cheung *et al.*, 2009). The ARE and ARS are determined using the equations 11 and 12 respectively (Han *et al.*, 2009).

The Average Relative Error (ARE)

$$ARE = \left[\sum |(q_c - q_e)/q_e|\right] \quad (11)$$

The average relative standard error (ARS)

$$ARS = \sqrt{\sum \left[\frac{(q_c - q_e/q_e)^2}{n-1}\right]} \quad (12)$$

Where q_c is the calculated quantity of adsorbate adsorbed onto the adsorbent and q_e is the experimental data and 'n' is the number of experimental data points.

Results and Discussion

In this study, each of the agricultural materials employed were carbonized at 300 - 600°C and thus flamboyant pod back are coded FB300- FB600; rice husk, RH300 - RH600 while milk bush kernel shell carbons are coded MB300 - MB600.

Influence of carbonization temperature and initial concentration on adsorption capacities of the activated carbons

The adsorption capacity of FB300 for the adsorption of acenaphthene from the simulated wastewater increased from 1.466 – 4.207 mg/g as the initial concentration of the polyaromatic hydrocarbon increased from 50 – 150 mg/L. Similarly, the adsorption capacities of FB500 increased from 1.538 – 4.504 mg/g as the initial concentration increased from 50 -150 mg/L. FB600 showed increased adsorption capacities from 1.634 – 4.754 mg/g as the initial concentration of acenaphthene increased from 50 – 150 mg/L. Furthermore, the

result shows that maximum adsorption capacity of rice husk for acenaphthene increased from 5.353 to 5.554 mg/g as the carbonization temperature increased from 300 – 600°C at a concentration of 150 mg/L. Similar trend was observed at other initial concentrations (50 – 125 mg/L) of acenaphthene in the aqueous media as the carbonization increased from 300 – 600°C. The adsorption capacity of MB300, MB500 and MB600 increased from 1.388 – 3.419 mg/g as the initial concentration of acenaphthene increased from 50 -150 mg/L. MB500 showed adsorption capacities which increased from 1.449 – 4.086 mg/g as the initial concentration of acenaphthene increased from 50 – 150 mg/L. Similarly, as the initial concentration of acenaphthene increased from 50 – 150 mg/L, the adsorption capacity of MB600 also increased from 1.469 to 4.331 mg/g. The results further showed that the maximum adsorption capacities of activated carbon obtained from milk bush kernel shell increased from 3.419 – 4.331 mg/g as the carbonization temperature increased from 300 – 600°C. Maximum adsorption capacities recorded for Flamboyant pod back are 4.207, 4.504 and 4.754 mg/g. The results show that there is increase in adsorption capacity as carbonization temperatures increased. Based on the maximum adsorption capacities obtained for the activated carbon produced from the selected agricultural raw material, the order of capacity is given as rice husk > flamboyant pod back > milk bush kernel shell. Bornemann *et al.*, (2007) equally observed that carbonization temperature influenced the sorption behaviour of aromatic hydrocarbon on charcoals prepared from grass and wood. Similarly, carbonization temperature influenced the adsorption of organic compound like phenol and methylene blue unto activated carbon produced from hazelnut (Ioannioudou and Zabaniotou, 2007). Furthermore, high adsorption capacities exhibited by these adsorbent may be attributed to the dispersion interactions between the Π (pi)-electrons of the aromatic ring (Moreno-Castilla, 2004).

Influence of carbonization temperature and initial concentration on removal efficiencies of the activated carbons

Figure 1 shows the removal efficiencies, RE (%) of the three activated carbons produced for the adsorption of naphthalene and acenaphthene under the influence of increasing carbonization temperature (300 – 600 °C) and initial concentration (50 - 150 mg/L) of the two adsorbates. Removal efficiencies of FB300, FB500 and FB600 decreased from 58.65 – 56.09, 61.53 – 60.01 and 65.39 – 63.38% respectively as the initial concentration of acenaphthene increased from 50 – 150 mg/L. However, the maximum removal efficiencies of the activated carbon flamboyant pod back; 58.65, 61.53 and 65.39% increased as the carbonization temperature increased from 300, 500 and 600 °C respectively. It can be deduced that increasing carbonization temperature favour the efficiencies of activated carbon obtained from flamboyant pod back for the removal of acenaphthene from water medium, however this can be improved at higher carbonization temperature and initial concentration lower than 50 mg/L. Similar trend was recorded for the adsorption of acenaphthene from the simulated wastewater unto the activated carbon obtained from milk bush kernel shell at various carbonization temperatures. The removal efficiencies of MB300, MB500 and MB600 decreased from 54.48 - 45.59, 57.96 - 54.48, and 58.75 - 57.74 %, respectively as the initial concentration of acenaphthene increased from 50 - 150mg/L. The maximum removal efficiencies of carbon produced from milk bush kernel at carbonization temperature of 300, 500 and 600 °C for the removal of acenaphthene (54.48,

57.96 and 58.75 %) are higher than the maximum removal efficiencies obtained for the removal of naphthalene (48.18, 53.55 and 56.41), respectively. It shows that efficient activated carbon from milk bush kernel shell may be produced at carbonization temperature of 600 °C and used to remove polyaromatic hydrocarbon at low concentration. The study showed that the removal efficiencies of all the agricultural raw materials investigated for the adsorption of acenaphthene increased with increasing carbonization temperature but decreased with increased initial concentrations. this showed increased removal efficiencies as initial concentration increased from 50 – 150 mg/L. Furthermore, the removal efficiencies of all the materials studied for the removal of acenaphthene are higher than those observed for naphthalene at a given initial concentration and carbonization. This may be attributed to the high partition coefficients (Log K_{ow}) of acenaphthene (3.989) and this has been reported to be a factor that influences the adsorption of polyaromatic hydrocarbons in aqueous medium onto activated carbon (Lemic *et al.*, 2007).
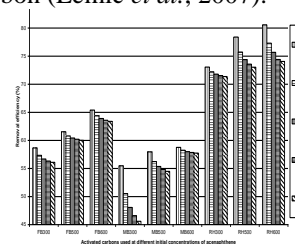

Figure 1: Removal Efficiencies (%) of activated carbons at different initial concentrations of acenaphthene in simulated waste water

Effect of carbonization temperature on Freundlich Isotherm Model

Figure 2 shows the plots of Inq_e (adsorption capacity) versus InC$_e$ (equilibrium concentration of adsorbate) used for determining the Freundlich isotherm parameters for naphthalene and acenaphthene adsorption, respectively, onto activated carbons produced. Freundlich isotherm parameters obtained for the adsorption of naphthalene and acenaphthene onto the adsorbents produced from agricultural raw materials are shown in Table 1. The Freundlich exponent 'n' for the adsorption of acenaphthene unto FB300, FB500 and FB600 are 1.10, 1.06 and 1.08, respectively. Furthermore, the Freundlich exponent 'n' for the adsorption of acenaphthene unto MB300, MB500 and MB600 are 1.45, 1.14 and 1.04, respectively. For the activated carbons derived from rice husk, RH300, RH500 and RH600, the values of 'n' obtained for adsorption of acenaphthene are 1.08, 1.29 and 1.38, respectively. The Freundlich exponent 'n' obtained for this study are above those obtained from a related study of adsorption of naphthalene and acenaphthene unto activated carbons derived from agricultural materials (Table 2) except for FB300 and RH600 which are less than value obtained for sugarcane baggasse (1.02). Furthermore, the Freundlich exponent 'n' obtained in this study are less than values obtained for adsorbent derived from resin (Long *et al.*, 2008). However, these values satisfy the condition, 1<n<10, which indicate that the adsorption of acenaphthene unto the activated carbon produced from the selected agricultural materials is favourable (Rao *et al.*, 2009). The corresponding large values of correlation coefficient (R^2) (Table 4.9) of R^2 (0.997 – 1.000) showed that the Freundlich model is good for describing the adsorption process of acenaphthene unto carbon derived from the agricultural raw materials investigated in this study (Crisafully

et al., 2007). The appropriateness of this model to the selection of acenaphthene is an indication of heterogeneity of the adsorbents (Olgun and Atar, 2009).
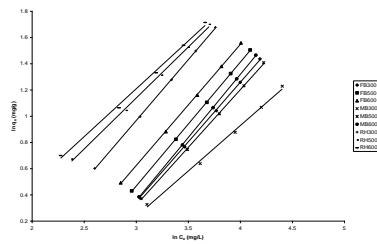
Figure 2: Freundlich adsorption isotherm of acenaphthene unto activated carbons produced

Effect of carbonization temperature on Langmuir Isotherm model

Figure 3 shows the plots of C_e/q_e (ratio of equilibrium concentration of adsorbate to adsorption capacity) versus q_e (adsorption capacity) using linear method and this was used to determine the Langmuir isotherm parameters for the adsorption of acenaphthene onto the three types of adsorbents produced at carbonization temperatures of 300, 500 and 600 ^{0}C. In the adsorption study of acenaphthene unto the activated carbon derived from flamboyant pod back, (Table 3) the monolayer capacity, Q_L, obtained for FB300, FB500 and FB600 are 29.07, 51.28, and 38.72 mg/g, respectively. With separation factor (R_L) of 0.73, 0.81 and 0.73 which satisfy the condition 0<RL<1, shows that Langmuir isotherm is favourable (Hameed and Rahman, 2008). The monolayer capacity, Q_L, of MB300, MB500 and MB600 are 7.59, 21.41 and 71.94 mg/g, respectively, which increased with increasing carbonization temperature of milk bush kernel shell. However, the Q_L; 45.87, 15.82 and 13.37 mg/g, obtained for RH300, RH500 and RH600 respectively, decreased as the carbonization temperature increased from 300 – 600 ^{0}C. The corresponding values of R_L, which ranged between 0.28 - 0.68, satisfied the condition 0<RL<1 and this shows that Langmuir isotherm is favourable (Hameed and Rahman, 2008). Furthermore, MB600 posses the largest monolayer capacity, 71.94 mg/g for the adsorption of acenaphthene unto the activated carbon produced while FB500 posses the largest monolayer, 294.118 mg/g, for the adsorption of naphthalene from the simulated water. The positive values of Q_L indicate high adsorption capacity of the activated carbon obtained from these raw materials (Rao *et al*., 2009). These values are however, below those obtained for the adsorption of organic compounds unto commercial activated carbons but compare well with activated carbon derived from agricultural raw materials (Table 4). Generally, the correlation coefficients (R^2) of these activated carbons are high (0.9172 - 0.9437) and such high degree of R^2, particularly for the Langmuir isotherm model, suggests that these activated carbons exhibit monolayer coverage with constant activation energy (Chiung *et al*., 2009, Han *et al*., 2009).
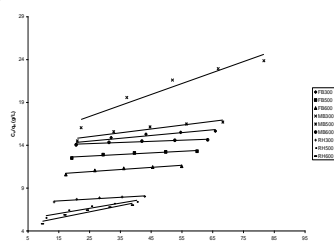
Figure 3: Langmuir adsorption isotherm of acenaphthene unto activated carbons produced

**Effect of carbonization temperature on Temkin Isotherm Model**

Table 5 shows the Temkin isotherm parameters (A, B and b) and their corresponding correlation coefficients (R^2). These parameters were obtained from the plot of q_e (adsorption capacity) versus InC_e (equilibrium concentration of adsorbate) (Figures 4) for acenaphthene adsorption onto the activated carbons produced at various carbonization temperatures. Temkin parameter constant (b) is related to the heat of adsorption in the adsorption process of acenaphthene unto the activated carbons produced from the selected agricultural raw materials. Heat of adsorption of acenaphthene unto carbon produced from flamboyant pod back (Table 5) ranges from 861.64 - 984.44 Jmol^{-1}, which decreased as the carbonization increased from 300 – 600 ^{0}C. Heat of adsorption of acenaphthene unto the MB and the values of 'b' obtained ranged from 911.02 - 1495.70 Jmol^{-1}. The heat of adsorption of naphthalene unto MB500 and MB600 are higher than heat of adsorption of acenaphthene unto the carbon derived from milk bush kernel shell (MB) at the same carbonization temperatures. Heat of adsorption of acenaphthene unto carbon obtained from rice husk ranged from 764.65 to 920.56 Jmol^{-1}, this increased as the carbonization temperature increased from 300 to 600 ^{0}C. Generally, with the highest value of heat of adsorption, the three activated carbon showed the same decreasing order (MB>FB>RH) of heat of adsorption for acenaphthene. Temkin isotherm is generally applied to the study of dye (Nemr *et al*., 2009; Olgun and Atar, 2009; Hans *et al*., 2009), and the values of heat of adsorption of Db-86 (dye) unto carbon produced from orange peels which ranged from 355.9 to 680.8 Jmol^{-1} (Nemr *et al*., 2009) are fairly less than those obtained in this study. Similarly, the heat of adsorption of phenol (79.78 Jmol^{-1}) unto activated carbon derived from Rattan Sawdust (Hameed and Rahman, 2008) is less than the heat of adsorption of acenaphthene in this study. The large values of correlation coefficients (R^2) obtained for the adsorption of acenaphthene unto selected adsorbents range from 0.9667 to 0.9766. Since these values compare well above the correlation coefficients (R^2) obtained for Langmuir isotherm model, it suggests that Temkin isotherm model can be applied to the study of adsorption capacity of activated carbon produced from milk bush kernel shell and rice husk for the removal of acenaphthene in simulated wastewater.
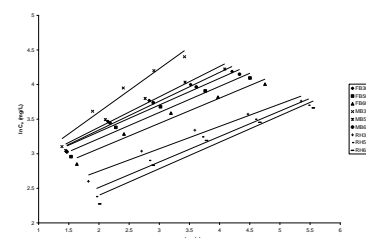
Figure 4: Temkin adsorption isotherm of acenaphthene unto activated carbons produced

Effect of carbonization temperature on Dubinin-Radushkevich Isotherm Model

Values of Dubinin-Radushkevich (D-R) isotherm model parameters (K_T and Q_{D-R}) obtained from the plot of Inq_e (adsorption capacity) versus ε^2 (Polanyi potential) in Figure 5 for the adsorption of acenaphthene unto the selected raw materials carbonized at various temperature of 300, 500 and 600 ^{0}C, are shown in Table 6. The essential characteristic of the Dubinin-Radushkevich isotherm is the estimation of the mean free energy of adsorption (E) often defined as the free energy

change involved in the transfer of one mole of adsorbate unto the surface of adsorbent (Nemr, 2009). The mean free energy of adsorption (E) obtained for the adsorption of acenaphthene unto carbon derived from flamboyant pod back range from 7.86 - 10.10 KJmol^{-1}. Constant value of mean free energy of adsorption (7.07 KJmol^{-1}) was obtained for the adsorption of acenaphthene unto MB300 and MB500 with mean free energy of adsorption of 7.86 KJmol^{-1}. High mean free energy of adsorption is obtained for the adsorption of acenaphthene unto rice husk ranged from 8.84 to 14.14 KJmol^{-1}, this and increased as the carbonization temperature increased from 300 to 600^{0}C though less than the mean free energy of adsorption of acenaphthene, 17.68, 35.35 and 35.35 KJmol-^{1} unto RH300, RH500 and RH600 respectively. Nemr *et al.*, (2009) suggested that the closeness of the values of mean free energy of adsorption as observed for the adsorption of acenaphthene unto MB300, MB500 and MB60 indicate that physico-sorption play the significant roles in the adsorption of these adsorbates from the simulated wastewater. Moreover, the values of mean free energy of adsorption observed in this study fall between 5 and 40 KJmol-1; it shows the type of adsorption involved in the study is physisorption (physical sorption) which usually takes place at low temperature (Cooney, 1999; Inglezakis and Poulopoulos, 2006). Furthermore, the applicability of this isotherm to the study of adsorption of naphthalene and acenaphthene was based on the large values of their correlation coefficients (R^{2}) Table (6).
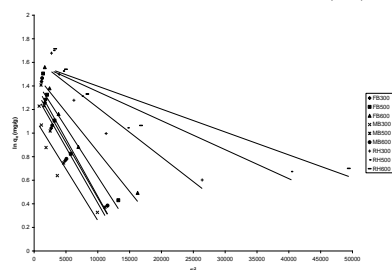
Figure 5: Dubinin-Rdushkevich adsorption isotherm of acenaphthene unto activated carbons produced

Error Analysis

The suitability of selected adsorption isotherms employed in predicting the fits of adsorption capacity is based on the low values of their average relative errors (ARE) and average relative standard error (ARS) (Hans *et al.*, 2009; Cheung *et al.*, 2009). The average relative errors (ARE) and average relative standard error (ARS) of Freundlich, Langmuir, Temkin and Dubinin-Radushkevich isotherm employed in this study are recorded in Table 7. The average relative errors of Freundlich, Langmuir, Temkin and Dubinin-Radushkevich isotherm for fitness in describing the adsorption of acenaphthene unto FB300, FB500 and FB600 ranged between 0.955 - 0.962, 11.33 - 13.67, 0.961 - 0.963 and 0.775 - 0.811, respectively. The large values of Langmuir confirm that it is less suitable for studying the adsorption of acenaphthene unto carbon derived from flamboyant pod back. With large values of ARE, Langmuir isotherm is less suitable for describing the adsorption of properties of acenaphthene unto MB300, MB500 and MB600. In the study of adsorption of acenaphthene, the ranges of ARE are 0.923 - 0.967, 2.62 - 29.13, 0.949 - 0.965 and 0.580 - 0.906 for the above stated isotherms, respectively. Error analysis of Freundlich, Langmuir, Temkin and Dubinin-Radushkevich range for the adsorption of acenaphthene is 0.88 - 0.944, 3.19 - 14.43, 0.670 - 0.955 and 0.669 - 0.802 for the isotherm models, respectively. In this case, only Langmuir isotherm is not

applicable to the study of adsorption of acenaphthene unto rice husk. Based on the ranges of ARE obtained in the study, the sequence of applicability of the selected adsorption isotherm is Dubinin-Radushkevich > Freundlich ≈ Temkin > Langmuir for acenaphthene as applied in the work of Baral *et al.*, (2009). The fitness of the isotherms to the experimental data can be optimized by error analysis particularly average relative standard error (ARS), (Cheung *et al.*, 2009). Isotherm models with very low values of ARS are the most suitable to the study (Hans *et al.*, 2009). The ARS for the adsorption of acenaphthene unto the same adsorbent ranged between 1.17 - 1.18, 15.41 - 26.54, constant at 1.18, and 1.35 - 1.41, respectively. With relatively low values of ARS, it shows that Temkin and Freundlich isotherms are the suitable models for fitting adsorption of acenaphthene unto carbon developed from Flamboyant pod back. Ranges of ARS of Freundlich, Langmuir, Temkin and Dubinin-Radushkevich isotherm models used to fit the adsorption capacities of acenaphthene unto carbon produced from milk bush kernel shell were in the ranges 1.13 - 1.18, 3.61 - 39.45, 1.16 - 1.18 and 0.98 - 1.62, respectively. From this, adsorption capacities fitted best with Freundlich and Temkin isotherm models. Furthermore, the ranges of ARS calculated for Freundlich, Langmuir, Temkin and Dubinin-Radushkevich isotherm models for the adsorption of acenaphthene unto rice husk were 1.07 - 1.16, 4.48 - 19.72, 1.00 - 1.17 and 1.14 - 1.40 for the above stated isotherm models respectively. Thus adsorption data of acenaphthene can be fitted with Temkin and Freundlich isotherms. Sequence of suitability of the selected isotherms in the study was Temkin ≈ Freundlich > Dubinin-Radushkevich > Langmuir for adsorption of acenaphthene (Baral *et al.*, 2009). It therefore shows that Temkin isotherm is the most suitable model for fitting experimental data obtained from adsorption of acenaphthene from simulated water unto activated carbon produced from rice husk, flamboyant pod back and milk kernel shell.

Conclusion

This study was conducted to determine the suitability and the performance of activated carbon produced from flamboyant pod back, milk bush kernel shell and rice husk for the effective removal of naphthalene and acenaphthene from simulated wastewater under the influence of carbonization temperature and initial concentration. The following conclusions could be made based on the analysis of the results obtained in this work. The adsorption capacities of all the activated carbons obtained from the selected raw materials are influenced by increasing carbonization temperature. Activated carbons obtained from rice husk at carbonisation temperature of 600 ^{0}C had the maximum adsorption capacity (5.554 mg/g) for the adsorption of acenaphthene while carbons produced from milk bush at carbonisation temperature of 300 ^{0}C had the minimum adsorption capacity (1.386 mg/g), from the simulated wastewater. The removal efficiencies of the investigated adsorbents generally rank high and the highest value (80.56 %) was obtained for the adsorption of acenaphthene by rice husk carbonized at 600 ^{0}C. Furthermore, the removal efficiencies obtained in the study decreased as the initial concentrations of the adsorbate increased. The four selected isotherm models; Freundlich, Langmuir, Temkin and Dubinin-Radushkevich described well the equilibrium adsorption of naphthalene and acenaphthene unto activated carbon derived from Flamboyant pod bark, milk bush kernel shell and rice husk. Sequence of suitability of the selected isotherms in the study was Temkin ≈

Freundlich > Dubinin-Radushkevich > Langmuir for adsorption of acenaphthene. It therefore shows that Temkin isotherm is the most suitable model for fitting experimental data obtained from adsorption of acenaphthene from simulated wastewater unto activated carbon produced from Flamboyant pod bark, milk bush kernel shell and rice husk.

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Table 1: Freundlich isotherms model parameters for acenaphthene adsorption onto activated carbons produced

Adsorbents	Acenaphthene		
	n	K_F (mg/g)	R^2
FB300	1.10	0.0936	0.9998
FB500	1.06	0.094	1.0000
FB600	1.08	0.1177	0.9999
MB300	1.45	0.1623	0.9977
MB500	1.14	0.1266	0.9997
MB600	1.04	0.0796	1.0000
RH300	1.08	0.1635	0.9999
RH500	1.29	0.3091	0.9987
RH600	1.38	0.3836	0.9981

K_F and n are Freundlich parameters while R^2 is the correlation coefficient

Table 2: Freundlich exponent 'n' of adsorption of acenaphthene unto some selected activated carbons

Adsorbent	Acenaphthene n	Reference
FB300	1.10	This study
FB500	1.06	This study
FB600	1.08	This study
MB300	1.45	This study
MB500	1.14	This study
MB600	1.04	This study
RH300	1.08	This study
RH500	1.29	This study
RH600	1.38	This study
Sugarcane baggasse	0.81	Crisafully *et al.*, 2008
Green coconut shell	0.42	Crisafully *et al.*, 2008
Chitin	0.55	Crisafully *et al.*, 2008
Chitosan	0.37	Crisafully *et al.*, 2008

Table 3: Langmuir isotherms model parameters for naphthalene and acenaphthene adsorption onto Selected Adsorbents

Adsorbent	Acenaphthene			
	Q_L (mg/ g^{-1})	K_L ($Lmol^{-1}$)	R^2	R_L
FB 300	29.07	0.0025	0.9172	0.73
FB 500	51.28	0.0016	0.9254	0.81
FB 600	38.76	0.0025	0.9251	0.73
MB300	7.59	0.0096	0.9437	0.41
MB 500	21.41	0.0034	0.9228	0.66
MB 600	71.94	0.001	0.925	0.87
RH 300	45.97	0.0031	0.9262	0.68
RH 500	15.82	0.0127	0.9306	0.35
RH 600	13.37	0.0175	0.932	0.28

Table 4: Langmuir isotherms model parameters for organic compounds adsorption onto some adsorbents

Adsorbent	Adsorabte	Q_L	References
FB 300	Acenaphthene	29.07	This study
	Acenaphthene	51.28	This study
FB 600	Acenaphthene	38.76	This study
MB 300	Acenaphthene	7.59	This study
MB 500	Acenaphthene	21.41	This study
MB 600	Acenaphthene	71.94	This study
RH 300	Acenaphthene	45.97	This study
	Acenaphthene	15.82	This study
	Acenaphthene	13.37	This study
Activated carbon	Phenol	322.5	Ahmaruzzaman and Sharma, 2005.
(CAC)	P-Nitrophenol	526.31	Ahmaruzzaman and Sharma, 2005.
	P-Chlorophenol	500.00	Ahmaruzzaman and Sharma, 2005.
Rice husk	Phenol	4.508	Ahmaruzzaman and Sharma, 2005
	P-Nitrophenol	15.31	Ahmaruzzaman and Sharma, 2005
	P-Chlorophenol	14.36	Ahmaruzzaman and Sharma, 2005
Rice husk char	Phenol	7.91	Ahmaruzzaman and Sharma, 2005
	P-Nitrophenol	39.21	Ahmaruzzaman and Sharma, 2005
	P-Chlorophenol	36.23	Ahmaruzzaman and Sharma, 2005
Rattan sawdust	Phenol	149.25	Hameed and Rahman, 2008
Sugarcane baggasse (fly ash)	Phenol	23.832	Hameed and Rahman, 2008

Table 5: Temkin Isotherm Model parameters for acenaphthene adsorption onto Selected Adsorbents

Absorbents	B	A	b (Jmol^{-1})	R^2
FB 300	2.3056	0.0878	984.44	0.9747
FB 500	2.5475	0.0916	890.96	0.9766
FB 600	2.6342	0.1034	861.64	0.9754
MB 300	1.5175	0.1070	1495.70	0.9701
MB 500	2.1821	0.0888	1040.16	0.9747
MB 600	2.4914	0.0842	911.02	0.9767
RH 300	2.9683	0.1321	764.65	0.9754
RH 500	2.5815	0.1888	879.23	0.9693
RH 600	2.4656	0.2214	920.56	0.9667

A,B and b are Temkin parameters while R^2 is the correlation coefficient

Table 6: Dubinin-Raduskevich Isotherm Model parameters for acenaphthene adsorption onto selected adsorbents

Adsorbent	K$_T$ (10^{-4})	Q$_{D-R}$ (mg/g)	R^2	E (KJmol^{-1})
FB 300	0.9	4.171	0.9417	7.86
FB 500	0.8	4.515	0.946	8.84
FB 600	0.7	4.730	0.9435	10.10
MB 300	0.9	3.245	0.9185	7.86
MB 500	0.9	4.022	0.9395	7.86
MB 600	1.0	4.364	0.9473	7.07
RH 300	0.4	5.334	0.9443	17.68
RH 500	0.2	5.187	0.9273	35.35
RH 600	0.2	5.180	0.9215	35.35

Q$_T$ and K$_T$ are Dubinin–Radushkevich parameters, R^2 is the correlation coefficient while E is the adsorption energy (KJ mol^{-1})

Table 7: Values of error analysis about isotherm equations for acenaphthene adsorption unto the activated carbons

Adsorbent	Freundlich Isotherm		Langmuir Isotherm		Temkin isotherm		D-R Isotherm	
	ARE	ARS	ARE	ARS	ARE	ARS	ARE	ARS
FB 300	0.960	1.18	11.33	15.41	0.963	1.18	0.775	1.35
FB 500	0.962	1.18	19.57	26.54	0.963	1.18	0.811	1.41
FB 600	0.955	1.17	13.67	18.58	0.961	1.18	0.793	1.38
MB 300	0.923	1.13	2.62	3.61	0.949	1.16	0.580	0.98
MB 500	0.945	1.16	8.25	11.25	0.962	1.18	0.906	1.62
MB 600	0.967	1.18	29.13	39.45	0.965	1.18	0.828	1.44
RH 300	0.944	1.16	14.43	19.72	0.955	1.17	0.802	1.40
RH 500	0.901	1.10	4.07	5.67	0.940	1.15	0.698	1.20
RH 600	0.88	1.07	3.19	4.48	0.670	1.00	0.669	1.14

ARE =Average Relative Error, ARS= Average Relative Standard Error and D-R =Dubinin-Raduskevich