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# Determination of Physico-Chemical Properties of Low Cost Adsorbent Produced from Activated Coconut Wastes

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# Abstract:

Activation of coconut waste to produce acid activated coconut shell (ACS) and acid activated coconut coir (ACC) was carried out. Surface properties e.g.  $P^{H}$ , Ash content (%), Moisture content (%), Bulk Density  $(g/m)^{-1}$ , Pore Volume (ml/g), Particle Density (ml/g)-1. Porosity (%), Iodine Number (mg/g), CEC (Meq/100g), Specific surface area ( $m^{2}/g$ ) and Surface charge density (Mq/100m<sup>2</sup>) of ACS, ACC were determined and found to be within the range when compared with commercial activated carbon.

Keywords: Coconut waste, activation, surface properties, characterization.

# 1. Introduction

Coconut shell and coir are materials composed of several constituents among them lignin acid cellulose which bear various polar functional groups including carboxylic and phenolic acid groups which can be involved in metal binding.Coconut shell is an agricultural based waste material and has the potential to sequester metal from solution (Gupta and others, 2003).These materials are in abundance, have high biosorption capacity, cost effectiveness and renewability as economic alternatives for water treatment and waste remediation (Gupta, 20003).

The activated carbon prepared from coconut shell and coir is highly porous amorphous solid consisting of micro crystallites with a graphite lattice and they are non-polar and cheap. The use of coconut shell and coir as biosorbent materials presents strong potential due to their high content of lignum around 35-45% and cellulose around 23-43% (Carjo and others, 2002).

Disposal of agricultural by-product is currently a major economic and ecological issue and the conversion of these agro-products to adsorbent, such as activated carbon represents a possible outlet. The activated carbon particle has mainly two types of pores existing in it; macro pores and micro pores through which adsorption takes place. The macropores provide a passage way to the interior of the particle into the micro pores but do not contribute substantially to the particle surface area. The micro pores, on the other hand are responsible for the large surface area of activated carbon particles created during the activation process. This paper therefore seeks to determine some physico-chemical properties of low cost absorbent from coconut waste.

# 2. Materials and Methods

The ripe (brown-Husk) coconut fruits *coco nucifera* from a mature tall tree of about 1.3kg per fruit were sourced from a compound in Agbor (Delta State, Nigeria). The shell and the coir were extracted properly washed with water and air-dried and finally oven-dried for 12 hours at a temperature of 80°C. Carbonization was carried out using a muffler furnace which allowed limited supply of air. The washed and sun-dried coconut shell and coir were carbonized in the furnace for 3 hours at 550°C after which the products were allowed to cool at room temperature. The cooled product was broken up to fine powder with a manual blender.

Sieve analysis was performed using the sieve screen (Shaker) to obtain final sample size of  $250um.25.00\pm 0.01g$  of the screened (250 um) fine carbonized unmodified coconut shell and the coir were carefully weighed into different beakers containing 500 ml of 0.05M H<sub>3</sub>PO<sub>4</sub> and further soaked in excess 0.05M H<sub>3</sub>PO<sub>4</sub>. The content of each beaker were thoroughly mixed and heated on a plate until it formed paste. The paste was filtered through a Whatman No. 541 filter paper and was then transferred into a crucible. The crucible was placed in a furnace and heated at 550°C for 2 hours. After cooling the activated sample (adsorbent) at room temperature, it was then washed with distilled water to a P<sup>H</sup> of 5.5 and dried on the hot plate at 100°C for up to 3 hours. The treatment of the adsorbent

with  $0.05M H_3PO_4$  solution aided in the removal of any debris or soluble biomolecules that might interact with the metal ions during the adsorption process and to open the micro pores of the adsorbent thereby making them ready for adsorption. The activated coconut shell (ACS) and activated coconut coir (ACC) were labeled accordingly

#### 2.1. Experimental Procedures

#### 2.1.1. Characterization of the Adsorbent

The activated coconut shell and coir were characterized in terms of  $P^{H}$ , ash content, bulk density, particle density, pore volume, porosity, moisture content, iodine number, cationexchange capacity (CEC), specific surface area and the surface charge density.

# 2.1.2. Determination of PH

The standard test method for  $P^{H}$  of activated carbon ASTM (American Society for Testing and Materials) D3838-80 was used.1g of activated carbon was weighed and transferred into a beaker. 100ml of distilled water was measured and added to the sample of activate carbon in the beaker and stirred for one hour. The sample was allowed to stabilized before  $P^{H}$  was measured using a  $P^{H}$  meter.

#### 2.1.3. 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.1.0g of activated sample was 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 a dedicator and thereafter, the weight was taken.

The ash content was calculated using the equation below:

Ash content (%) =  $\frac{(Ash weight on drying,g)}{Oven weight dry} x100$ 

#### 2.1.4. Bulk Density (BD) Determination

The BD was determined as the ratio of the bulk of the test samples to its bulk volume. A clean dry density bottle was weigh and its weight was recorded (D1). The density bottle was gradually filled up to the stopper (while tapping) with the samples and measured (D2). By difference (D2-D1), the bulk mass of the samples was calculated. Then a weighed (known) sample was placed in a dry measuring cylinder and tapped until the carbon samples occupy a minimum volume known as the Bulk volume ( $B_v$ ), this was read from the calibration.

The bulk density (BD) was then calculated from the equation.

$$BD = (D2 - D1)/Bv$$

#### 2.1.5. Determination of Pore Volume and Particle Density.

The specific gravity bottle method was used. The pore volume was obtained as the difference in bulk volume  $(B_v)$  and grain volume  $(G_v)$  the grain volume is determined by filling water about 10ml in a graduated cylinder and tapped, then volume quantity of samples until the gains occupy a minimum volume.

Pore volume(Pv) = Bv - Gv

The particle density was determined by the inverse of the pore volume.

Particle density  $Pd = \frac{1}{Pm}$ 

#### 2.1.6. Determination of Porosity

The porosity was calculated as the ratio of the between the particle density (Pd) and the bulk density (BD) to the particle density (Pd) which is given the equation below.

Porosity=(Pd - BD)/Pd

#### 2.1.7. Determination of Moisture Content

The standard test method for moisture content in activated carbon as in ASTM 28D67-99 was used. A crucible was dried cooled in a desiccator and weighed. The activated carbon sample was then thinly spread in the crucible and weighed (Wi).the crucible and sample was then dried in an air-circulation oven at 105°c constant weight(Wf).

Moisture content (%) =  $\frac{\text{Loss in weight on dry } ((Wi)-(Wf).x \ 100)}{\text{Initial sample weight } (Wi).}$ 

#### 2.1.8. Determination of the Iodine Number

Iodine number is a fundamental parameter used to characterize the activated carbon performance. It is a measure of the micro pore content of the activated carbon and is obtained by the adsorption of the iodine from solution by the activated carbon sample. A

standard iodine solution (0.1m) was first titrated against a standard 1.0m solution of  $N_{a2}S_2O_3.5$  H<sub>2</sub>O.(titrant) and the value was recorded.50ml of the standard solution of iodine (0.1m) was measured and transferred into a100ml conical flask mixed with 5% potassium iodide(Ki) per liter and 1g of activated carbon was weighed and introduced into the conical flask. The flask was sealed using cellophane paper and agitated using a mechanical shaker for about 1 hour. After shaking, the sample of 50ml aliquot was titrated against 1.0m  $N_{A_2}$  S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O to clear colorless solution which is the end point using starch indicator. The iodine number was calculated as the number of moles iodine adsorbed per gram of activated carbon.

The iodine number was calculated using the equation:

Iodine number (mg/g) = 
$$\frac{S_{TV} M_V x 253.8}{T_{VI} W}$$

Where;

 $S_{tv}$  is the difference between the value of the iodine solution and the titre value of the sample in iodine solution against  $N_{A2} S_2$  $O_{3}.5H_{2}O.$  $S_{tv} = T_{vc} T_{vc}$  Where  $T_{VI}$  is the 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 50ml aliquot.

#### 2.1.9. Determination of Cation Exchange Capacity (CEC)

The CEC of the coconut waste were determined by the copper bis-ethylenediamine complex method (Atef S. A, 2009). A quantity of 50ml of 1.0M copper chloride-CuCl<sub>2</sub> solution was mixed with 102ml of 1.0M ethylenediamine- $C_2H_8N_2$  (en) solution to allow the formation of the  $\{Cu(en)_2\}^2$  complex. A slight excess of the amine ensures complex formation of the complex. The solution is diluted with water to 1000ml to give a 0.05M solution of the solution.1.0g of the coconut waste was mixed with 5ml of the complex solution in a 100ml flask and diluted with distilled water to the 25ml mark and the mixture was agitated for 30minutes and centrifuged. The concentration of the complex remaining in the supernatant was determined by mixing 5ml of it with 5ml of 0.1MHC1 to destroy the  $\{Cu(en)_2\}^2$  complex, followed by adding 0.5g KI and then titrating 25ml of the complex eudiometrically with 0.1M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O (Thio solution) in the presence of starch as an indicator.

The CEC was calculated by the formula:

$$CEC (meq/100g) = \frac{M C V (x-y)}{100m}$$

Where

M= molar mass of complex C= Concentration of thio solution V= Volume (ml) of the complex taken for iodometric titration m= mass of adsorbent taken x= Volume (ml) of thio required for blank titration (without the adsorbent)

y= Volume (ml) of thio required for the titration (with the adsorbent).

#### 2.1.10. Determination of Specific Surface area (SSA)

The specific surface area of the coconut waste was determined using the ethylene glycol mono ethylether (EGME) method.1g of oven dried coconut waste was weighed and transferred into a beaker.3.0ml of ethylene glycol was measured and gently added to the sample of activated coconut waste in the beaker. The mixture was carefully swirled until uniform slurry was observed. The slurry was placed in vacuum desiccators (having anhydrous calcium chloride and ethylene glycol as desiccant with lid)

Weighing of the sample/ethylene glycol mixture was done at intervals of 10 hours until difference between two successive weights was less than 0.001g. The weight obtained at this stage was obtained after 30 hours which was used for the calculations The SSA was calculated using the equation:

Specific surface Area (SSA) =  $W_2 - W_1$ 0.000286

Where:

W<sub>2</sub> is weight of final slurry and

 $W_1$  is weight of sample used.

0.000286 is a constant representing the weight of ethylene glycol required to form a mono molecular layer on a square meter of surface  $(g/m^2)$ .

#### 2.1.11. Determination of Surface Charge Density (SCD)

The SCD (Meq/m<sup>2</sup>) was calculated from the value of CEC and SSA as the ratio of the two given by:

SCD=\_\_\_\_ SSA

# 3. Results and Discussion

## 3.1. Physico-Chemical Properties/Structural parameters of Adsorbents

The results of the various experiments and analysis performed in this work are presented in this section

| Parameters                                       | Activated<br>Coconut Shell<br>(ACS) | Activated<br>Coconut Coir<br>(ACC) |
|--|-------------------------------------|------------------------------------|
| $P^{H}$  | 7.4                                 | 7.6                                |
| Ash content (%)                                  | 1.96                                | 2.61                               |
| Moisture content(%)                              | 5.04                                | 4.76                               |
| Bulk Density (g/ml)                              | 0.55                                | 0.48                               |
| Pore Volume (ml/g)                               | 0.4                                 | 1.3                                |
| Particle Density (ml/g) <sup>-1</sup>            | 2.5                                 | 0.77                               |
| Porosity (%)                                     | 0.78                                | 0.38                               |
| Iodine Number (mg/g)                             | 334.9                               | 315.1                              |
| CEC (Meq/100g)                                   | 32.9                                | 28.9                               |
| Specific surface area (m <sup>2</sup> /g)        | 818.18                              | 755.25                             |
| Specific charge density (Meq/100m <sup>2</sup> ) | 0.04                                | 0.03                               |

Table 1: Physico-Chemical/Structural parameters of Adsorbents

The important physic-chemical/structural parameters used in this work are presented in Table 1.

The  $P^{H}$  value of activated carbon is a measure of whether it is acidic or basic. From the table above, it was observed that adsorbents were slightly basic i.e. 7.4 and 7.6 for ACS and ACC respectively. For most applications,  $P^{H}$  of 6-8 is thought to be acceptable. Tarawou T.J. (2009). Ash content affects activated carbon. It reduces the overall activity and efficiency of reactivation. Therefore, the lower the value of ash content the better the activated carbon for use as adsorbent. Tarawou T.J. (2009).

The moisture content (MC) of the ACS biomass was 5.04%, which shows that the volatile materials were removed at the high temperature. This implies that there is a low amount of ash, moisture and volatile matter, thus the activated samples should be good adsorbents for use in adsorption. Bulk density of adsorbent determines the amount of adsorbent that can be contained in a filter of a given solid capacity and the quantity of the treated liquid that is retained by the filter cake. Bulk density is affected by the raw material used and the degree of activation. From the Table 1, the bulk density for the activated samples was 0.55g/ml and 0.48g/ml for ACS and ACC respectively.

Pore volume is the sum total of the volume of all pores in one gram of adsorbent. The Table also shows applicable porosity for activated carbon samples. Porosity describes the number of pores present in a sample. Porosity therefore enhances the adsorption capacity of an adsorbent. The higher the porosity, the higher the adsorption capacity of the adsorbent. From the Table 1 porosity is of the order ACS>ACC.

Iodine number is a fundamental parameter used to characterize activated carbon performances. It is a measure of the micro-pore content of the activated carbon and is obtained by the adsorption of Iodine from solution by activated carbon sample. Iodine number is a test for porosity. A sample with a high iodine number is of higher porosity.

Cation-exchange capacity (CEC) is the maximum quantity of total cations, of any class, that a sample is capable of holding, at a given  $P^{H}$  value, available for exchange with a given solution. CEC is used as a measure of fertility, nutrient retention capacity, and the capacity to protect groundwater from cation contamination. It is expressed as miliequivalent of hydrogen per 100g of dry sample (meq/100g), or the SI unit centi-mol per Kg (cmol/Kg). The numeric expression is coincident in both units.

Adsorbents have electrostatic surface charges that attract the solution ions, and hold them. This holding capacity varies for one sample to another. The micro pores are responsible for the large surface and of activated carbon particles and are created during the activated process. It is in the micro pores that adsorption largely takes place. The performance of the ACC is probably due to the large surface area and the pore volume of structure. Large surface areas are requirements for good adsorbent. Most widely used commercial activated carbon (CAC) has a specific surface area of the order of  $800-1500^2$ /g. Tarawou T.J. (2009).

From the Table, surface area for the activated sample ACS (818.18) and ACC (755.3) were fairly high and used within the range of commercial grade. These values show that ACS/ACC will have recurrent solute solution adsorption properties.

#### 4. Conclusion

This study was conducted to optimize laboratory conditions for maximum sorption of  $Fe^{3+}$  ion from metal solutions using modified agricultural by-products of coconut shell and coir.

Acid activation of the adsorbents might have caused some modification in the adsorbent surface Properties e.g.  $P^{H}$ , Ash content (%), Moisture content (%), Bulk Density (g/ml), Pore Volume (ml/g), Particle Density (ml/g)<sup>-1</sup> Porosity (%), Iodine Number (mg/g), CEC (Meq/100g), Specific surface area (m<sup>2</sup>/g) and Surface charge density (Meq/100m<sup>2</sup>)

The results obtained showed that both chemically modified (with orthophosphoric acid) coconut coir and shell are efficient adsorbents for the removal of  $Fe^{3+}$  ion from aqueous solutions.

# 5. References

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