PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM FLUTED PUMPKIN (*TELFAIRIA OCCIDENTALIS* HOOK.F) SEED SHELL

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ABSTRACT

Ten samples of activated carbon were successfully prepared by carbonization and activation of fluted pumpkin seed shell. Thermal activation was achieved at 300°C, 400°C, 500°C, 600°C and 700°C, while chemical activation was achieved by first impregnating the prepared raw material with activating reagents, and then carbonization at 500°C. Characteristics of the activated carbons were determined using standard methods. Increasing the activation temperature from 300°C to 700°C showed improved characteristics and optimum temperature for production of activated carbon was obtained at 500°C after 2 hours. The iodine values for physically prepared carbons were generally lower, compared to chemically prepared carbons. Phosphate removal efficiency and rates of phosphate removal were satisfactory for all prepared activated carbons though the chemically prepared samples had higher efficiencies. Results reveal that quality activated carbon could be produced from this agro waste.

Keywords: Characterization, fluted pumpkin, seed shell, activated carbon.

INTRODUCTION

Activated carbon (AC) is a generic term for a family of highly carbonaceous materials none of which can be characterised by a structural formula (Bansal *et al.*, 1988). It is perhaps one of the most important types of industrial carbon materials and is prepared by carbonization and activation of a large number of raw materials of organic origin such as wood, coal, and lignite (Zhonghuo *et al.*, 2001). Characteristics of AC depend on the physical and chemical properties of the raw materials as well as method of activation (Lua and Gua, 2001). The process for preparing activated carbons involves carbonization and activation of the carbonized product by physical or chemical activation (Ogbonaya, 1992; Jagtoyen *et al.*, 1998). Six different ways of combining the carbonization and activation processes are simplified in the works of Lafi, (2 001); Dastgeib, and Rockstraw, (2001); Ahmenda *et al.*, (2001) and Diao *et al.*, (2002).

The carbonization process enriches the carbon content and to creates an initial porosity in the char while activation further develops the porosity and creates some ordering of the structure, thereby generating a highly porous solid as the final product (Xia *et al.*, 1998; Johns *et al.*, 1999; Hernandez, 2007).

One major drawback of naturally occurring organic substances as precursors for activated carbon is that the resulting pore size distribution cannot be controlled (Lain *et al.*, 1989; Adebayo and Aloko, 2007). This led to the use of synthetic resins and polymers such as polyvinyl chlorides, and zeolytes (Carrott *et al.*, 2001). The last few decades have witnessed increasing technology that requires the use of activated carbons in a wide range of applications involving adsorption

(Njoku and Ubani, 2009; Melik, 2002). As a result of increasing demand for adsorbents locally available carbonaceous materials have proven worthy for producing ACs (Odozie and Akaranta, 1986; Sharat, 2008). In search of alternative sources and cheaper carbons, agro wastes with average carbon content of 35 percent have attracted the interest of researchers (Okiemmen *et al.*, 2007; Ekpete *et al.*, 2010).

Fluted pumpkin seed shell constitutes the largest morphological part of the plant. Seeds and leaves of this fruit have been investigated (Horsfall and Spiff, 2005; Agartemor, 2006; Sara *et al.*, 2008). Adesola *et al.*, (2006) reports that over 80 tonnes of waste is being produced yearly within the eastern states of Nigeria. Therefore by producing AC, wealth can be created from this waste, at same time cleaning the environment of this nuisance that has often been breathing grounds for rodents and mosquitoes in places where the plants is cultivated (Okolie and Nyanayo,1998; Okereke, 2006). In this study activated carbons were produced from fluted pumpkin seed shell and its determined characteristics reported.

MATERIALS AND METHODS

Sample Preparation

Fluted pumpkin seed shells, (FPS) were collected from Madonna University, Elele Campus and were cut into smaller sizes about 2 x 2mm using a knife, then sundried for 16 days before milling with a hand propelled kitchen machine to increase the surface area. The puverised, FPS was put into polyethylene bags and preserved till use in the laboratory.

The procedure by Grigis (1999) with slight modifications was used. The milled substances were washed with distilled water and rinsed with 1% HCl, to remove water soluble impurities; such as metal ions and surface adhered particles. It was kept in 0.1M NaOH solution overnight to remove lignin and in 0.1 M CH₃COOH to remove alkalinity developed due to NaOH. There after it was washed well with distilled water till the wash water became colorless and then dried at 110°C in an oven for two hours to get rid of moisture and other volatiles. The pretreated materials were then used for carbonization, activation and characterization.

Carbonization and Physical Activation

This was carried out according to the method used by Abdul *et al.*, (2008). Five 100 g of 150-300 μ m particle size of the pre-treated raw material were in turns placed in platinum crucible and carbonized at different temperatures of 300, 400, 500, 600 and 700°C for 2 hours each. It was ensured that little or no oxygen is present during carbonization and activation by first filling the furnace with a stream of CO₂. At the end of 2 hours the sample was allowed to cool to room temperature, removed and sieved with a 300 μ m sieve placed in an air tight bottle and labelled FAC300°C, FAC400°C, FAC500°C, FAC600°C and FAC700°C respectively and collectively called physically prepared fluted pumpkin seed shell activated carbon (PFAC).

Chemical Activation and Carbonization With, H₃PO₄, H₂SO₄, Na₂CO₃, NaOH, NaCl

Chemical activation was carried out according to the methods of Girgis, (1999), and Ajayi and Olawale, (2009) with slight modification. 30% wt of aqueous solution of sodium hydroxide (NaOH) was mixed with 50 g sample of particle size 2-to-2.8 mm in the ratio 1:6. The mixture was left to soak for 12 hours and later heated to form a paste. The paste was placed in a preprogrammed furnace and carbonized at 500°C for 1 hour in an inert atmosphere. The sample was allowed to cool to room temperature overnight. It was then removed, neutralized with HCl and washed with distilled water till the pH was constant. Then it was dried at 105°C in an oven and later removed to cool at room temperature. The carbon produced was sieved with 106 µm, put in an air tied bottle labeled FPMNaOHAC. This procedure was repeated with Na₂CO₃, H₂SO₄, H₃PO₄, and NaCl neutralizing with HCl and NaOH as appropriate. The end products were put in air-tied containers and labeled as FPMNa2CO3AC, FPMNaClAC, and FPMH2SO4AC respectively and collectively called chemically prepared fluted pumpkin mesocarp activated carbon (CFAC).

S/N.	Sample code	Short form	Method used in activation process					
1	FPMAC300	FAC1	Using a Pt crucible at 300°C in inert atmosphere.					

Table 1: List of various carbons prepared by physical and chemical activation methods

S/r	N. Sample code	Short form	Method used in activation process
1	FPMAC300	FAC1	Using a Pt crucible at 300 ^o C in inert atmosphere.
2	FPMAC400	FAC2	Pt crucible at 400°C in inert atmosphere.
3	FPMAC500	FAC3	Pt crucible activated at 500°C in inert atmosphere
4	FPMAC600	FAC4	Pt crucible activated at 600°C in inert atmosphere
5	FPMAC700	FAC5	Pt crucible activated at 700°C in inert atmosphere
6	FPMH ₃ PO ₄ AC	FAC6	Chemical method with H_3PO_4 activated at $500^{0}C$.
7	FPMNa ₂ CO ₃ AC	FAC7	Chemical method using Na_2CO_3 and 500^0C .
8	FPMH ₂ SO ₄ AC	FAC8	Chemical method using H_2SO_4 at $500^{0}C$
9	FPMNaOHAC	FAC9	Chemical method using NaOH at 500°C
10	FPMNaClAC	FAC10	Chemical method using NaCl at 500 [°] C

FPMAC: Fluted pumpkin mesocarp activated carbon; PFAC: Physically prepared activated carbon (FAC1- FAC5); CFAC: Chemically prepared activated carbon (FAC6 - FAC1

Determination of Ph and Conductivity

The pH and conductivity were determined according to the method of ASTM D3838-80 with slight modification as follows; 1.0 g of each carbon was weighed and transferred into a 250 ml beaker and 100 ml of distilled water was added and stirred for 1 hour. Samples were allowed to stabilize and then pH measured using an electronic pH/Conductivity meter, Jenway 430 Model. The same samples were further used for electrical conductivity (EC) of the AC_S and results read off in µS (Toles et al., 1998).

Determination of Iodine Number

This was done according to the ASTM D 4607-94 method as modified by Okuo and Ozioko (2001). 0.1 M sodium thiosulphate solution was titrated against 20.0 ml carbon sample free aliquot solution (prepared by centrifuging 0.5 g of the AC sample in 25.0 ml of 0.097 M iodine solution). Freshly prepared starch solution (5ml) was used as indicator.

Similarly the quantity of thiosulphate needed to titrate 20.0ml of the blank solution was determined. Each titration was carried out in triplicate and the average titre used in calculating the iodine value (IV) using equation 1:

$$I.V. = \frac{v - x}{y} x \frac{v}{w} x M (126.9) mg of Iodine / g of carbon sample \dots (1)$$

Where x = volume of the thiosulphate used for carbon free aliquot, y = volume of thiosulphate used for blank solution, W = weight of sample, M = molarity of iodine solution used, and V = volume of iodine solution used for titration.

Pore (Void) Volume Determination

Each AC sample (1 g) was immersed in water and boiled for 15 mins. in order to displace air from the pores. The samples were superficially dried and reweighed. The pore volume was calculated as increase in weight (∂w) divided by the density (ℓ) of water at 20°C using equation 2

$$\frac{\partial w}{\ell_{120}} = pore \ volume$$
.....(2)

Ash Content Determination

Ash content determination was done according to the ASTM D2866-94 method. Dry AC sample (1.0g) was placed in to a porcelain crucible and transferred into a preheated muffle furnace set at a temperature of 1000°C. The furnace was left on for one hour after which the crucible and its content was transferred to desiccators and allowed to cool. The crucible and content was reweighed and the weight lost was recorded as the ash content of the AC sample (W_{ash}). Then the % ash content (dry basis) was calculated from the equation.....5

Where w_0 is the dry weight of carbon sample before ashing.

Moisture Content Determination

Moisture content was determined according to ASTM 2867-99. Each AC sample (2g) was weighed and dried in a furnace continuously. The drying sample was constantly reweighed at a 10 mins interval until a constant weight (W_p) was obtained. The crucible and its content was retrieved and cooled in desiccators. The difference in weight (∂W) was recorded and the moisture content (MC) calculated from equation...4 as loss in weight on drying divided by initial weight of carbon multiplied by 100.

$$MC = \frac{(2 - W_p)}{W_o^1} x \, 100 \, \dots \, (4)$$

Where W_p = weight of Carbon retrieved from the furnace and W_o^i = initial dry weight of the AC sample.

Fixed Carbon Determination

By assuming that the sulphur content was negligible in all cases the fixed carbon content (FC) was given as:

 $FC = (Y_{ch}-U-VC-Ash)/Y_{ch}(\%)....(5)$

Where, VC = Volatile Content (%)/, Ash = ash content (%), U = Moisture content (%)

 Y_{ch} = Carbon yield (%)

Carbon Yield

The total yields were determined after sample processing in terms of raw material mass. The dried weight, W_o of each pre-treated sample was determined using Metler balance and the carbon yield calculated as $Y_{ch} = \frac{W}{W_0} X 100......$ (6)

Where Y_{ch} = Carbon yield (%); w = dried weight of AC prepared; W_o = pre-treated sample used in the carbonisation and activation processes

Bulk Density and Porosity Determination

Where r = radius of cylinder, h = height of cylinder, $M_s = mass$ of cylinder, $G_s = specific gravity$, $P_w = density$ of water. The volume of void (V_v) was obtained as

The bulk density and porosity were calculated as

Bulk density
$$(B.d) = \frac{mass \ of \ carbon \ sample}{volume}$$
(9)

Adsorption Experiment

The adsorption capacity, which was expressed in terms of percent phosphate adsorbed, was carried out according to method by Ochonogor, (2005). Standard phosphate solutions were prepared by dissolving 1 g of anhydrous potassium phosphate in distilled water. Seventy five millilitre of 0.5 g L⁻¹ of the potassium phosphate solution were placed in five 100 ml bottles each time with adsorbent doses of 2 gL⁻¹. The contents were shaken in a vertical shaker for 3 hours to attain equilibrium concentration. After equilibrium the contents of the bottles were filtered through Whatman No. 1 filter paper and analyzed for residual phosphate concentrations (APHA *et al.*, 1998). The experimental procedure was repeated at different phosphate concentrations and percent phosphate calculated as change in phosphate concentration divided by initial concentration multiplied by 100.

Determination of Energy values of carbons

The energy values were determined by bomb calorimeter, Parr, model 1281 following the standard method ASTM D1989 – 95.

STATISTICAL ANALYSIS

Data obtained from this study was analyzed using SPSS version 10.0. Data for this study was reported as mean of triplicate analysis where appropriate. Difference in means was calculated using analysis of variance (ANOVA) at 0.05 level of significance.

RESULT AND DISCUSSION

Table 1 shows the list of activated carbons (ACs) prepared from fluted pumpkin mesocarp (FPM) and method of activation of each FAC_1 to FAC_5 were prepared by physical activation at same temperature as temperature of carbonization of biomass while FAC₆ to FAC₁₀ were chemically activated using, H₃PO₄, H₂SO₄, NaOH, Na₂CO₃, and NaCl. The temperatures of carbonization and activation were carefully selected to maximize development of carbon molecular architecture. It was however observed that charring occurred only in cases where the atmosphere was partially inert. Carbonization was most preferred at 500°C and thus this was chosen as activation temperature for all CFACs. The range of changes in the properties of the carbons depends on the type of raw materials used, the way of obtaining them, the conditions of activation process (Buszek et al., 2000). All carbons characterized were of a particle size of 100 μ m – 300 μ m using same raw materials and keeping time of activation constant throughout while varying temperature and activation method. The finer the particle size the better the access to the surface area and the faster the rate of phosphate adsorption of AC. This agrees with the work of Collin and Yii (2008). In vapour phase systems this needs to be considered against pressure drop, which will affect energy cost. Careful consideration of particle size distribution can provide significant operating benefits (Gimba and Musa, 2007).

It was noticed that the changes in characteristics of various activated samples were due to activation processes, activating reagent and temperature in accord with similar previous findings (Jagtoyen, 1998; Buszek *et al.*, 2000).

The pH of PFAC_s were generally acidic and range from FAC₃ (6.3) to FAC₄ (6.8) except FAC₅ (7.6) that was nearly neutral. The pH values of CFACs where generally alkaline (7.6 – 8.7), except FAC₇ (6.2) acidic. Carbons of pH 6-8 are useful for most applications (Okiemmen *et al.*, 2007; Khadija *et al.*, 2008). Therefore all ten ACs could be acceptable for most applications involving adsorption from aqeous solutions. It should be noted that distinctly acidic ACs are not desirable (Ahmedna *et al.*, 2006). Therefore in terms of pH, physical activation does not favor preparation of activated carbon from fluted pumpkin mesocarp. Too high pH indicates much contamination while too low pH indicates that acid wash was incomplete.

The conductivity for both PFACs and CFACs showed similar trends as pH, even-though, it was observed that the conductivities of CFACs were higher than those of PFACs. The conductivity test is important because it shows the presence of leachable ash which is considered an impurity and undesirable in AC. Good conductivity in ACs ranges from 51.85 μ S to 70.75 μ S (Khadija *et al.*, 2008). In comparison with commercial carbons most carbons prepared in this study exhibited high conductivity values thereby suggesting that acid and water wash may not have been able to reduce leachable ash to levels observed in commercial carbons. This could be due to presence of

substantial amounts of water soluble minerals remained in the ACs. Such high leach-able mineral content are unacceptable especially, for uses such as commercial sugar decolonization and water purification since ash is considered an impurity. Ash content indicates the quality of an activated carbon. It is the residue that remains when carbonaceous portion is burned off. The ash consists mainly of minerals such as silica and oxides of aluminum, iron, magnesium, and calcium (oxides). Ash contents of PFACs were generally higher than those of CFAC_S with FAC₁₀ (10.9) having lowest ash content while FAC₅ (18) had the highest ash content amongst all ten AC samples.

The iodine number is the amount of iodine, in milligrams, adsorbed per gram of carbon when the equilibrium concentration (C_e) of iodine is 0.02 M. It has been established that the iodine number in mg/g gives an estimate of the surface area in m²/g (Gergova *et al.*, 1994), and measures the porosity for pores with dimensions between 1.0 - 1.5nm (Collin *et al.*, 2006). The removal of iodine by the activated carbons is related to their porosity characteristics which determine the degree of accessibility of these molecules. A lower iodine number can be ascribed to the presence of pores narrower than 1.0 nm, which make up most of the structure of these carbons (Khadija *et al.*, 2008). For carbons prepared by physical activation, the mass of iodine absorbed was generally low (92 to 261 mg/g), whereas carbons prepared by chemical activation exhibited a higher iodine adsorption capacity (240-268 mg/g) indicating improved porosity. These carbons contain mostly micro pores with a small contribution of mesoporosity.

It should be noted here that all ten carbons samples showed (FAC₁ to FAC₁₀) lower iodine values implying lower surface areas than typical range of 500 to 1200 mg/g. AC recommended for water treatment, are to show iodine values ranging from 600 to 1100 mg/g (AWWA, 1991). Thus the ACs produced in this study are advisable for use in water treatment but could show lower efficiency if used for effluent treatment without additional treatment measures.

Bulk density is the mass of carbon that can be contained in a filter of a given solid capacity and the amount of treated liquid that can be retained by the filter cake. The higher the density the better the filterability of activated carbons. However, ACs with bulk density of 0.5 g/cm³ is adequate for decolourization of sugar (Khadija *et al.*, 2008). According to AWWA, (1991), lower limit of bulk density is 0.25 g/cm³ for granular activated carbon could be put into practical use. (AWWA, 1991) .The bulk density of all PFACs were higher than this lower limit, and generally increased from lower temperature to higher temperature, activated carbons ranging from FAC₁ (0.48) activated at 300°C to FAC₅ (0.56) activated at 700°C. FAC₄ activated showed abnormally high bulk density of 0.58 g/cm³. The bulk densities of CFAC showed similar trend as iodine number, increasing from (FAC₅ and FAC₆) to (FAC₈ and FAC₉) and finally (FAC₁₀).

Pore volume for PFACs increased in the order, $FAC_1 > FAC_2$ and $FAC_4 > FAC_3 > FAC_5$, while for CFACs pore volume increased as $FAC_7 > FAC_6 > FAC_8$ and $FAC_9 > FAC_{10}$ (Table 2). Pore volume supports the concept that porosity is better in CFACs than PFACs. Porosity determines adsorptive capacity and it could be predicted that CFACs will perform better in adsorption than PFACs. Larger pore volumes are advantageous in removing larger molecules from aqueous media, therefore CFACs showed better potentials of removing larger molecules from aqueous media than PFACs.

The carbon yield for PFAC ranged from 26 to 31% while CFAC showed a range of 23-37% carbon yield. These values are typical range for most AC prepared from agro waste. The highest

fixed carbon amongst PFACs was observed for FAC₃ (70.6) while FAC₂ (60) was the lowest. Among CFACs fixed carbon ranged from FAC₆ (64) to FAC₁₀ (68). There was a wider range in fixed carbon values for PFAC than CFAC.

Moisture content tended to be unusually low for all FPMAC showing that these carbons were properly prepared and handheld. It should be noted that when exposed to air the AC are capable of adsorbing moisture from atmosphere. This normally could lead to high moisture content.

The energy values of the ACs carbons were in the order: $FAC_6 > FAC_5 > FAC_1 > FAC_3 > FAC_4 > FAC_7 > FAC_8 > FAC_2 > FAC_{10} > FAC_9$. Energy values indicate how close to carbon (allotrope graphite) the activated carbon is. Higher energy values indicate good carbonization and good activation process. Higher energy values show better quality activated carbon. Values for both PFACs and CFACs compared well but were all less than energy value of commercial activated carbon.

All PFAC showed increased phosphate adsorption capacity as contact time increased. Adsorption capacities ranged from 31.33 ± 1.15 % as in FAC₂ at a contact time of 2 hours to 58.67 ± 1.12 % as in FAC₅ at contact time of 6 hours. The rates of phosphate adsorption with contact time for PFACs and CFACs are shown in figure I and 2 respectively. Figure 1 further supports the claim that FAC₅ had better adsorption characteristics. The rates take similar shapes primarily because the adsorption condition , adsorbate and adsorbent were identical. However, the rates all seem to level off starting from contact time of 4 hours. This could be a result of exhausted sites for adsorption in carbons. Notice that at contact time of 6 hours the rates of time are significantly close in their value.

The phosphate adsorption capacity for CFAC ranged from FAC6 (50.60 \pm 1.15) to FAC10 (69.70 \pm 0.58). All CFAC had phosphate capacities higher than 50% after 1 hour and a general decrease at higher contact times. At all higher contact times the phosphate adsorption capacities were basically the same. At 1 hour contact time both PFAC and CFAC showed good rates of adsorption of phosphate. As time increased the rates of adsorption decreased drastically. Notice that such a decrease is highest between contact time of 1 hr and 2 hours, followed by a gradual decrease. It could be explained in terms of carbon active sites (CAS). At the beginning the active sites are many and the surfaces are still largely vacant (Gergova *et al.*, 1994). With time the CAS are consumed and so the rate of adsorption of adsorption drops, at contact time of 6 hours the rate of adsorption of phosphate drops to minimal compared to the initial phosphate adsorbed.

CONCLUSION

The present study has revealed that valuable adsorbents could be recovered from fluted pumpkin mesocarp given the renewable, cheap abundant source of this waste. Result of this study could provide activated carbon consumers with cost effective and environmentally friendly alternative sources. Though most characteristics were generally below expected standard values, activated carbons prepared by physical activation were better adsorbents than those prepared by chemical activation even though the chemically prepared carbons exhibited better characteristics. The variations in characteristics were a function of activation process, activating reagent and temperature. However the detailed chemical nature and surface of both PFAC and CFAC are important for explaining the observed differences in adsorption and characteristics.

	pН	Cond.	IV	Bd	PV	CY	FC	MC	Ash	EV	PA
FAC1	6.7	128	92	0.32	2.4×10 ⁻⁶	28	68.0	2.2	14	18050	68
FAC2	6.5	192	107	0.34	2.7×10 ⁻⁶	28	60.0	2.6	14	14990	52
FAC3	6.2	209	210	0.36	3.2×10 ⁻⁶	31	70.6	2.1	12	17408	67
FAC4	6.8	223	98	0.42	2.7×10 ⁻⁶	30	64	2.0	13	16995	65
FAC5	7.6	252	261	0.37	3.4×10 ⁻⁶	36	65.6	1.9	18	18266	64
FAC6	8.2	265	240	0.36	3.8×10 ⁻⁶	30	64.0	4.2	16	19092	50
FAC7	6.2	295	246	0.38	3.4×10 ⁻⁶	31	68.0	3.0	13	16560	48
FAC8	8.6	310	245	0.36	4.0×10 ⁻⁶	37	68.6	2.7	11	16204	59
FAC9	8.6	311	245	0.32	4.0×10 ⁻⁶	37	68.6	2.7	11	16204	47
FAC10	7.6	301	268	0.35	4.1×10 ⁻⁶	40.2	68.5	3.6	09	14186	58

 Table 2: Physicochemical characteristics of prepared activated carbons

Cond. Conductivity (μ S), IV: Iodine value (mgI/g), Bd: Bulk density (g/ml), PV: Pore volume (m³/g), CV: Carbon yield (%), MC: Moisture content (%), Ash: Ash content (%), EV: Energy value (J/g), PA: Phosphate adsorption capacity (% g phosphate), FC; fixed carbon.



Figure 1:Rates of standard phosphate adsorption at varying contact time for PFACs



Figure 2: Rates of standard phosphate adsorption at varying contact time for CFACs

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