

## Preparation and Characterization of Activated Carbon derived from Fluted Pumpkin Stem Waste (*Telfairia occidentalis Hook F*)

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Available online at: [www.isca.in](http://www.isca.in)

(Received 17<sup>th</sup> April 2011, accepted 29<sup>th</sup> April 2011)

### Abstract

*Powdered activated carbon was prepared from activated carbon derived from fluted pumpkin stem waste. Carbonisation was done at 350°C for two hours and allowed to cool at room temperature for three hours before activation with 0.3M H<sub>3</sub>PO<sub>4</sub> (Ortho phosphoric acid) and heated to 300°C for thirty minutes. Characterization of pH, bulk density, pH<sub>pzc</sub>, porosity and iodine number was conducted and compared to a commercial activated carbon. The data obtained when subjected to t-Test statistical analysis showed that, the activated carbon samples,  $t_{exp} < DF$  for the following properties, volatile matter, phenol content of the activated carbons and particle density indicating no significant difference on both activated carbon. A significant difference in the properties of moisture, pH, porosity, ash content, iodine number, carboxylic acid content, lactones, pH<sub>pzc</sub> and basic sites content were observed on the activated carbons.*

**Key words:** Activation, carbonisation, fluted pumpkin, moisture content, iodine number.

### Introduction

There is therefore a growing interest in cost effective, innovative materials and methods that will be useful in the treatment of industrial waste streams. Many agricultural by-products that are available at little or no cost for example chicken feathers<sup>1</sup>, cassava waste<sup>2</sup>, rice husks<sup>3</sup> Nypah palm<sup>4</sup> have been reported to be capable of removing substantial amounts of metal ion and organic pollutants from aqueous solutions. Activated carbons are predominantly amorphous solids with large internal surface areas and pore volumes. These unique pore structures play an important role in many different liquid and gas phase applications because of their adsorptive capacity<sup>5</sup>. Activated carbon can be made from a variety of precursor materials including coal, wood, coconut shell, etc by some form of activation process<sup>6</sup>. More recently interests have been shown in the preparation of activated carbon using agricultural by- products as precursor materials<sup>7-9</sup>.

The production of activated carbon from agricultural by-products has potential economic and environmental impacts. First, it converts

unwanted, low –value agricultural waste to useful, high-value adsorbents.

Second, activated carbons are increasingly used in water to remove organic chemicals and metals of environmental and /or economic concern. Third, it will reduce the importation of activated carbon wherefore increasing our economic base in the country<sup>10</sup>.

The general process to produce activated carbon is based on carbonizing and activating the carbonaceous precursor material. Activation may be achieved physically ; a two stage process involving carbonisation of the precursor material followed by controlled gasification (steam flow, temperature, heating rate etc )of the char or chemically, in which the precursor material is impregnated with a chemical agent and then heated to a temperature of 450-700°C<sup>11</sup>. For this work absolute temperature was obtained at 350°C. Activated carbon has several important uses including solution purification such as in the clean-up of cane, beet and corn sugar solutions, removal of tastes and odours from domestic and industrial water supplies, vegetable and animal fat and oils, alcoholic beverages, chemicals and pharmaceuticals and in wastewater treatment. It

also finds uses in purification of gases, liquid phase recovery and separation processes as well as its use as catalyst and catalyst supports. For liquid phase or decolourization purposes, it is normally used in the powdered form whilst for vapour or gas adsorption, it is used in the form of hard granules. Liquid adsorption or decolourising activated carbons are usually light, fluffy powders produced from low-density materials such as sawdust or peat. Gas phase adsorption on activated carbons are hard, dense granular materials produced from high density raw materials such as coconut shells, palm kernel shells coal or coke.<sup>12-13</sup>

Fluted pumpkin (*Telfairia occidentalis* Hook F) is a creeping vegetative shrub that spreads low across the ground with large lobed leaves and long twisting tendrils<sup>14</sup>. The genus *Telfairia* cucurbitaceae comprises two species, *T. pedata* and *T. occidentalis*. *T. pedata* is grown in East Africa for its oil and protein rich seeds while *T. occidentalis* is grown in some parts of West Africa for its nutritional leaves and seeds<sup>15</sup>. Harvesting of fluted pumpkin takes place 120 - 150 days, after sowing. After harvesting, the leaves are carefully removed from the stems which are discarded as wastes. The seed contains 13% oil and is used for cooking, marmalade manufacturing and cookie formulations<sup>16</sup>.

Several workers have reported the nutritional composition, chemical characterization and functional properties of fluted pumpkin seeds<sup>17-20</sup>. Preliminary investigations showed that several tons of these waste are produced daily in market places in Nigeria but scarcely useful and therefore create environmental nuisance. The aim of this paper is to produce activated carbon from fluted pumpkin stem waste, activate and characterize the carbon. Also to compare its value with a commercial activated carbon.

## Material and methods

**Sample collection and preparation:** The fresh fluted pumpkin (*Telfairia occidentalis* Hook F) stem waste was collected from Iwofe market Rumuolumeni Port Harcourt. The stems were washed thoroughly with water, cut into smaller bits rinsed with distilled water, air-dried and later

oven dried at 105°C for 10h. The oven dried sample was carbonized to obtain the carbonized biomass. The commercial activated carbon analytical reagent (AR) was bought from a scientific store in Port Harcourt.

**Carbonization:** The carbonization of the air dried fluted pumpkin stem was carried out in the Plant Physiology and Anatomy Laboratory of the University of Port Harcourt, Nigeria, using a muffle furnace (Carbolite Sheffield England LMF 4) which allows limited supply of air. Carbonisation was done at 350°C for two hours and allowed to cool at room temperature for three hours before activation.

**Acid activation of the carbon:** A carefully weighed 25.0 ± 0.01g carbonised carbon was placed in a beaker containing 500cm<sup>3</sup> of 0.3mol/dm<sup>3</sup> ortho-phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). The content of the beaker was thoroughly mixed and heated until it formed a paste. The paste was then transferred to an evaporating dish which was placed in a furnace and heated at 300°C for thirty minutes. This was allowed to cool and washed with distilled water to a pH of 6.7± 0.12, oven dried at 105°C for four hours to constant weight and ground. It was sieved with a 106µm mesh to obtain a fine powdered fluted activated carbon which was kept in an air tight vial and used for the various experiments.

**Determination of pH.:** The standard test method for determination of activated carbon pH ASTM D3838-80 was used. 1.0g of fluted activated carbon (FAC) and commercial activated carbon (CAC) was weighed and transferred into a beaker. 100ml of distilled water was measured and added and stirred for one hour. The samples were allowed to stabilize before the pH was measured using a pH meter. Samples were run in triplicates.

**Determination of porosity/ bulk density:** A cylinder and an aluminium plate were each weighed. A sample of activated carbon FAC and CAC was put into the cylinder and reweighed. The samples were transferred into the aluminium plate and put into an oven so as to dry it to constant weight at a temperature of 105°C for one hour.

The weight of the dried samples was taken again after drying. A clean dry well corked density bottle was weighed. The bottle was filled with water, corked and reweighed, small quantities of samples of activated carbons were taken and ground to powder, sieved using 106µm and gradually put into the bottles with little amount of water and weighed again. The bulk density, dry density and porosity were calculated using the following expressions

$$\text{Bulk density} = \frac{\text{mass of wet sample}}{\text{mass of volume}} \dots\dots(3.1)$$

$$\text{Dry density} = \frac{\text{mass of dry sample}}{\text{volume of cylinder}} \dots\dots(3.2)$$

$$\text{Porosity } (\eta) = V_v/V_t \dots\dots\dots(3.3)$$

Where  $V_v$  = volume of void,  $V_t$  = total volume

The volume of void  $V_v$  was obtained by first determining the total volume of the cylinder ( $V_t = \pi r^2 h$ ) used for the experiment and also determining the volume of solid i.e. the activated carbon used ( $V_s = M_s/G_s P_w$ ).

- $r$  = radius of cylinder
- $h$  = height of cylinder
- $M_s$  = mass of solid
- $G_s$  = specific gravity
- $P_w$  = density of water

Then, volume of void ( $V_v$ ) was obtained as  
 $V_v = V_t - V_s \dots\dots\dots(1.0)$

**Determination of the amount of iodine adsorbed:** For the iodine number test Gimba and Musa<sup>21</sup> was adopted. A stock solution was prepared containing 2.7g of iodine crystals and 4.1g of potassium iodide per litre. The prepared stock solution was standardized using a standard solution of sodium thiosulphate. Into a 100cm<sup>3</sup> volumetric flask, 0.5g of the activated carbon and 10cm<sup>3</sup> of 5% v/v hydrochloric acid was introduced.

The flask was swirled until the carbon was wetted. Then 100cm<sup>3</sup> of the stock iodine solution was added and agitated at a fast speed, using an electric shaker for a period of 60minutes. The mixtures were filtered through a sintered glass

crucible. An aliquot portion (20ml) was titrated with 0.1M sodium thiosulphate using starch as indicator. The concentration of iodine adsorbed by the activated carbon at room temperature was calculated as amount of iodine adsorbed in milligrams.

$$\frac{Img}{g} = \frac{(B-S)}{B} \cdot \frac{VM}{W} \times 253.81 \dots\dots\dots(1.1)$$

Where B and S are the volumes of thiosulphate solution required for blank and sample titrations respectively. W is the mass of activated carbon sample, M is the concentration (mol) of the iodine solute, 253.81 is the atomic mass of iodine and V is 20ml aliquot.

**Determination of moisture content**

Thermal drying method was used in the determination of moisture content of the samples. 1.0g of the dried activated carbons were weighed in triplicates and placed in washed, dried and weighed crucible. The crucibles were placed in an oven and dried at 105°C to constant weight for 4 h according to the method of Rengaraj<sup>22</sup>. The percentage moisture content (%MC) was computed as follows:

$$\text{Moisture}(\%) = \frac{\text{loss in weight on drying (g)}}{\text{initial sample weight}} \times 100 \dots\dots(1.2)$$

**Determination of ash content/volatile matter:**

The standard test method for ash content-ASTM D2866-94 was used. A crucible was pre-heated in a muffle furnace to about 500°C, cooled in a desiccator and weighed. 1.0g of activated carbon samples were transferred into the crucibles and reweighed. The crucibles containing the samples were then placed in a cold muffle furnace and the temperature was allowed to rise to 500°C. It was removed and allowed to cool in a desiccator to room temperature (30°C) and reweighed again. The ash content was calculated using the equation.

$$\text{Ash}(\%) = \frac{\text{Ash weight (g)}}{\text{Oven dry weight (g)}} \times 100 \dots\dots\dots(1.3)$$

Volatile matter was calculated using the equation;

$$\text{Volatile matter}(\%) = \frac{\text{Weight of volatile component(g)}}{\text{Oven dry weight (g)}} \times 100 \dots\dots(1.4)$$

**Determination of oxygen containing functional groups:** The Boehm titration method was used for this analysis. 1.0g of the activated carbons were kept in contact with 15ml solution of NaHCO<sub>3</sub> (0.1M), Na<sub>2</sub>CO<sub>3</sub> (0.05M) and NaOH (0.1M) for acidic groups and 0.1M HCl for basic groups /sites respectively at room temperature for more than 2 days. Subsequently, the aqueous solutions were back titrated with HCl (0.1M) for acidic and NaOH (0.1M) for basic groups. The number and type of acidic sites were calculated by considering that NaOH neutralises carboxylic, lactonic and phenolic groups, Na<sub>2</sub>CO<sub>3</sub> neutralises carboxylic and lactonic groups and that NaHCO<sub>3</sub> neutralises only carboxylic groups.

Carboxylic groups were therefore quantified by direct titration with NaHCO<sub>3</sub>. The difference between the groups titrated with Na<sub>2</sub>CO<sub>3</sub> and those titrated with NaHCO<sub>3</sub> was assumed to be lactones and the difference between the groups titrated with NaOH and those titrated with Na<sub>2</sub>CO<sub>3</sub> was assumed to be phenol. Basic sites were determined by titration with HCl. Neutralisation points were known using pH indicators of phenolphthalein solution for titration of strong base and strong acid, methyl red solution for weak base with strong acid and pH together. In order to neutralize basic groups /sites, remaining HCl in the solution was back titrated with 0.1M NaOH.

**Specific gravity determination:** The relative density bottles were cleaned, dried and weighed empty. One third of the activated carbons were put into it and weighed again. The relative density bottles were filled with water and reweighed. Finally the bottles were emptied, cleaned of the activated carbon particles filled with water alone and weighed. The specific gravity was calculated in duplicate.

**Determination of point of zero charge (pH<sub>pzc</sub>):** The pH point of zero charge determination (pH<sub>pzc</sub>) of the activated carbons were carried out by adding 0.1g of activated carbons to 200ml solution of 0.1M NaCl whose initial pH has been measured and adjusted with NaOH or HCl. The containers were sealed and placed on a shaker for 24hrs after which the pH was measured. The pH<sub>pzc</sub> occurs

when there is no change in the pH after contact with adsorbent.

**Student's t-test analysis:** The student's t-Test analysis of the mean values of the surface properties of activated carbon derived from fluted activated carbon (FAC) was compared with those of commercial activated carbon (CAC). This was done by formulating a null hypothesis that; there is no significant difference between the surface properties of CAC and activated carbon derived from fluted pumpkin stem waste (FAC)

$$t = \frac{X_1 - X_2}{\sqrt{\frac{s_1^2}{N_1} + \frac{s_2^2}{N_2}}} \dots \dots \dots (1.5)$$

Where x = mean value of fluted and commercial activated carbon. N = number of measurement, S = standard deviation from the mean, DF = degree of freedom. To test the null hypothesis (H<sub>0</sub>) the experimental t-values (t<sub>exp</sub>) for any degree of freedom at confidence level of 95% is compared with the difference value (DF).

If t<sub>exp</sub> > DF, H<sub>0</sub> is rejected and it is concluded that there is a significant difference between FAC and CAC.

If t<sub>exp</sub> < DF, H<sub>0</sub> is accepted and it is concluded that there is no significant difference between FAC and CAC.

## Results and discussion

**Carbonization:** For activated carbon to have the desired properties, the temperature of carbonisation must be well controlled. The temperature must be sufficiently high to dry and volatilise all non carbon substances during carbonisation. If the temperature of carbonisation is too high it greatly affects the activity of the carbon produced. The same applies to the temperature at which the carbonised product is activated. The temperature of carbonisation and activation influences the molecular architecture of the carbonised material, leading to increase in surface area and internal pores. Disturbances in the elementary microcrystalline structure by the presence of imperfect or partially burnt off carbon layers changes the arrangement of electron cloud in the carbon skeleton. As a result, unpaired electrons appear and the condition influences the

adsorption properties of the activated carbon, especially for polar and polarisable substances. Carbonisation in most experiments were usually done at 550°C, but for fluted pumpkin, the best temperature was obtained at 350°C and above 350°C it completely ashes.

#### Surface characterization of activated carbon:

The proximate analyses as presented in table 1 showed a low amount of moisture, ash and volatile matter, indicating that the particle density is relatively small and that the biomaterial should be an excellent raw material for adsorbents to be used in column or fixed-bed reactors. Ash content can also affect activated carbon i.e. it reduces the overall activity of activated carbon. It also reduces the efficiency of reactivation, the lower the ash value therefore the better the activated carbon for use as adsorbent. The porosity of FAC and CAC are better than those of other biomaterials previously tested in our laboratory<sup>2</sup>.

Porosity describes the number of pores present in a sample. Porosity therefore enhances adsorption capacity of the adsorbent. From the table 1 above, porosity is in the order FAC > CAC.

Iodine number is a fundamental parameter used to characterise activated carbon performance. It is a measure of the micropore content of the activated carbon and is obtained by the adsorption of iodine from solution by the activated carbon sample. The micropores are responsible for the large surface area of activated carbon particles and are created during the activation process. It is in the micropores that adsorption largely takes place. Table 1 suggested that the values of iodine number obtained for FAC ( $224.90 \pm 0.50$ ) is greater than the value obtained for CAC ( $200.36 \pm 0.30$ ).

The type and concentration of surface functional groups of activated carbons was determined by the Boehm titration technique<sup>23</sup>. The surface titration method stipulates that only strongly acidic carboxylic groups are neutralized by sodium bicarbonates ( $\text{Na}_2\text{HCO}_3$ ), where as those neutralized by sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) are thought to be lactonic and carboxylic group. The weakly acidic phenolic groups only react with

strong alkali, sodium hydroxide (NaOH). Neutralization with hydrochloric acid (HCl) characterizes the amount of surface basic group's pyrones and chromenes that are present in the activated carbon. It should be noted that the Boehm titration for determining the amounts and types of surface functional groups on activated carbon has been widely used by many researchers<sup>24,25</sup>.

The pH of FAC and CAC were found to be 6.7 and 7.0 respectively. It has been reported by Ahmedna<sup>11</sup> and Okieimen<sup>7</sup> that for most applications, carbon pH 6-8 is acceptable. The bulk density is another important physical parameter especially when an activated carbon product is to be investigated for its filterability. This is because it determines the mass of carbon that can be contained in a filter of given solids capacity and the amount of treated liquid that can be retained by the filter cake. When used as the same weight per volume ratio, FAC having bulk density of  $0.77 \pm 0.02\text{g/cm}^3$  as against  $0.66 \pm 0.05\text{g/cm}^3$  for CAC will be able to filter more liquor volume before available cake space is filled. Generally, carbons with bulk density of about  $0.5\text{g/cm}^3$  are adequate for sugar decolouration<sup>7</sup>.

**Student's t-test analyses:** The surface properties of the activated carbon derived from fluted pumpkin stem waste was compared with commercial activated carbon. The statistical t-Test analyses were carried out to determine if there was any significant difference between the properties obtained for the FAC and CAC. A 95% level confidence level corresponding to 0.05 level of significance was chosen and the calculated  $t_{\text{exp}}$  values given in Table 2. From the Table 2 above, it was found that, the activated carbon samples,  $t_{\text{exp}} < df$  for the following properties, volatile matter, phenol content of the activated carbons and particle density. This shows that there is no significant difference between the mean values of volatile matter, phenol content and particle density, of the activated carbon samples derived from fluted and commercial activated carbon. There is a significant difference in the properties of moisture, pH, porosity, ash content, iodine number, carboxylic acid content, lactones, pH<sub>pzc</sub> and basic sites content of the activated carbons. In

spite of the significant differences that existed for most of the surface properties between the commercial and the fluted activated carbon, the activated carbons were very efficient and effective in the removal of phenol and chlorophenol from solution showing that, though surface properties of adsorbents play significant role in sorption, such properties also depends on the method and conditions of activation which may therefore not be the sole determining factor for adsorption as illustrated in this study

### Conclusion

The prepared activated carbon derived from fluted pumpkin stem waste (FAC) compared favourably to the commercial activated carbon (CAC). The pilot study to develop a national capability for the production of activated carbon will among several benefits contribute to measures for abating the environmental degradation caused by dumping of agricultural wastes.

### Acknowledgement

This research was conducted as a part of Ph.D dissertation in the Department of Pure and Industrial Chemistry in the University of Port Harcourt. The authors are grateful to Mr Nnamdi Tamunotroko Peter and Mr Festus of the Postgraduate Chemistry Laboratory and Geology Laboratory of the University of Port Harcourt. Mr Nickol Wekwu and Mr Graham Woji, both of the Department of Chemistry Rivers State University of Education, Port Harcourt, for their technical assistance and the use of facilities in their Laboratories.

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**Table-4.1: Physicochemical Analysis of Fluted Activated Carbon (FAC) and Commercial Activated Carbon (CAC)**

PROPERTY	FAC	CAC
pH	6.7 ± 0.12	7.0 ± 0.01
Particle density g/cm <sup>3</sup>	0.79 ± 0.01	0.63 ± 0.01
Bulk density $\rho_{wet}$	0.77 ± 0.02	0.66 ± 0.05
Porosity %	0.89 ± 0.01	0.77 ± 0.05
Moisture %	19.50 ± 0.02	16.67 ± 0.07
Ash %	22.38 ± 0.02	17.1 ± 0.01
Volatile matter %	40.15 ± 0.06	37.5 ± 0.03
Iodine number	224.90 ± 0.50	200.36 ± 0.30
Carboxylic(acidic functions)	0.08 ± 0.01	0.12 ± 0.02
Phenol	0.23 ± 0.03	0.27 ± 0.02
Lactones	1.12 ± 0.01	0.61 ± 0.03
Basic sites	0.49 ± 0.02	0.55 ± 0.02
PHpzc	6.5 ± 0.02	7.03 ± 0.01

Results are given as mean ± S.D (triplicate measurements)

FAC: Fluted Activated Carbon      CAC: Commercial Activated Carbon

**Table-2:  $t_{exp}$  values of the various surface properties of the fluted and commercial activated carbon**

Property	Carbon type	Mean	SD	df	t	Decision
pH	FAC	6.70	0.02	4	74.06	Significant
	CAC	7.00	0.01			
Particle density/g/cm <sup>3</sup>	FAC	3.12	2.52	4	0.16	Not significant
	CAC	3.50	3.22			
Porosity	FAC	0.89	0.01	4	4.13	Significant
	CAC	0.77	0.05			
Moisture %	FAC	19.50	0.02	4	141.5	Significant
	CAC	16.67	0.02			
Ash%	FAC	22.38	0.02	4	91.03	Significant
	CAC	17.10	0.10			
Volatile matter %	FAC	40.06	3.22	4	1.25	Not significant
	CAC	37.32	2.02			
Iodine number	FAC	224.90200	0.50	4	72.74	Significant
	CAC	.36	0.30			
Carboxylic	FAC	0.08	0.01	4	317	Significant
	CAC	0.12	0.02			
Phenol	FAC	0.23	0.03	4	2.00	Not significant
	CAC	0.27	0.02			
Lactones	FAC	1.12	0.01	4	22.20	Significant
	CAC	0.61	0.03			
Basic	FAC	0.49	0.02	4	300	Significant
	CAC	0.55	0.02			
pHpzc	FAC	6.50	0.02	4	41.66	Significant
	CAC	7.00	0.01			