# Physicochemical Characteristics of Biocarbons obtained from Nipa Palm (Nypa Fruiticans Wurmb) Leaves

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#### **Abstract**

Physicochemical characteristics are essential in assessing the performance indices of potential biocarbons. In an attempt to find the feasibility of developing an eco-friendly and economically sustainable adsorbent for the removal of contaminants from industrial wastewater; the physicochemical characteristics of biocarbons generated from Nipa palm (Nypa fruticans Wurmb) leaves at the same carbonization temperature with differential chemical - modifications were investigated using standard testing methods. The differentially modified biocarbons were obtained through single step pyrolysis after saturating the biocarbons with H<sub>2</sub>O, H<sub>2</sub>SO<sub>4</sub> and KOH. The domino-effects obtained show that maximum biocarbon yield of 46.6 ± 0.21 % was recorded for basic reagent surface-modified biocarbon (BAC) followed by acidic reagent modified biocarbon (AAC) with a value of  $35.1 \pm 0.19\%$ ) and physically carbonized biocarbon (PCC) respectively. The data showed that, amongst the Nipa palm derived biocarbons, the BAC had the highest fractional burn-off. The percent ash and moisture contents of biocarbons (PCC: 5.8, AAC: 4.3, BAC: 3.7 for ash content and PCC: 6.5, AAC: 6.4, BAC: 7.1 for moisture content) produced from Nipa palm leaves was relatively low; while pH values of the biocarbons produced and characterized have basic characteristics with pH between 6.41  $\pm$  0.11 to 7.81  $\pm$  0.12 which is within the acceptable pH range of 6 - 8. The density of the biocarbons was in the range of  $0.64 - 0.89 \text{ g/cm}^3$ , while porosity ranged from 66 - 79 % with BAC recording the utmost bulk density (0.89 g/cm<sup>3</sup>) and porosity (79 %). The BAC biocarbons exhibit lower attrition values and showed higher surface area. The iodine number in mg/g follows the order: BAC (814.5 mg/g) > PCC (622.8 mg/g) > AAC (431 mg/g). The overall results indicate that the type of chemical activating reagent plays a significant role in the physicochemical properties of a particular biocarbon and that impregnating the biomass of Nipa palm with KOH produced the best biocarbon amongst the activating reagents used in this work. This implies that, BAC biocarbon will be a superior adsorbent with high micropore content needed for liquid phase adsorption. The inexpensive Nipa palm leaves biocarbons has characteristics comparable to commercial granulated activated carbon.

Keywords: Nypa palm, biocarbon, physicochemical characteristics, carbonization, adsorption, chemical activation.

## Introduction

Nipa palm (*Nypa Fruiticans* Wurmb) is a monotypic monocot genus belonging to the family Palmae or Arecaceae<sup>1</sup> that grows in the soft muddy banks of brackish tidal portions of rivers; and forms the transition between mangrove and freshwater swamps. The Nipa palm is the only palm that can be found in most tropical mangrove systems<sup>2</sup> and in the Niger Delta it has become an invasive species. It is rapidly invading the mangrove ecosystem and gradually eliminating the mangrove floral species such as *Rhizophora spp.* and *Avicennia Africana*, which normally provide the people with fuelwood, poles, tannin and offer excellent environment for shelter, nurseries and breeding grounds for fish, oysters and clams. This treat to the mangrove environment by the invading Nipa palms has been a source of concern to the people who depend on the mangrove resources for their socio-economic livelihood<sup>3</sup>.

Nipa palm is regarded as an invading palm in Nigeria due to

limited understanding and fewer researches on the uses, characterization and mass commercial utilization of the nipa palm; however, local people harvest several hundred metric tons per annum of nipa palm trees for various traditional uses. This versatile palm has historically provided useful products such as leaves for roof thatching, leaf petioles as floats; fronds for fish poles; and the leaflet midribs for ropes. The dried petioles and stalks are used for fuel in firewood-scare regions. Petioles have also been chopped and boiled to obtain salt. Young leaflets are used as cigarettes wrappers; older ones to weave hats, umbrella, raincoats, baskets, mats, and shopping bags. However, local communities may be looking for other lucrative alternatives because nipa palm is available in all seasons and frequency of harvest does not affect the growth of the palm. Nypa palm also produces large quantities of sugar-rich sap that can be used for bioethanol production<sup>4</sup>.

The Food and Agricultural Organization of the United Nations (FAO) described Nipa palm as a non-endangered and

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underutilized palm not only in South Asia<sup>5</sup> but also in Africa.

Due to this inadequate perception, several eradication efforts have been implemented by various governments in Nigeria, but they have largely been unsuccessful.

Physicochemical properties are important factors in the specification of commercial carbons for point of use (POU) applications. Each activated carbon has its peculiar physicochemical properties which are dependent on the starting raw material and the activation method employed; which may be physical or chemical method. The physical method of activation involves carbonization of raw material in inert atmosphere and activation of the char in the presence of carbon dioxide or steam, while chemical activation, on the other hand, consists of impregnation of chemicals into the raw materials followed by pyrolysis<sup>6</sup>.

Literature survey indicates that many scientists have investigated the production, characterization and adsorption studies of biocarbons obtained from different raw biomaterials. Some of these include melon seed husk<sup>7</sup>, lapsi seed stone<sup>8</sup>, palm oil shell<sup>9</sup>, maize cobs<sup>10</sup>, cassava peel waste<sup>11</sup>, corn cob<sup>12</sup>, banana empty fruit bunch<sup>13</sup>, fluted pumpkin stem waste<sup>14</sup>, palm kernel shell<sup>15</sup>, sugarcane bagasse<sup>16</sup> and so on.

Nigeria's coastline contains one of the world's largest Nipa palm vegetation and has the potential of being converted to profitable venture. However, due to the relatively little work being published on the preparation and characterization and of Nipa palm derived biocarbons, this paper reports the physicochemical characteristics of biocarbons produced by one step pyrolysis from Nipa palm leaves.

## **Material and Methods**

**Sample Collection:** Fresh fronds of Nipa palm (*Nypa fruticans Wurmb*) were collected during low tide at the Ogbunabali waterfront along Eastern Bypass in Port Harcourt, Nigeria. The Nipa palm leaves were separated from the fronds and the stalks carefully removed. The samples were washed with plenty of water to remove surface impurities and sun dried. This was followed by oven drying at  $110^{\circ}$ C for several days. The dried leaves were grounded using a blender to fine powder, sieved to mesh size  $110 - \mu m$  to obtain the dried leaves biomass, which was used to produce the biocarbons.

**Production of Biocarbon:** About  $25 \pm 0.01$  g of the Nipa palm leaves biomass (< 110 mesh size) was placed in a clean and preweighed crucible containing some amount of distilled water to form a paste. The paste was transferred to a clean and preweighed crucible and placed in a furnace and was heated gradually to  $550^{\circ}$ C for 10 min. The mixture was allowed to remain at this temperature in the oven for 2 hrs, after which it was poured from the crucible into a bowl containing ice block crystals. The excess water was drained and the samples were

sun dried. The carbonized sample was washed, using 10% HCl to remove surface ash, followed by hot water wash and rinsing with distilled water to remove residual acid, the solids were then sun dried, followed by oven drying at  $100^{\circ}$ C for one hour and weighed. The dried sample was sieved through a 106- $\mu$ m mesh Tyler sieve and fractions < 106- $\mu$ m was collected for use. This is the carbonization step which produced the physically carbonized carbon (PCC). The sealed oven provided an oxygen-deficient condition. Under such oxygen-deficient conditions the biomass was thermally decomposed to porous carbonaceous materials and hydrocarbon compounds  $^{17}$ .

**Production of Activated Biocarbons by One Step Process:** The activated carbons were prepared by a one step pyrolysis method<sup>18</sup>. The activated carbons produced were called (i) Acidic reagent modified carbon (AAC) and (ii) Basic reagent modified carbon (BAC).

Acidic Reagent modified Carbon:  $25 \pm 0.001$  g Nipa palm leaves biomass was placed in a beaker containing 50 ml of 10% (3.27 M) H<sub>3</sub>PO<sub>4</sub> solution. The content of the beaker was thoroughly mixed and heated to form a paste. The paste was transferred to a clean and pre-weighed crucible and placed in a furnace and was heated gradually to  $450^{\circ}$ C for 10 min. After activation, the mixture was allowed to cool to room temperature and weighed. The pyrolyzed carbons were washed, using 10% HCl to removed surface ash, followed by warm water. Rinsing was done with distilled water to removed residual acid until a neutral pH was achieved. The sample was then dried in an oven at  $110^{\circ}$ C overnight. The final product was ground and sieved through a 150-μm mesh Tyler sieve. Fractions < 150-μm were collected and kept in an air-tight container for used. Washing was complete when a pH of  $7\pm0.5$  was ascertained 17.

Basic Reagent modified Carbon: 25 ± 0.001 g Nipa palm leaves biomass was placed in a beaker containing 50 ml of 10% KOH solution. The 0.9 M standard solution was made by adding 49.93 grams of KOH into 1000ml distilled water. The content of the beaker was thoroughly mixed and heated to form a paste. The paste was transferred to a clean and pre-weighed crucible and placed in a furnace and was heated at 450°C for 10 min. After activation, the mixture was allowed to cool to room temperature and weighed. The pyrolyzed carbons were washed, using 10% HCl to removed surface ash, followed by warm water. Rinsing was done with distilled water to removed residual acid until a neutral pH was achieved. The sample was then dried in an oven at 110°C overnight. The final product was ground and sieved through a 150-µm mesh Tyler sieve. Fractions < 150-\mu were collected and kept in an air-tight container for used. Washing was complete when a pH of 7±0.5 was ascertained<sup>17</sup>.

**Estimation of Carbon Yield:** The carbon yield was calculated by applying the formula<sup>19</sup>.

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$$x(\%) = \frac{m}{m_0} \times 100$$

Where x = carbon yield (%), m is mass of carbon (g) and  $m_{g}$  is mass of raw sample (g).

Estimation of Activation Burn-off: The total mass loss during activation can be obtained; this is because a starting material of mass  $m_o$  decreases to mass m during activation. This decrease is called the activation burn-off. The activation burn-off is defined mathematically as

$$f = 1 - \frac{m}{m_0}$$

**Determination of Attrition:** The attrition of the carbons was determined using a wet attrition test<sup>20</sup> with slight modification. 1.0 gram of PCC, AAC, BAC and CAC were separately added to 100 ml of acetate buffer (0.07 M sodium acetate and 0.03 M acetic acid with pH 4.8) in 250 ml Erlenmeyer flasks. The solutions were stirred for 4 h at ambient temperature on magnetic stirrer (model REMI, 2MLH) at 200 rpm. The samples were then poured onto a 150 µm mesh screen, and the retained carbon was washed with 250 ml of distilled water. After washing, the carbon was transferred to a pre-weighed silica crucible and dried at 90°C in an oven for 4 h. The samples were removed and allowed to cool in desiccators and weighed. The percentage of attrition was calculated using the formula:

$$\% \ Attrition = \frac{wt. of \ carbon \ retained \ by \ sieve \ (g)}{initial \ sample \ wt. before \ sieve \ (g)} \ x \ 100$$

**Determination of Ash Content:** The standard test method for ash content - ASTM D2866-94 was used. A crucible was preheated in a muffler furnace to about 500°C, cooled in desiccators and weighed. 1.0 gram of PCC, AAC, BAC and CAC were separately transferred into the crucible and reweighed. The crucible containing the sample was placed in the cold Muffler furnace and heated to 500°C. It was removed and allowed to cool in desiccators for 30 min and thereafter, the weight was taken. To ascertain constant weight, the process was repeated in one hour interval. The percentage ash content<sup>21</sup> is given as ratio of Weight of ash (g) and Weight of dry sample (g) multiplied by 100:

$$\% \ Ash = \frac{W_2 - W_o}{W_1 - W_o} \ge 100$$

Where;  $W_0$ ,  $W_1$  and  $W_2$  are weight of empty crucible, crucible + fresh sample and weight of crucible + ashed sample respectively with units in gram.

**Determination of Moisture Content:** Thermal drying method was used in the determination of moisture content of the samples. 1.0g of dried PCC, AAC, BAC and CAC were separately weighed in triplicate and placed in washed, dried and pre-weighed crucibles. The crucibles were placed in an oven

and dried at  $105^{\circ}$ C to constant weight for 4 h. The percentage moisture content (% MC) was computed as follows<sup>22</sup>:

Moisture (%) = 
$$\frac{(W_i - W_i)}{W_i} \times 100$$

Where;  $W_i$  = weight of carbons before drying;  $W_f$  = weight of sample after drying

**Determination of pH:** The standard test method for determination of pH of activated carbon ASTMD3838-80 with slight modification was used. The pH of PCC, AAC, BAC and CAC were determined by weighing 1.0g of sample into a beaker. 25 ml of distilled water was measured and added to the sample in the beaker and stirred for one hour. The pH meter was calibrated at pH 4, 7 and 9.2. The pH electrode was dipped into the solution and the value was measured from the meter. Samples were run in triplicates. Samples with undesirable pH were washed continuously until a pH of 7 ± 0.5 was attained.

**Determination of Porosity and Density:** 1.0 g sample of PCC, AAC, BAC and CAC were separately dispersed in 20 ml water  $(V_w)$  in a graduated cylinder with the aid of a shaker, this was further centrifuged for 5 minutes. The resulting volume of the water was read as  $V_T$  and recorded. The equation below was used for the calculation of the porosity and bulk density as the case may be given as<sup>23</sup>:

Porosity 
$$(\infty) = \frac{V_W}{V_T}$$

$$Bulk\ density = \frac{R_{aw}}{(1-x)}$$

$$R_{aw} = \frac{M_a}{V_w}$$

**Determination of Surface Area:** The specific surface area of PCC, AAC, BAC and CAC were estimated using Sears' method as modified by Alzaydien<sup>24</sup>. 1.5 g of each carbon were separately placed in 250 ml conical flasks containing 100 ml of diluted hydrochloric acid of a pH = 3. Then a 30 g of sodium chloride was added with stirring and the volume was made up to 150 ml with deionized water. The pH of the solution was adjusted to 4.0 with HCl solution. The solution was titrated with 0.10 M NaOH and the volume, V, needed to raise the pH from 4 to 9 was then recorded. The surface area according to this method was calculated by the following equation:

$$S(m^2g^{-1}) = 32(V - 25)$$

Where; V is the volume of sodium hydroxide required to raise the pH of the sample from 4 to 9. This volume was measured in

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replicate and the average value was taken for the surface area calculation.

**Determination of Iodine Number:** 5g of PCC, AAC, BAC and CAC were separately taken in 250 ml flask and 10 ml of 5% HCI was added. The flask was swirled until the carbon became wet. Then 100 ml of stock iodine solution (2.7 g of Iodine (Merck) and 4.1 g of potassium iodide (Merck) in 1 L of deionized water) was added to it and the mixture was shaken for 5 minutes on a shaker. All the samples were filtered through Whatman No.1 filter paper. Fifty ml of filtrate was titrated with 0.1 M sodium thiosulphate until the solution become pale yellow. Then 1 ml of starch indicator solution (1%) was added and titration was continued with sodium thiosulphate until the solution become colorless. A blank was prepared without adding carbon. The iodine number (IAN) was calculated from the relationship

Iodine (mg/g) = 
$$\frac{126.9 \text{ M}(V_2 - V_1)}{W}$$

Where: 126.9 is molecular weight of iodine (g/mol); M = molarity of thiosulphate solution (mol/l);  $V_1 = \text{volume of the thiosulphate (ml) used for the titration of the sample -treated aliquot; } V_2 = \text{volume of the thiosulphate (ml) used for the blank titration}$ ; W = mass of the sample used (g)

Analytical Precision and Quality Control: Care was taken during sample collection and preservation. Glassware was properly cleaned and reagents were of analytical grade. Reagent blanks, method blanks and working standards/solutions were prepared by using freshly prepared double distilled water. The reliability of experimental results was obtained by carrying out triplicate analysis and the average, recorded as mean ± standard deviation. AquaNuchar SA-1500; a premium wood based commercial activated carbon CAC) used for treatment and purification of potable water was employed to compare the performance efficiency of the biocarbons produced.

#### **Results and Discussion**

**Preparation and Activation of Carbons:** Nipa palm leaf is a lignocellulosic material, with lignin (33.8 wt %), cellulose (28.9 wt %) and hemicelluloses (23.6 wt %) as the main components<sup>25</sup>. During carbonization and activation, the thermal-degradation reactions in the inert atmosphere of the furnace converts the lignocellulosic substance into biocarbons residue and liberate most of the non-carbon elements, mainly gases and leaving behind a rigid carbon skeleton in the form of aromatic sheets and strips<sup>26</sup>.

The physical activation process can be written as

$$\underbrace{10(C_6H_{10}O)}_{\text{list no cellulos}} \xrightarrow{\Delta} \underbrace{5C_6H_8O}_{\text{elocarbon}} + 15H_2O + 4CO_2 + 8CO + \underbrace{3C_6H_{10}O_5}_{\text{Leves lucosan (tark)}}$$

The biocarbon and char formed in the absence of oxygen inside the furnace could be presented as

$$3C_6H_{10}O_5 \xrightarrow{\Delta} 8H_2O + C_6H_2O + 2CO_2 + CH_4 + H_2 + 7C$$

After pouring the mixture into the ice water bath, the carbonaceous materials undergo hydrolysis reaction with loss of volatile matter as

$$2C_6H_3O + H_2O \rightarrow H_2 + 3CO_2 + CO$$

The biocarbon react with water causing weakening of the structure of the material, loss of volatile matter, increase in elasticity and swelling of the particle<sup>26</sup>.

$$C + H_2O \rightarrow CO + H_2$$

The biocarbon was subjected to chemical activation by impregnating with H<sub>3</sub>PO<sub>4</sub> and KOH respectively.

Chemical Treatment by H<sub>3</sub>PO<sub>4</sub>: The reaction with H<sub>3</sub>PO<sub>4</sub> begins as soon as the components are mixed; the acid first attacks hemicelluloses and lignin because cellulose is more resistant to acid hydrolysis<sup>26</sup>. The acid hydrolyzes glycosidic linkages in lignocellulose and cleave aryl ether bond in the lignin. The reaction between biocarbon and H<sub>3</sub>PO<sub>4</sub> can be written as

$$H_3PO_4 + HO - cellulose \rightarrow Cellulose - O - PO_3H_2 + H_2O$$

Cellulose 
$$-0$$
  $-PO_2H_2 \xrightarrow{\Delta} PPO - C + H_2O + H_2 + CO_2 + CO + CH_4$ 

The scission of the PPO – C bonds leads to formation of a polyaromatic units and transformation of the phosphorous group to  $H_4P_2O_7$ .

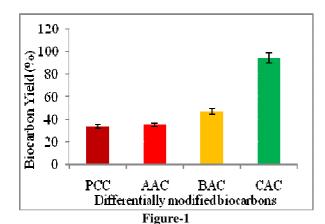
$$C + 2H_3PO_4 \xrightarrow{\Delta} H_4P_2O_7 + H_2 + CO$$

**Chemical Treatment by KOH**: The chemical treatment with KOH impregnation is to develop porosity and also increase adsorption affinity. The global proposed reaction occurring stoichiometrically between carbon and KOH is presented as follows<sup>26</sup>:

$$C + 6KOH \xrightarrow{\Delta} 3H_2 + 2K_2CO_3 + 2K$$

**Bio Carbon Yield:** Bio Carbon yield is the amount of original precursor remaining after pyrolysis and activation treatment<sup>27</sup>. Thermal-degradation of biomass usually results in solid, liquid and gaseous products. The yield of these products is dependent upon process parameters such as sample size, reaction temperature, heating time and reacting medium<sup>28</sup>. The biocarbon yield is altered, when different activation conditions

are applied. In this present study, the sample processing parameters used for the production of biocarbons from Nipa palm leaves biomass were finely divided particle size of 150 μm, reaction temperature of 550°C and heating time of 2 hr. The only process parameter varied was the reacting medium, which include using water, acidic reagent and basic reagent. The biocarbon yield for PCC, AAC, BAC and compared with CAC are presented in figure-1. The data show that the different chemical activating reagents applied in this study have different reactive efficiency. The highest biocarbon yield of  $46.6 \pm 0.21$ % was recorded for basic reagent surface-modified biocarbon (BAC). This was followed by acidic reagent modified biocarbon (AAC) with a value of  $35.1 \pm 0.19\%$ ) and physically carbonized biocarbon (PCC) respectively. The commercial activated carbon was also taken through the same procedures used for the Nipa palm derived biocarbons and the data used to compare with the properties of Nipa palm derived biocarbons as presented in figure-1. The result showed that biocarbon yield for Nipa palm derived biocarbons was good. The comparative analysis revealed that BAC is of higher quality than AAC and PCC.



Percent biocarbon yield for different biocarbons from Nipa palm leaves biomass and commercial activated carbon SA-1500  $(\overline{x} \pm s.e)$ 

**Activation Burn-off:** Burn-off is the weight loss of pyrolyzed char and represents the volatile organic matter and gases. Low burn-off is usually undesirable as they lead to low surface area which in turn may lead to a low adsorption capacity for target molecules.

The fractional burn-off increased when different activation conditions were applied. The data as presented in figure-2 showed that, among the Nipa palm derived biocarbons, the BAC had the highest fractional burn-off.

**Attrition:** The mechanical strength of biocarbon is measured by attrition and is a significant parameter for understanding biocarbon's relative loss during transportation, handling, and regeneration. A biocarbon should possess adequate mechanical strength to resist the abrasion consequential from continuous

use. The attrition measured as hardness for biocarbons derived from Nipa palm range from 70.11% to 86.44% as shown in figure-3. Hardness of commercial granulated activated carbon (GAC) is in range of 70-90%. The percent attrition of the biocarbons from Nipa palm leaves shows that BAC produces the hardest material, followed by PCC and AAC biocarbons in that order.

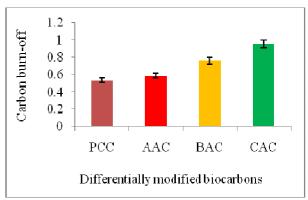
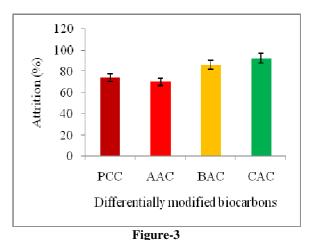


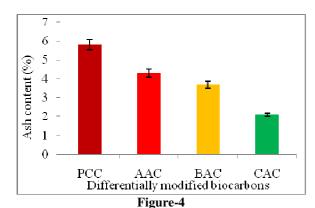
Figure-2
Fractional biocarbon burn-off from Nipa palm leaves biomass and commercial activated carbon SA-1500  $(\overline{x} \pm s.e)$ 



Hardness (attrition) for biocarbons from Nipa palm leaves and commercial activated carbon SA-1500  $(\bar{x} \pm s. \epsilon)$ 

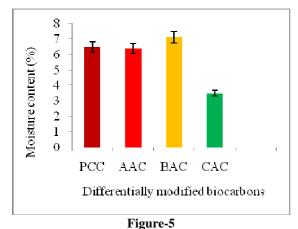
Ash Content: Ash content is the residue that remains when carbonaceous fraction of a biomass is burned off. Ash is not an attractive constituent of biocarbon and is therefore well thoughtout as impurity. Consequently, the ash content is an indicator of the quality of biocarbons. Usually materials with the lowest ash content turn out the most active products. Also, high ash content is objectionable for biocarbon because it diminishes the mechanical strength of biocarbon and its point of use capacity. The ash content of all the biocarbons produced from Nipa palm leaves was relatively low (figure-4). The ash content of PCC, AAC and BAC were 5.8 %, 4.3 % and 3.7%, and respectively.

The percentage low ash content of the biocarbons is a suggestion that Nipa palm leaves biomass could generate high biocarbon yield that are comparable to CAC.



Percent ash content of biocarbons from Nipa palm leaves and commercial activated carbon SA-1500 ( $\overline{x} \pm s.e$ )

Moisture Content: The removal of volatile materials present in a sample during thermal-degradation process is measured by the moisture content. Assessment of the data (figure-5) showed a relatively low amount of moisture content for all the biocarbons produced from Nipa palm leaves. The data showed that the moisture content were PCC (6.5 %), AAC (6.4 %) and BAC (7.1 %) respectively. The moisture content of a commercial activated carbon is between 2–5%. The percent moisture content obtained for the samples indicate that the particle density is relatively small and that the biocarbon produced from Nipa palm could be an excellent material for use in column or fixed-bed reactors.



Percent moisture content of biocarbons from Nipa palm leaves and commercial activated carbon SA-1500  $(\bar{x} + s.\epsilon)$ 

**pH:** The acid or basic nature of a biocarbon depends on its preparation and inorganic matter and chemically active oxygen groups on its surface as well as the kind of treatment to which the biocarbon was submitted. The pH of PCC, AAC, BAC and

compared with the CAC is presented in figure-6. The pH of the biocarbon derived from acidic reagent treatment was slightly lower than carbons impregnated with KOH and H<sub>2</sub>O. n general, the biocarbons produced from Nypa palm leaves have basic characteristics with pH between 6.41  $\pm$  0.11 to 7.81  $\pm$  0.12 which is within the acceptable pH range of 6 - 8 for most applications such as water treatment and decolorization<sup>29</sup>. The basic characters of biocarbons occur from delocalized  $\pi$ electrons on the condensed polyaromatic sheets. These electronrich Lewis base sites develop as oxygen is removed from the sample during thermal decomposition in the furnace. This treatment does not only removes inherent acidic groups, but also other oxygen-containing functionalities that decrease the basicity of carbon by attracting and thus localizing  $\pi$ -electrons of the condensed polyaromatic sheets<sup>29</sup>. The result showed that, the biocarbons produced from Nipa palm leaves has pH values that fell within the basic end of the pH scale and therefore these carbons could serve as potential adsorbents for organic contaminants.

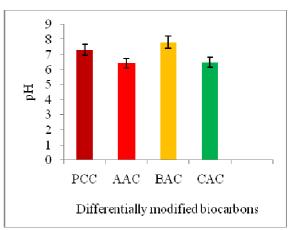


Figure-6 pH of biocarbons produced from Nipa palm leaves and commercial activated carbon SA-1500 ( $\overline{x} \pm s.\sigma$ )

**Bulk Density:** Bulk density is an essential characteristic of biocarbon. A higher density biocarbon will hold more adsorbate per unit volume. This means that higher density makes available greater volume activity and normally point toward better quality biocarbon.

The density is associated to the preparatory raw material used in the production of the biocarbon. For instance, wood based biocarbons have a lower density than coal based carbons. When two carbons differing in bulk density are used at the same weight per liter, the carbons with higher bulk density will filter more liquor volume before the available cake space is filled. As the biocarbon adsorbs compounds, the density increases and therefore this property is useful for determining the degree of saturation of exhausted biocarbons and the effectiveness of reactivation when density goes down again. The density of the

commercial activated carbon (CAC) was found to be 0.43 g/cm<sup>3</sup>. The biocarbons prepared from Nipa palm leaves compared well with the commercial activated carbon as reported in figure-7. The density of these biocarbons was in the range of 0.64-0.89 g/cm<sup>3</sup>. The data showed that BAC would filter more volume of aqueous sample if these carbons were used for filtration of the same liquor, having the higher density than others.

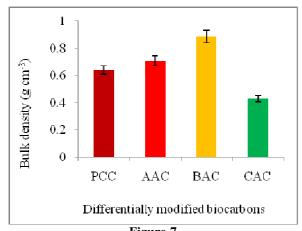
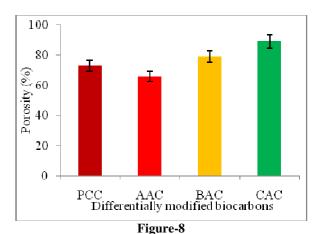


Figure-7
Bulk density of biocarbons from Nipa palm leaves and commercial activated carbon SA-1500 (\*\*\mathbb{T} \opprox \mathbb{S}. \varepsilon)

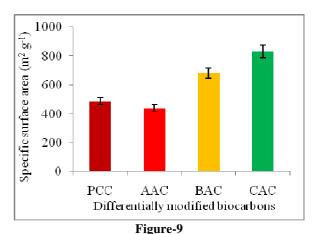
Porosity: Development of internal porosity occurs during thermal-degradation of carbon to form surfaces capable of attachment of adsorbates. The pores in biocarbons are scattered over a wide range of size and shape. According to Aziza and coworkers<sup>23</sup>, biocarbon has the highest volume of adsorptive porosity than any known material; and that pore structure in wood derived biocarbons is a recollection of the texture of the precursor wood and hence differ based on the type of wood/plant. Nipa palm is a soft wood and is expected to produce biocarbons with large pore volume which could be applicable in aqueous phase adsorption. The porosity of PCC, AAC and BAC ranged from 66 - 75 %. Since the same temperature and residence time was used for all the samples, there was no significant difference in the porosity content of all the four biocarbons. The data as presented in figure-8 revealed that BAC had the highest porosity measurement of 79 %, followed by PCC (73 %) and AAC (66 %) respectively.

**Surface Area:** The surface area of biocarbon makes the material suitable for adsorption. Large surface area increases or greatly enhances the adsorbents capacity and efficiency because it provides huge pore volumes thereby increasing the number of available active sites. Also, the larger the surface area, the higher the pore volume, porosity and this structural property lowers tortuocity of the adsorbent. In figure-9, the specific surface areas of biocarbons derived from Nipa palm leaves are 488, 441 and 681 m²/g for PCC, AAC and BAC respectively as compared to 832 m²/g for CAC. The data for surface area

showed that BAC had the largest surface area; and it is comparable to the CAC used with surface area between  $900 - 1000 \text{ m}^2/\text{g}$ .



Porosity of biocarbons produced from Nipa palm leaves and commercial activated carbon SA-1500  $(\bar{x} \pm s.e)$ 



Surface area of biocarbons produced from Nipa palm leaves and commercial activated carbon SA-1500  $(\bar{x} \pm s.e)$ 

**Iodine Number:** The iodine number in chemistry is the mass of iodine in grams that is consumed by 100 grams of a chemical substance. Iodine numbers are used to determine the amount of unsaturation in fatty acids. This unsaturation is in the form of double bonds, which react with iodine compounds. The higher the iodine number, the more C = C bonds are present in the fat. In adsorption, it the amount of iodine adsorbed by one gram of biocarbon at equilibrium. The iodine number is a measure of the porosity of a material by adsorption of iodine from a solution. A sample with a higher iodine number is of higher porosity. Figure-10 shows that the iodine number in mg/g follows the order: BAC (814.5 mg/g) > PCC (622.8 mg/g) > AAC (431 mg/g). Thus, the results obtained from this work indicate that, BAC with highest iodine number is of the highest porosity among the various samples of the Nipa palm biocarbons. This

implies that, BAC biocarbon will be a superior adsorbent with high micropore content needed for liquid phase adsorption. The iodine number obtained for BAC is comparable to CAC

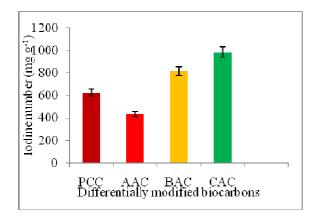


Figure-10 Iodine number of biocarbons produced from Nipa palm leaves and commercial activated carbon SA-1500 ( $\overline{x}\pm s.\varepsilon$ )

# Conclusion

Nipa palm is the only palm species that proliferate rapidly which is adapted to the mangrove zone of coastlines and estuarine habitats in many parts of the world. In the Niger Delta region of Nigeria it outgrows and eliminates the more economic mangrove forest and yet does not have any economic value for the Government at the moment. In order to find an economic value for the highly abundant and non-seasonal plant; the leaves of Nipa palm was converted to a number of biocarbons using water, acid and base and characterized for physicochemical properties by means of standard techniques. The outcome of this investigation reveals that biocarbons developed from Nipa palm leaves can be an attractive option for organic contaminants removal from aqueous solution. Furthermore, chemical modification has been shown to play a significant role in the physicochemical properties of the biocarbon and that Nipa palm leaves could be a dependable source for the production of lowcost, locally accessible, exceedingly efficient and eco-friendly adsorbents as a perfect substitute to the existing methods for eliminating organic contaminants in wastewater. Therefore, Nipa palm is a very useful mangrove species in the Niger Delta area and not an evil forest and therefore every effort should be aimed at non-eradication of plant.

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