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# Preparation and Characterization of Activated Charcoal derived from Wood Apple Fruit Shell

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***Abstract:*** **Fine powdered activated charcoal was prepared from Wood Apple fruit shell. Carbonization was done at $300^0$C and $500^0$C for two hour and allowed to cool at room temperature. Chemical activation was achieved by impregnating the prepared charcoal with activating reagent 1N $ZnCl_2$ and heated to $300^0$C and $500^0$C temperature respectively for one hour. Characteristics of the activated carbon were determined using standard methods given by AWWA, CEFIC and also by scanning electron microscopy SEM. A significant difference in the properties of $p^H$, Conductance, Ash content, moisture, Methylene blue value, Iodine value and calorific value were observed on the activated carbons**

***Index Terms:*** **Carbonization, Activation, Characterization, Wood apple fruit shell.**

## I. INTRODUCTION

The phenomenon of Adsorption is a widely used as an effective physical method for elimination or lowering the concentration of wide range of dissolved pollutants (organic and inorganic) in an effluent. Activated carbon (AC) is the best adsorbent that can be used effectively for removal of broad spectrum of pollutants from air, soil and liquids. Activated carbon is prepared by carbonization and activation of a large number of raw materials of organic origin such as wood, coal, coconut shell and lignite (Zhonghuo, et al , 2001). Characteristics of the ACs depend on the physical and chemical properties of the raw materials as well as the methods of activation (Lua, et al, 2001). The carbonization process enriches the carbon content and introduces the porosity in the char while activation further develops the porosity and creates some ordering in the structure.

The wood apple tree is native to India. Wood Apple is known by various names like elephant fruit and monkey fruit in different countries. In India it is known as Wood Apple fruit due to its hard wooden shell. Other most common name of it is Bael fruit. The wood apple is an herb with the botanical name Limonia acidissima. Inside the hard wooden shell of fruit it consists of a brownish pulp and small white seeds .This fruit has great medicinal values. The health benefits of wood apple include (Ilango, et al, 2009) relief from constipation, indigestion, peptic ulcer, piles, respiratory problems, diarrhea, and dysentery. The vast array of health benefits attributed to wood apples are mainly due to their nutrients, vitamins, and organic compounds, including tannins, calcium, phosphorous, fiber, protein, and iron. Regular consumption of wood apple is recommended for people with kidney disorders, liver problems and also for diabetic patients. It prevents scurvy, effective for ear aches, purifies blood and removes toxins. It also acts as an excellent energy booster.

It is rich source of cellulose, proteins and lignin. The tons of Wood Apple fruit shells were discarded and send garbage as useless material and It is very essential to find applications and uses for these shells as a management of waste now days is becoming very serious environment issue. Therefore an attempt was made to prepare the low cost adsorbent from Wood Apple fruit shells. The waste hard shells are low cost, non-hazardous and environment friendly biomaterials which can be used as adsorbent in various applications.

The literature work revealed the suitability of variety of agro based materials like groundnut husk, rice husk (Shivakumar, et al, 2015; Shivakumar, et al, 2014), coconut coir pith (Shankar,et al, 2014), Orange peel (Ashtaputrey, et al, 2016), Pineapple peel (Ashtaputrey, et al, 2016) and other biomaterials to treat the industrial waste water. The present study focused to prepare the activated carbon by the carbonization of Wood Apple fruit shell and then chemical activation of carbon.

## II. EXPERIMENTAL

### *A. Sample collection and preparation:*

For the present study Wood Apple fruit were purchased from local market. The shell of the fruit was removed and cut into smaller sizes followed by washed with water. Washed Wood Apple fruit shell then sundried for fifteen days. The pieces were then dried in an oven at 1100C for an hour to remove the moisture.

*B. Carbonization:*

Carbonization of well dried sample was then carried out in a muffle furnace (Bio-technics India, Model BTI 40) by placing a sample in a silica crucible at different temperatures of 3000C and 5000C for one hour each. It was ensure that little or no oxygen was present during carbonization. The charcoal thus produced was withdrawn from the furnace, cooled, Washed with tap water and dried in an oven at 1100C and ground in a mortar by means of pestle applying moderable pressure. They were sieved through 100 – 200 mesh sieves. Then chemical activation was carried out according to the methods of Girgis (Grigis, et al, 1999) with Slight modification. 1N aqueous solution of Zinc Chloride (ZnCl2) was mixed with 25 g sample in the ratio 1:5. 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 the same temperature of initial carbonization (3000C and 5000C) for one hour. The sample was allowed to cool to room temperature overnight. It was then removed, washed with distilled water. Then it was dried at 1050C 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 and labelled as WAAC300 and WAAC500 respectively and collectively called Wood Apple fruit shell activated carbons.

Table: I List of activated carbons prepared by chemical activation method.

| Sr. No. | Sample code | Activation Method |
|---|---|---|
| **1** | WAAC300 | Chemical activation using 1N $ZnCl_2$ at $300^0$C |
| **2** | WAAC500 | Chemical activation using 1N $ZnCl_2$ at $500^0$C |

*C. Determination of pH and Conductivity:*

The pH and conductivity were determined according to the standard test methods of ASTMD3838-80. 1g of sample was weighted and transferred into a beaker and 100 ml of distilled water was added and stirred for one hour. Samples were allowed to stabilize and then pH was measured using a Toshniwal pH meter Model CL 54. The conductivity of supernatant solution was also recorded every time using Systronics conductometer Model 304.

*D. Ash content determination:*

Ash content determination was done according to ASTMD2866-94 method. 1g Dry sample (Wo) was placed into a porcelain crucible and transferred into a preheated muffle furnace set at 10000C temperature. The furnace was left on for one hour after which the crucible along with content was transferred to desiccator and allowed to cool. The crucible along with the content was reweighted (Wash) and the weight loss was recorded as the ash content of the AC sample. Then the percentage ash content was determined from the equation,

$$\text{Ash } \% = \frac{100 \text{ X Wash}}{\text{Wo}} \ \%$$

*E. Volatile matter content:*

A known quantity of sample was taken in cylindrical crucible closed with a lid. It was then heated to 925˚c for exactly 7 minutes in a muffle furnace. Then the crucible was cooled in a desiccator and weighted. Volatile matter content was determined from the equation,

$$\text{VM } \% = \frac{100[100(\text{B}-\text{F}) - \text{M}(\text{B}-\text{G})]}{[(\text{B}-\text{G})(100-\text{M})]}$$

Where B=Mass of crucible, lid and sample before heating
F=Mass of crucible, lid and contents after heating
G=Mass of empty crucible & lid
M=% of moistures determined

*F. Moisture content:*

Small amount of activated carbon sample weight was measured and then taken in a petri dish. It was spread nicely on the dish. It was then heated in an oven at a temperature of (105-110) ˚c for 1.5hr. The petri dish was left open or not covered during heating process. After heating petri dish was removed and cooled in a desiccator. After cooling the weight of dried sample was measured. Moisture content was determined from the equation,

$$\text{M } \% = \frac{100(\text{B}-\text{F})}{(\text{B}-\text{G})}$$

Where B=weight of Petri dish +original sample.
F=weight of Petri dish+ dried sample.
G= weight of Petri dish.

*G. Iodine Value:*

This was done according to the ASTMD4607-94 method as modified by Okuo and Ozioko (Ozioko, et al, 2001). 0.1M sodium thiosulphate solution was titrated against 20ml carbon sample free aliquot solution (Prepared by centrifuging 0.5g of the AC sample in 25ml of 0.1M Iodine solution). Freshly prepared 1% starch solution (5ml) was used as indicator.

Similarly the quantity of thiosulphate needed to titrate 20ml of blank solution was determined. Each titration was carried out in triplicate and the average titer volume used in calculating the iodine value (IV) using equation,

$$\text{IV } = \frac{(\text{v}-\text{x})}{\text{y}} X \frac{\text{v}}{\text{w}} X \, M(126.9) mg \ of \ Iodine/g \ of \ carbon \ sample$$

Where x=volume of thiosulphate used for carbon free aliquot.
y= volume of thiosulphate used for blank solution.
w= weight of sample.
M= Molarity of Iodine solution used for titration.
V= Volume of Iodine solution used for titration

*H. Methylene blue value/ Decolourisation power:*

Methylene blue test solution was prepared by dissolving 1500mg of pure dye in 1000ml of water using volumetric flask. The solution was then allowed to stand overnight. The solution was then checked by diluting 5ml with 0.25% acetic acid in 1lit. Volumetric flask and measuring the absorbance at 620nm for 1cm. (The absorbance must be 0.840 ±0.01).

0.1g of activated carbon was added to 10ml of methylene blue

test solution in a 50ml glass Stoppard flask. Which was then shaken till gets decolorized. The addition of 1ml of methylene blue test solution was continued as long as decolourisation occurs within 5 minutes. The total volume of test solution added till decolourisation is expressed in terms of mg of methylene blue adsorbed by 1g of activated carbon as methylene blue value of the sample by using the formula.

$$\text{Methylene blue value} = \frac{15\ X\ V}{10\ X\ M}$$

Where V=Volume of methylene blue consumed.

M=Mass in gm of activated carbon taken for the test.

*I. Calorific value:*

The energy values were determined by bomb calorimeter following the standard method ASTMD1989-95.

*J. Fixed carbon content:*

Fixed carbon content was determined by using the following equation.

Fixed carbon FC = 100 – (% moisture content+ % volatile matter + % ash content)

## III. RESULTS AND DISCUSSION

Table 1 shows the activated carbons prepared from the carbonization of Wood Apple fruit shells followed by chemical activation using 1N ZnCl2 at the same temperature of carbonization. All carbons characterized were of particle size of 106 µm. It was noticed that the change in characteristics of various activated samples were due to activation temperature.

The pH of both the ACs was slightly acidic in the range 5 to 6. The carbons of pH range 6 to 8 are useful for most applications (Okiemmen, et al, 2007; Khadija, et al, 2008). Hence the studied ACs could be acceptable for most applications involving adsorption from aqueous solutions.

The conductivity study shows the presence of leachable ash which is considered as an impurity and undesirable in AC. Good conductivity in ACs ranges from 51.85 µS to 70.75 µS. (Khadija, et al, 2008) In comparison with commercial carbons most carbons prepared in this study exhibited high conductivity values which suggested that water wash may not have been able to reduce leachable ash to level observed in commercial carbons. This could be due to presence of substantial amount of water soluble minerals remained in the ACs.

The ash content shows the amount of inorganic substituent present in the carbon. From table: 2 it was found that all the ACs has less ash content which can increase the fixed carbon value. High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity.

Volatile matter is due to presence to organic compounds present in the raw material. From the data it was clear that all the carbons have a good percentage of fixed carbon.

The moisture content of the carbons should be in between 5-8 %. All the activated carbons studied had the moisture content in this range.

Iodine adsorption is a simple and quick technique to determine the adsorptive capacity of AC, also known as iodine number often reported in mg/g (typical range 500-1200mg/g. It has been established that the iodine number gives an estimate of the surface area in m2/g (Gergova, et al, 1994), and is related to the porosity characteristics of the AC. A lower iodine number can be ascribed to the presence of pores narrower than 1.0 nm (Khadija, et al, 2008). It should be noted WAAC300 showed the high value of iodine number implying higher surface area where as sample WAAC500 has the low iodine value implying the lower surface area. AC recommended for water treatment expected to have iodine value in the range 600 mg/g to 1100 mg/g as per mentioned in AWWA, 1991. Thus the ACs produced in this study are having more efficiency for use in water treatment.

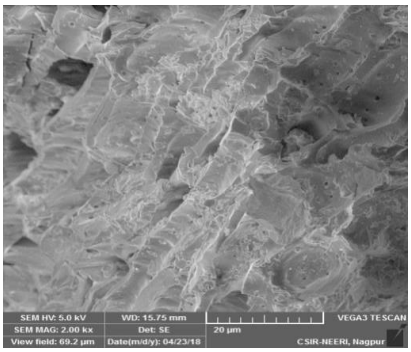
Similarly methylene blue value is an indication of adsorptive capacity of AC for molecules with dimensions similar to methylene blue and other high molecular weight substances like dye molecules. It also helps to determine the surface area which results from presence of pore sizes greater than 1.5 nm. Both ACs shows the Methylene blue value greater than 400 indicates that the carbon is good for dye adsorption.

The calorific values indicate how the activated carbon is close to allotrope graphite carbon. Higher values indicates good carbonization and good activation process resulting in good quality AC. The calorific values of ACs were found in the order WAAC500 > WAAC300. The fixed carbon values are in the range 55 to 71.

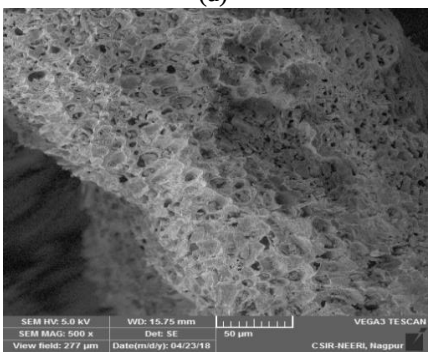
Table: II Analysis of WAAC samples.

| Sr. No. | Parameters/Characteristics | WAAC300 | WAAC500 |
|---|---|---|---|
| **1** | pH | 5 | 6 |
| **2** | Conductivity(mS) | 0.081 | 1.28 |
| **3** | Ash content (%) | 1.75 | 3.82 |
| **4** | Volatile content (%) | 36.90 | 17.48 |
| **5** | Moisture content (%) | 5.42 | 7.75 |
| **6** | Iodine Value (mg/g) | 898 | 783 |
| **7** | Methylene blue value (mg/g) | 405 | 420 |
| **8** | Calorific value (Kcal/Kg) | 6176 | 6521 |
| **9** | Fixed carbon (%) | 55.93 | 70.95 |

Scanning Electron Microscopy is the best tool for the study of porosity by the surface structure morphology characterization of Activated charcoal. SEM images of WAAC300 and WAAC500 are shown Fig. I , The SEM images clearly show that WAAC300 has non uniform cavities and pores whereas WAAC500 has large number of circular holes which appears like a honey comb structure. It clearly reveals that the WAAC300 has more surface area in comparison to that WAAC500. This transformation may be due to continuous heating at higher temperature in activation process of WAAC500.




(a)




(b)

Fig I: SEM images of WAAC (a) WAAC 300 and (b) WAAC 500

## CONCLUSIONS

The result of the study could provide activated carbon consumers with cost effective and eco-friendly alternative sources. The AC prepared at lower temperature of carbonization was found to exhibit better Characteristics than at the higher temperature of carbonization. The ACs were better adsorbents for water treatment.

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