

**PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM  
MELON SEED HUSK**

**ABSTRACT**

Activated carbon, known for its remarkable adsorption properties, is widely used in various applications, including wastewater treatment, air purification, gas and vapour purification. This study explored the production and physicochemical characterization of activated carbon derived from melon seed husks, a readily available agricultural waste product in Nigeria. The process involved carbonization of the husks followed by impregnation with calcium chloride. Physicochemical analysis was performed on the resulting melon seed husks activated carbon (MHSAC) to assess its properties. The analysis revealed favourable characteristics with a pH level of 6.52, ash content of 5.13%, moisture content of 11.36%, density of 0.453g/ml, and an iodine number of 1759.18mg/g, meeting the criteria for an effective adsorbent. These findings demonstrate the potential of melon seed husk an agricultural byproduct, as a valuable source for generating high-quality activated carbon.

**Key Words:** Activated carbon, Melon seed husk, Carbonization, Physicochemical analysis, Calcium chloride, Characterization.

**1. INTRODUCTION**

Activated carbon also called activated charcoal is a highly porous and extensively processed form of carbon, characterized by its significantly large surface area, microcrystalline, non-graphitic, and amorphous structure, resulting in a network of interconnected pores [1, 2]. Generally, activated carbon exists mainly in two forms, namely powder and granules. Granular

activated carbon (GAC) ranges from 0.2 to 5 mm and is applied in liquid and gas phase while Powder activated carbon (PAC) comes in smaller sizes (less than 0.18 mm) and applied in the liquid phase and for flue gases treatment [3]. Powdered activated carbon and granular activated carbon are commonly utilized for wastewater treatment or water purification. However, GAC is mostly used in industrial adsorption purposes due its larger particle sizes. PAC have greater exterior surface and faster adsorption capacity than GAC, but its lower particle sizes make it difficult to regenerate and requires discarding after usage limiting its industrial application [4].

The physicochemical characteristics of activated carbon encompass a range of properties that include both physical and chemical attributes. The physical attributes of activated carbon, such as its surface area and bulk density, along with chemical traits like pH, ash content, and conductivity, play a crucial role in determining its applicability and suitability for specific purposes. These physicochemical characteristics determine the performance, suitability, and applications of activated carbon in various industries. Understanding these properties is crucial for selecting the appropriate activated carbon type for specific purification or adsorption requirements [5]. Zulkarnia & Rezki (2018) [6] stated that parameters such as moisture content, ash content, and pH level have the potential to impact the adsorption characteristics of activated carbon.

Activated carbon have several applications such as wastewater treatment, air purification, water purification, gas and vapour purification, solvent recovery, environmental remediation, chemical processing, odour control, etc. The use of carbon spans back in time. During the ancient Egyptian era around 1500 BC, charcoal was utilized by the Egyptians as both a medicinal adsorbent and a purifying agent. In 1773, Scheele utilized charcoal to treat gases, and in 1786, it was employed to decolorize aqueous solutions marking the first quantification of coal's adsorptive power in the

liquid phase [3]. However, the concept of "activation" as we understand it today emerged in the 18th century. In 1822, Bussy introduced the first activated carbon produced through a combination of physical and chemical activation by heating blood with potash, which proved to be more efficient than char [7]. Commercially viable activated carbon emerged with the contributions of Swedish scientist Von Ostrejko, who obtained patents in 1900 and 1901 [3].

Despite the wide application of activated carbon, the cost of commercially available activated carbon is high and may not be easily affordable. In addition, the use of imported activated carbon may have negative environmental impacts due to transportation emissions. Activated carbon is imported into underdeveloped countries like Nigeria in large quantities and at a very high cost [8]. Using locally sourced activated carbon could be a possible solution to this problem. Local activated carbon may be more affordable and environmentally sustainable, and its properties may be tailored to local conditions. A significant amount of agricultural residue is produced each year in Nigeria, and this waste may be utilized to satisfy local needs or even be exported to promote economic development and prevent environmental deterioration. One of the agricultural wastes generated daily in Nigeria is the melon seed husk. Melon seed (*Citrullus colocynthis* L.) is a part of the cucurbitaceae family, which includes many varieties of melons. It is also known as "egusi" in West Africa. It is one of the 300 melon species found in tropical Africa. Waste (melon seed husk) is generated on daily basis in the country as a result of melon seed consumption. The economic value of melon seed husk has not been fully explored and is often overlooked. If well studied, the seed husk will not only improve the country's economy but will also reduce the amount of waste generated daily, which could result in environmental contamination and pollution. Therefore, the aim of this study was to ascertain if activated carbon can be effectively produced from melon seed husk, an everyday agricultural waste product.

## **2. MATERIALS AND METHODS**

### **2.1 Required Material**

Melon seed husk, calcium chloride, iodine solution, muffle furnace (electric furnace), oven, crucible, sieves, draining tray, aluminum dish, desiccator, beaker, graduated measuring cylinder, Whatman filter paper, pH meter, magnetic stirrer, electric blender, etc., were used for this research. The melon seed husk which was obtained from a local market served as the precursor material, undergoing carbonization and activation processes to produce activated carbon. Calcium chloride was employed as an activating agent during impregnation or activation of the melon seed husk (char). An iodine solution was used to determine the iodine number, evaluating the adsorption capacity of the resulting activated carbon. The muffle furnace and oven facilitated heat treatment processes, including carbonization, and drying of the melon seed husk. A crucible potentially aided in specific sample processing or mixing. Sieves categorized particle sizes, while the draining tray handled materials during various stages. An aluminum dish was utilized for sample preparation and weighing. The desiccator stored samples under dry conditions, and the beaker contained liquids. A graduated measuring cylinder was used to ensure accurate volume measurements. Whatman filter paper was used for facilitating filtration processes, while the pH meter measured solution or suspension acidity. The magnetic stirrer effectively mixed solutions or suspensions while the electric blender was used to crush the carbonized melon seed husk into powder form.

### **2.2 Collection and Pre-treatment of Raw Material**

Melon seed husk (MSH) was sourced from a local market in Port Harcourt, Nigeria. To prepare the melon seed husks for carbonization, it was properly washed with warm water and rinsed with

distilled water. This was done to ensure all residues were removed and there was no presence of substances that could affect the results. After washing, it was sun-dried for one week to reduce moisture content (Figure 1a).

### **2.3 Carbonization of Melon Seed Husk**

The dried melon seed husk was placed in a muffle furnace for carbonization to produce charcoal (Figure 1b). The temperature regulator of the furnace was set at 500°C. The sample was heated for 1 hour, and after which it was allowed to cool to room temperature. Thereafter, the sample was crushed using a blender and sieved using sieve with a mesh size of 850µm to obtain powdered carbon as shown in Figure 1c. The crushed char was transferred into a container and properly sealed for experiment purposes.

### **2.4 Activation of Carbon**

Chemical activation was adopted in the activation of the carbonized product (char). This method was used as it improves the porosity and adsorption efficiency of the char. To activate the carbonized melon seed husks, it was impregnated with calcium chloride by adding the calcium chloride solution into the plastic container containing the char. The container was properly sealed and left for 24 hours. After which the treated carbon was transferred into a draining tray and allowed to drain for 1 hour in order to retrieve the carbon. For removal of trace chemicals from the char, the sample was washed and rinsed five times using distilled. Thorough washing is essential to get rid of the chemical solution, which otherwise will remain in the char. It is one of the most common problems in the preparation of activated carbon using chemical activation. After washing, the sample was returned to the draining tray to remove the water. Following this, it was transferred into an oven, the temperature was set at 110°C and was allowed to bake for

about 3 hours. After baking, the activated carbon was removed from the oven and crushed using a blender.

## **2.5 Characterization**

To characterize the activated carbon produced from melon seed husk, standardized methods were used as proposed by the American Society of Testing and Methods (ASTM). The test included the determination of moisture content, ash content, pH, bulk density, and the iodine number of the activated carbon.

### **2.5.1 Determination of Moisture Content**

The moisture content of the activated carbon was determined using the oven drying technique. A clean aluminum dish was washed thoroughly and dried in the oven, then cooled in the desiccator before being weighed. 2g of the sample was put in the dish and the weight of the dish containing the sample was taken. Thereafter, it was dried in the oven at 105°C for an hour and then cooled in the desiccator. After which the dish containing the oven-dried sample was weighed. The percentage moisture content was calculated using Equation (1).

$$\text{Moisture content} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (1)$$

where  $W_1$  is the initial weight of empty crucible,  $W_2$  is the weight of crucible plus sample before drying, and  $W_3$  final weight of crucible plus sample after drying.

### **2.5.2 Determination of Ash Content**

Exactly 1.0g of the sample was weighed in a platinum crucible and recorded as  $W_1$ . The sample was transferred to muffle furnace at the temperature of 550°C for 8 hours to obtain a white ash. The platinum crucible was removed and placed in a desiccator to cool and then weighed. The

value obtained was recorded as  $W_2$ . The ash content of the activated carbon was calculated in percentage using Equation (2).

$$\% \text{ ash} = \frac{W_1 - W_2}{W_s} \times 100 \quad (2)$$

where  $W_1$ ,  $W_2$  and  $W_3$  are the initial weight of empty crucible, weight of crucible plus sample before drying and the weight of the sample used respectively.

### 2.5.3 Determination of pH

A pH meter with glass electrodes was used to ascertain the activated carbon's pH. After being turned on, the pH meter was given 15 minutes to warm up. To standardize the glass electrode, 2g of activated carbon was dissolved in 100ml of deionized water using a standard buffer with a pH of 7.0. To achieve appropriate dilution, the liquid was heated and agitated for 5 minutes using a magnetic stirrer. Then, it was filtered at room temperature using Whatman filter paper. The pH meter was then used to determine the pH of the filtered sample.

### 2.5.4 Bulk Density

A graduated measuring cylinder with a 10 mL capacity was weighed. The cylinder was topped out with the test sample (activated carbon). The test sample's weight inside the test tube or cylinder was calculated and expressed as a volume ratio. The bulk density (BD) was calculated using Equation (3).

$$BD = \frac{W}{V} \quad (3)$$

where  $W$  is the weight of sample and  $V$  the volume occupied by the sample.

### 2.5.5 Determination of Iodine Number (IN)

The iodine number of the activated carbon was determined using the ASTM D4607-94 method. This method measures the amount of iodine adsorbed by 1.0 gram of carbon when the iodine concentration in the filtrate is 0.02N. The procedure involves treating a standard iodine solution with different weights of activated carbon. The activated carbon sample was first treated with 5% hydrochloric acid (HCl), boiled, and then cooled. Next, 0.1 N iodine solution was added and stirred. The resulting solution was filtered, and a portion of the filtrate was titrated with 0.1 N sodium thiosulfate using starch as an indicator. The iodine amount adsorbed per gram of carbon (X/M) was plotted against the iodine concentration in the filtrate (C) using logarithmic axes. The procedure was repeated with different carbon masses whenever the residual iodine concentration was outside the range of 0.008 to 0.04 N. A least square fitting regression was applied to analyze the three points, and the iodine number was determined when the residual concentration was 0.02 N. The X/M and C values were calculated by the Equations (4) and (5), respectively.

$$X/M = \frac{N_I \times 126.93 \times V_I - \frac{V_I + V_{HCl}}{V_F} \times N_{Na_2S_2O_3} \times 126.93 \times V_{Na_2S_2O_3}}{M_c} \quad (4)$$

$$C = N_{Na_2S_2O_3} \times V_{Na_2S_2O_3} \quad (5)$$

where  $N_I$  is the iodine solution normality,  $V_I$  is the added volume of iodine solution,  $V_{HCl}$  is the added volume of 5% HCl,  $V_F$  is the filtrate volume used in titration,  $N_{Na_2S_2O_3}$  is the sodium thiosulfate solution normality,  $V_{Na_2S_2O_3}$  is the consumed volume of sodium thiosulfate solution, and  $MC$  is the mass of activated carbon.

### 3. RESULTS AND DISCUSSION

Carbon (char) was successfully produced from melon seed husk by carbonization process and chemically activated using calcium chloride as activating agent. The activated carbon was

characterized to determine the pH, ash content, moisture content, apparent density and iodine number. These results are discussed in the subsequent subsections.

### 3.1 Characterization of the Activated Carbon

The physicochemical characteristics of the activated carbon produced from the melon seed husks and activated using Calcium Chloride ( $\text{CaCl}_2$ ) are presented in Table 1. The characterization involved the determination of the activated carbon's pH, moisture content, bulk density, and iodine number. The pH of the activated carbon which was determined using a digital pH meter was 6.53. The ash content, moisture content, and the iodine number were 5.13%, 11.36%, and 1759.18%, respectively.

Table 1: Physicochemical characteristics of melon seed husk activated carbon

Parameters	Values
pH	6.52
Moisture content (%)	11.36
Ash content (%)	5.13
Bulk Density ( $\text{g/cm}^3$ )	0.453
Iodine Number (mg/g)	1759.18

#### 3.1.1 pH

The pH of the carbon after activation as shown in Table 1 is 6.52, which is slightly acidic. Low pH activated carbon (AC) typically has a positive surface charge due to protonation of surface

functional groups [9]. At low pH conditions the adsorption capacity of activated carbon increases. In liquid phase, as pH decreases the rate of adsorption surges [10]. This pH of 6.52 is therefore an indication that the melon seed activated carbon could be a good adsorbent in the removal of impurities from solution. Ekwierhoma et al. (2018) [11] obtained a similar pH (6.7) in their study in which they stated that this kind of activated carbon is preferable in the removal of polar organic compounds from water. Madu & Lajide (2013) [2] confirmed that activated carbon is more effective at low pH than high pH.

### **3.1.2 Ash Content**

The amount of ash present in the activated carbon is relatively minimal at 5.13%. This is important as ash content greatly impacts the overall quality and performance of the activated carbon. It is one of the major parameters that influences the adsorption properties of activated carbon. Activated carbon with high content of ash (>20%) reduces its specific surface area, potential reactivation and generates leakage of impurities. For an activated carbon to serve as a good adsorbent, its ash content should not be too high. It should be less than 20% [12]. The low ash value obtained (as shown in Table 1) is an indication of good adsorption capacity of activated carbon produced using melon husk.

### **3.1.3 Iodine Number**

One important metric that can be used to evaluate the adsorption capability of activated carbon is the iodine number. The iodine number, measured in mg/g of carbon, reflects the porosity of the activated carbon, and indicates the amount of iodine that can bind to a specific amount of activated carbon. The iodine number obtained from the test was 1759.18 mg/g (as shown in Table 1) According to the Indonesian National Standard (SNI 06-3730-1995), an effective

activated carbon should have a minimum iodine number of 750 mg/g. Budianto et al. (2019) [13] conducted a study which found high iodine numbers (769–1019 mg/g) in their activated carbons. They noted that these high values indicate that the production conditions of the activated carbons met the requirements set by the Indonesian National Standard (SNI) in terms of iodine number. This also suggests that the activated carbon has high porosity and activity level, as a higher iodine number indicates a higher degree of activation.

#### **3.1.4 Moisture Content**

The amount of water that typically attaches to activated carbon is referred to as its moisture content. According to Indonesian Industrial Standard (SII No. 0258-88), the maximum moisture level for activated carbon is 15%. It is a crucial parameter since it influences how well adsorption works. The capacity of activated carbon for adsorption could rise if there is less moisture present[6]. Aside the adsorption efficiency, high moisture is undesirable as it may increase storage and transportation costs. The activated carbon's moisture content obtained from the experiment is 11.36% which is below the 15% limit of the Indonesian Industrial Standard. This is an indication of possible good adsorption performance of the activated carbon.

#### **3.1.5 Bulk Density**

Bulk density refers to the weight of a given volume of the sample in the air, encompassing both the pore structure and the spaces between the particles [14]. The density of the activated carbon significantly influences the amount of adsorbate it can capture [15]. The higher the density of an activated carbon the greater the filtration ability [16]. For activated carbon to be used for practical purposes, the activated carbon should have a minimum bulk density value of  $0.25\text{g/cm}^3$  as

specified by the American Water Work Association [17]. As shown in Table 1, the obtained bulk density is  $0.453\text{g}/\text{cm}^3$ . This implies that the activated carbon can be successfully used in practical conduction. Powdered activated carbons needed for the removal of colour usually have bulk density that ranges from  $0.25$  to  $0.75\text{g}/\text{cm}^3$ . This value of  $0.453\text{g}/\text{cm}^3$  is within the range of the bulk density for powdered activated carbon required for removal of impurities.

#### 4. CONCLUSION

This research was carried out to investigate the potential of melon seed husks, an agricultural waste, in the production of activated carbon. Activated carbon was produced from the melon seed husks by impregnation of the husks with calcium chloride after carbonization. Physicochemical analysis was conducted on the melon seed husks activated carbon (MHSAC) to determine its characteristics. The result of this analysis revealed that the pH level, amount of ash, moisture content, density, and iodine number of the activated carbon prepared from melon seed husks were  $6.52$ ,  $5.13\%$ ,  $11.36\%$ ,  $0.453\text{g}/\text{cm}^3$ , and  $1759.18\text{mg}/\text{g}$  respectively. These parameters were all within their optimal ranges, affirming the suitability of the melon seed husk activated carbon as a high-quality activated carbon material for various applications.



Figure 1a: Raw melon seed husk

Figure 1b: Carbonated melon seed husk

Figure 1c: Crushed carbonated melon seed

## Reference

1. Duruaku, J. N. (2012). Modification of surface, physical and chemical properties of activated carbons for water purification (Bachelor's thesis). Caritas University, Amorji-Nike, Enugu.
2. Madu, P. C. and Lajide, L. (2013). Physicochemical Characteristics of Activated Charcoal Derived from Melon Seed Husk. *Journal of Chemical and Pharmaceutical Research*, 5 (5) 94-98.
3. Alves, A. T., Lasmar, D. J., Miranda, I. P. A., Chaar, J. S., and Reis, J. S. (2021). The Potential of Activated Carbon in the Treatment of Water for Human Consumption, a Study of the State of the Art and Its Techniques Used for Its Development. *Advances in Bioscience and Biotechnology*, 12(6).
4. Allen, S. J. and Koumanova, B. (2005) Decolorization of Water/Wastewater Using Adsorption. *Journal of the University of Chemical Technology and Metallurgy*, 40, 175-192.
5. Ahmed, M. J., and Theydan, S. K. (2012). Physical and chemical characteristics of activated carbon prepared by pyrolysis of chemically treated date stones and its ability to adsorb organics. *Powder Technology*, 229, 237-245.
6. Zulkania, A., F., G. H., and Rezki, A. S. (2018). The potential of activated carbon derived from bio-char waste of bio-oil pyrolysis as adsorbent. *MATEC Web of Conferences*, 154.
7. Bubanale, S., and Shivashankar, M. (2017). History, Method of Production, Structure and Applications of Activated Carbon. *International Journal of Engineering Research & Technology (IJERT)*, 6(6).
8. Olafadehan, O.A., Jinadu, O.W., Salami, L. and Popoola, O. T. (2012). Treatment of Brewery Wastewater Effluent using Activated Carbon Prepared from Coconut Shell. *International Journal of Applied Science and Technology*, 2 (1), 165-178.
9. Lafi, R., Montasser, I., and Hafiane, A. (2019). Adsorption of congo red dye from aqueous solutions by prepared activated carbon with oxygen-containing functional groups and its regeneration. *Adsorption Science & Technology*, 37(1-2), 160-181.
10. Aznar, S. J. (2011). Characterization of activated carbon produced from coffee residues by chemical and physical activation (Master's thesis). KTH Chemical Science and Engineering, Stockholm, Sweden
11. Evwierhoma, E. T., Madubiko, O. D. and Jaiyeola, A. (2018). Preparation and Characterization of Activated Carbon from Bean Husk. *Nigerian Journal of Technology*, 37, 674 – 678.
12. Ayvral, C. (2009). Elimination of aromatic pollutants by catalytic oxidation on activated carbon (Doctoral thesis) University of Toulouse.
13. Budianto, A., Kusdarini, E., Effendi, S. S. W. and Aziz, M. (2019). The Production of Activated Carbon from Indonesian Mangrove Charcoal. *IOP Conference Series: Materials Science and Engineering*, 462.
14. Zhonghua, H., Srinivasan, M. P., and Yaming, N. (2001). Novel activation process for preparing highly microporous and mesoporous activated carbons, *Carbon*, 39, 877-886
15. Ravichandran, P., Sugumaran, P., Seshadri, S., & Basta, A. H. (2018). Optimizing the route for production of activated carbon from *Casuarina equisetifolia* fruit waste. *Royal Society Open Science*, 5(7)

16. Verla, A. W., Horsfall, M., Verla, E. N., Spiff, A. I., & Ekpete, O. A. (2012). Preparation and characterization of activated carbon from fluted pumpkin (*Telfairia occidentalis* Hook.f) seed shell. *Asian Journal of Natural & Applied Sciences*, 1 (3), 36-50.
17. Tchuifon, T. D. R., Anagho, S. G., Nche, G. N., and Ketcha, J. M. (2015). Adsorption of salicylic and sulfosalicylic acid onto powdered activated carbon prepared from rice and coffee husks. *International Journal of Current Engineering and Technology*, 5, 1641-1652.

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