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The synthesis of activated carbon from gmelina trunk

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Abstract

Activated Carbon is the product of charcoal impregnation process that has a higher adsorption capacity and more benefits than regular charcoal. Therefore, this study aims to produce activated carbon that meet standard. Carbonization (pyrolysis) of carbon obtained from Gmelina (Gmelina Aboreal) wood was achieved at temperature of 480 °C for 2 hours. The carbonized carbon was activated via the chemical activation process using HNO3. Characterization of Iodine Number, Ash content Moisture content, Bulk Density, Free Carbon Brunauer-Emmet- Teller (BET), X-Ray Diffraction was conducted. The result obtained from carbonized Gmelina Nitric Acid Activated Carbon was found to have iodine number of 1050 mg/g, Ash Content of 11%, Moisture Content of 8.2%, Bulk Density of 0.5%, Free Carbon of 53% and Brunur Emmet-Teller (BET) surface area of 100 m^2/g. X-ray diffraction analysis showed 91% amorphous and 9% crystallinity.

Keywords: Pyrolysis; Characterization; Adsorbent; Gmelina Trunk

1 Introduction

The production of adsorbents (activated carbons) is of high economic value due to its wide range of utilization in both domestic and industrial applications such as water purification, gas purification, metal extraction, gold recovery, pharmaceuticals, wastewater treatment, gas and filter mask air filters and compressed air filters. Activated carbon is also suitable for deodorizing closed spaces such as refrigerators and warehouses. Apart from its wide range of applicability, adsorbents are produced from locally sourced materials that are cheap, available and environmentally friendly (Mlilo *et al*., 2010; Kofa *et al*., 2017; Medellin-Castillo *et al*., 2007; Nursiah *et al*., 2023; Buhani *et al*., 2015; Chaudhuri *et al*., 2016; Calvete *et al*., 2010; Yothin *et al*., 2014). Dagde *et al*., in 2019 reported that local raw materials such as goat bones and egg shells can be utilized for the production of adsorbents capable of removing fluoride ions from water. The steady state process was in an absorption column with the capability to allow interaction between the absorbents bone char, egg char and the water contaminated with fluoride ion. They emphasized on the importance of water for plant and animal survival and the need to improve the quality drinking water to meet daily demand of billions of people (Garfi *et al*., 2016). Contaminants such as turbidity, organic matter and pathogens must be reduced to standard specification quality for drinking water to be made portable using treatments such as coagulation, flocculation, sedimentation, filtration, adsorption, disinfection and reverse osmosis (Garfi *et al*., 2016; Saitoh et al., 2014; Saeed et al., 2016; Buhani & Suharso, 2019; Ali *et al*., 2012), ceramic water filters (Lantagne *et al*., 2017), cation and anion exchange resins (Wu *et al*., 2008), biological treatment (Rodrigues *et al*., 2014), separation method (Pankaj & Joy, 2009) . these methods are chosen depending on the areas of concern by the researcher and what is meant to be achieved at the end of the treatment. According to World Health Organization in 2004, the quality and accessibility of drinking water are of paramount importance in a bid to control the risks to public health. When the concentration of fluoride in water exceeds 1.5 mg/mol., it becomes a contaminant which causes health challenges like dental fluorosis, skeletal fluorosis, muscle

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fiber generation and other health issues (Delgadillo-Velasco *et al*., 2017). Other symptoms are stained teeth, bone diseases, tooth decay, stooped backs, crooked hands and legs, non-skeletal fluorosis such as harmful effects to erythrocytes, ligaments, spermatozoa, thyroid glands and destruction of filaments in the muscles leading to muscle weakness. Also, the gastrointestinal system is affected causing gastric irritation such as nausa, vomiting and gastric pain (Wong & Stenstrom, 2017). This fluoride could be found naturally in groundwater when there exist slow dissolution of rocks or minerals which contains sellaite (MgF_2), fluorspar (CaF_2) and cryolite (Na_3AlF_6) (Delgadillo- Velasco *et al*., 2017).

Furthermore, anthropogenic activities can contaminate water sources (groundwater) with fluoride, the use of pesticides and fertilizers containing fluoride (Tovar-Gomez *et al*., 2013) or through the discharge of industrial effluents into water bodies (Ramya *et al*., 2019; Buhani, 2011) as well as industrial processes such as cement, bricks, electronic manufacturing, aluminum smelting and refining can contaminate groundwater with fluoride (Snyder, 2014; Buhani *et al*., 2019; Wong & Stenstrom, 2017). Major water contaminants such as dyes that originate from textile industries can be reduced by oxidation (Vaiano *et al*., 2015; Collazzo *et al*., 2012). Research has established that the main source of water supply for human consumption in several countries is via the groundwater (Dagde *et al*., 2019). Presently, there is a report in twenty six countries around the world of high fluoride content in drinking water (Wong & Stenstrom, 2017). In gas processing industries, triethylene glycol (TEG) absorbent just like adsorbent is utilized for the absorption of water content of the natural gas to meet pipeline specification standard for transmission, processing and storage of sales gas (Wosu & Aworabhi, 2025; Wosu & Ikenyiri, 2024; Wosu *et al*., 2023a; Wosu *et al*., 2023b). Also, monoethanolamine (MEA) is utilized in CO^2 absorption (Dadet *et al*., 2025).

Adsorbents produced from the pyrolysis of sewage sludge is utilized in the removal of metallic materials such as copper, zinc and cadmium from wastewater (De Filippis *et al*., 2013; Olugbenga *et al*., 2015). Activated carbon produced from olive stones are utilized in gold recovery.

This research is focused on the production of novel adsorbent (activated carbon) from gmelina trunk that can be utilized in adsorption processes. Figure 1 shows activated carbon in powder, granules and pellets form. While Figure 2 is a picture of a gmelina tree and wood.

Figure 1 Powder, Granules, Pellets Activated Carbon

Figure 2 Gmelina tree and Gmelina wood

2 Materials and Method

2.1 Materials

The materials and apparatus utilized in this research are gmelina trunk obtained from Rivers State University, Port Harcourt, Nigeria, pyrolytic reactor equipped with condenser, measuring cylinders, heating mantle, desiccators, crucibles, funnels, filter papers, weighing balance, retort stand, spatula, thermocouple, density bottle, crusher, moisture cans, muffle furnace, conical flask, pipette, measuring cylinder, burette, glass rod and wash bottle.

2.2 Methods

The research methodology is experimental and the procedures adopted includes;

2.2.1 Preparation of the Gmelina Trunk

The raw material was washed several times to remove the dirt and impurities present on the materials, then the washed materials was dried in an oven at a low temperature of 60 °C to 65 °C for 24 h to remove the moisture content. The dried material was then store in a dry container in desiccators until needed. The dried raw gmelina wood was finally sent for ultimate and proximate analysis.

2.2.2 Carbonization of Gmelina Wood

An amount of gmelina Wood was measured using top loading balance, and then cut into smaller fractions, washed and dried. This initial measurement was done to determine the amount of gmelina Wood required for treating an amount of underground water. The dried smaller fractions of gmelina Wood were passed to the pyrolyzer for heating up to a temperature of 400 ^{o}C to 500 ^{o}C for two hours. At time greater than two hours, the product was cooled to room temperature and the pyrolyzed gas leaves through the condenser. The major product (solid) was then passed into a crusher for size reduction and a separation technique called sieving was carried out to obtain 300micron sized particles of the product.

2.2.3 Chemical Activation of Carbonized Gmelina Wood

The chemical Activation of carbonized gmelina Wood was done using Nitric acid HNO_3. The carbonized gmelina Wood was weighed into a beaker containing dilute Nitric acid HNO_3. A continuous mixing of the reactant mixture (carbonized gmelina wood and acid) was carried out until it turns into paste and it was then transferred into crucibles for heating in a muffle furnace to a temperature of about 500 ^{o}C for two hours. After heating, the mixture was then cooled to room temperature and washed with distilled water to pH of 7. This mixture was then dried in oven to 105 ^{o}C for at least three hours to obtain a final particle. This particle was called acid activated carbonized gmelina Wood and was used as the adsorbents.

2.2.4 Characterization of Activated Carbonized Gmelina Wood

The produced adsorbent was characterized using ASTM method as written in the paper work of Ademiluyi *et al*, (2009). The characterization of the activated carbon involves the determination of properties such as bulk density (gm^{-3}), pore volume (cm^3) percentage burnt off, moisture content, ash content (%), particle size, benzene adsorption, absorptive capacity (mg/g), methylene blue and iodine number (g of iodine/kg of carbon), Scanning Electron Microscope SEM, X-Ray Fluorescence XRF, and BET.

Scanning Electron Microscope (SEM)

Scanning Electron Microscope was used to capture the morphological structure of the Gmelina wood. The samples were prepared in carbon black stack material on sample holders as nitrogen inert gas was applied to remove some unbound activated carbon from the black carbon stack surface of the sample holder, when the energy (electron) became developed, the pointer starts to capture at different magnification and resolution of the surface texture.

It was also used to determine the surface texture and porosity of the sample (gmelina activated carbon) in secondary electron imaging mode. The unloaded shows that the presence of an organized pore region in the fibro vascular bundle wall after the activation that permits the diffusion of the contaminants in a faster way to the inner regions of the produced gmelina/KOH. Amorphous structures without definite shapes and a loaded sample has also been investigated.

X-Ray Florescence XRF

XRF analysis was carried out to find out the type and concentration of elements crystallographic structure. The result indicates that elements were detected which helps to improve the adsorption process when the AC was used as adsorbent. The result shows the element analysis of AC produced from gmelina wood from XRF method. The data indicate concentration of different elements present in the sample, which may be used for the adsorption spectra were analyzed and concentrations of the element present in the samples were obtained.

- Moisture Content Determination

This method was used to determine the percentage of water in a sample by drying the AC is a constant weight. Scale out 5g of the activated carbon in a Petri dish. Allow at 105•C for half hour, continue at 30 minutes intervals until a stable weight was obtained. The moisture weight can be calculated as wet basis and as dry basis.

$$\%M_c \text{ wet basis} = \frac{Wi - Wf}{Wi} \quad \ldots\ldots\ldots\ldots\ldots\ldots(1)$$

$$\%Mc \text{ dry basis} = \frac{Wi - Wf}{Wf} \quad \ldots\ldots\ldots\ldots\ldots\ldots(2)$$

where W_i is initial weight (g), W_f is the final weight after drying (g) and M_c is the moisture content (%)

- Ash Content Determination

A crucible was washed and dried in a hot air oven for 30 minutes at a temperature of 105•C. After heating, it was then cooled in a desiccator for 30 minutes. The crucibles were weighed using analytical balance. So that 1g of sample was weighed into the crucibles and its weight was recorded. It was transferred to a muffle furnace maintaining a temperature of 450•C for one hour. For complete de-carbonization to obtain white ash. The crucibles and ash were cooled and the weight was recorded. The ash content is mathematically expressed as;

$$\text{Ash content m} = \frac{Df}{Dt} x100\% \quad \ldots\ldots\ldots(3)$$

where D_t is the oven dry weight of carbon sample and D_f is the final ash weight of carbon sample

- Carbon Yield

Weight of both pyrolyzed and unpyrolyzed gmelina carbon was recorded. The total yields were determined after sample processing in terms of raw gmelina.

$$Y_{CH} = \frac{W_{CH}}{W_o} x100\% \quad \ldots\ldots\ldots\ldots\ldots 4)$$

where W_o is the mass of the gmelina activated carbon retrieved from the furnace and

W_{CH} is the Mass of air- dried Gmelina bases activated carbon

- Determination of Bulk Density

This was determined using an empty measuring cylinder, it was weighed and the weight noted. The cylinder was then filled with the Activated Carbon and the top layer of the graduated cylinder was tapped, and then tapped for ten times. The volume occupied by the packed sample was recorded as V if W_C was the weight of the empty cylinder and W the weight cylinders d sample, then weight of the sample was obtained by $W_S = W - W_C$

The bulk density (Bd) was calculated using equation below

$$Bd(g/ml) = \frac{Ws}{Vs} \quad \ldots\ldots\ldots\ldots(5)$$

- Fixed Carbon

Results obtained from yield of activated carbon, ash content and moisture content computed together to account for fixed carbon content.

$$\text{Fixed Carbon Content} = \frac{Y_{CH} - A_C - M_C}{Y_{CH}} \text{x}100\% \quad \text{.....(6)}$$

where Y_{CH} is the yield of carbon, A_c was the ash content and M_c is moisture content

- Iodine Number

Determining the iodine number is one of the methods to determine the adsorption capacity of activated carbons. It is a measure of the micropore (0–20 Å) content of the activated carbon by adsorption of iodine from solution. The typical range is 500–1200 mg/g, which is equivalent to surface area of carbon between 900 and 1100 m^2/g (Saka, 2012). Iodine method was used to determine the surface area of the activated carbon. 0.3g of sample A was weighed and centrifuged in 0.1M iodine solution. 0.1M sodium thiosulphate solution was titrated against 10ml of sample A free aliquot (solution obtained after centrifuge) using 2 drops of freshly prepared starch solution as indicator. The volume of thiosulphate required to titrate 30ml of blank solution (purely iodine solution was also determined. All the titrations were done in duplicate and average value recorded. The surface area and the iodine number were calculated as standard (Saka, 2012).

3 Results and Discussion

Table 1 shows the yield of produce Activated Carbon (AC). The yield of activated carbon was calculated from the formula in equation (7). The most important parameter affecting activated carbon yield are the carbonation temperature and time.

Table 1 Weight of Materials and Operating Conditions

Weight of the wet wood (GW)	1550 g
Weight of the dry wood	1422.9
Weight of the charcoal	170g
Weight of the activated Carbon	580g
Carbonization time	2hours
Carbonization temperature	480 ^oC

Using the Table, the yield of activated carbon can be calculated as;

$$Yield = \frac{weight\ of\ activated\ carbon\ produced\ (g)}{weight\ of\ dry\ gmelina\ wood\ (g)}\ X\ 100 \quad \text{.....(7)}$$

The calculation shows that 1422.9g of gmelina wood produced 580g of activated carbon, amounting to about 40.76% yield.

3.1 Comparisons of Properties of Produced Activated with Commercially Produce Activated Carbon

Table 2 shows the comparison between the produced activated carbon with commercial activated carbon. Moisture content was calculated and the value was 8.2% which was close to standard thereby indicating that the adsorption capacity of the activated carbon was high. Low moisture content will cause an increase in the performance of the activated carbon. High temperature pyrolysis will cause the C and H bonds in the charcoal to be released respectively. Thus, decreasing the crystallinity of the activated carbon. Also, the ash content was found to be 11% which was equal to standard. The bulk density falls within the standard and a moderate bulk density will promote adsorption. Ash content has an influential role in affecting the quality of the activated carbon. The presence of excessive ash content can lead to clogging of the pores thereby reducing the surface area of the activated carbon. Decrease in ash content indicates increase in the surface area.

Fixed carbon was low (53%) but however, fall in the range of standard. Iodine number is a measure of the activity level and the porosity of the activated carbon. Free carbon content is affected by cellulose and lignin content that can be

converted to carbon atoms. Low fixed carbon is caused by the breakdown of the activated carbon structure at high temperature.

Higher iodine number indicates higher degree of activation. Therefore, the higher the iodine number, the higher the degree of adsorption (Industrial Standard, 1995).

The findings show that moisture content, ash content and free carbon all met the standard values.

Table 2 Components of Produced Activated Carbon with Industrial Standard

Properties of Activated carbon	Produced Activated Carbon	Natural Industrial Standard
Moisture content	8.2%	<15%
Ash Content	11%	<10
Bulk Density	0.5g/cm^3	<5<7g/cm^3
Iodine Number	1050mg/g	>750mg/g
Fixed Carbon	53%	>50%

3.2 X-ray Diffraction Pattern of Gmelina Wood Activated Carbon

Appearance of broad diffraction background and the absence of sharp peak reveal a predominantly amorphous (Little or no long term orderly arrangement of the atoms or molecules) structure (Omri & Benzina, 2012). There two broad diffraction background corresponding to 2tither = 26° and 44°in the spectrum corresponding to diffraction of (111°) and (100°) respectively as seen in the graph of Figure 3.

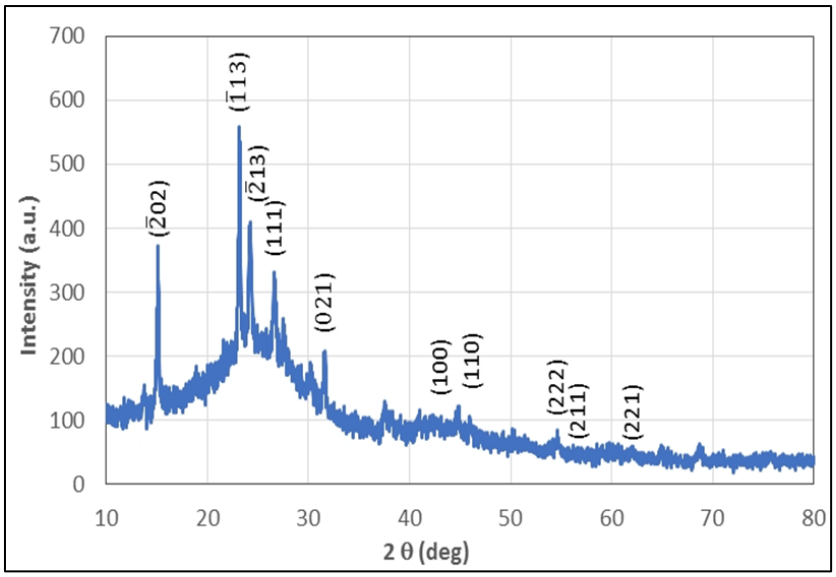

Figure 3 X-Ray Diffraction Pattern of Gmelina wood Activated Carbon

The crystallinity recorded by gmelina wood activated carbon (GWAC) was 9% crystalline and 91% amorphous. This value was close to previous study for bamboo and coconut shell activated carbon (Omri & Benzina, 2012). Predominantly amorphous solid have large internal surface area and pore volume. It was found that produced activated carbon had very little crystalline structure (little orderly arrangement of molecules and atoms) and large amorphous structure in graphite (carbon). Therefore, activated carbon with 91% amorphous structure would have large internal surface area and pore volume (Omri & Benzina, 2012).

3.3 Brunauer-Emmet-Teller (BET) Isotherm of the Produced Gmelina Wood Activated Carbon

Brunauer-Emmet-Teller of the Gmelina wood activated carbon was detected by Nitrogen adsorption at 77.3k or adsorption isotherm. The adoption isotherm was found to be 1000 m^2/g. Increase in adsorption isotherm indicate large

surface area which increases the adsorptive property of the produced activated carbon. This value was close to previous done work done by (Saka, 2012)

4 Conclusion

This study obtained several tests on the produced activated carbon from gmelina wood and the result was compared with Industrial Standard (1995). To do this, the carbonization process of the gmelina wood was carried out using pyrolysis reactor for 2hours at temperature of 480 °C in order to produce charcoal. The char was then impregnated with nitric acid. Brunauer-Emmet-Teller (BET) surface area of the produced activated carbon was detected by Nitrogen adsorption at 77.3 K. Total yield of the produced activated carbon were determined in terms of raw Gmelina wood. X-Ray diffraction (XRD) analysis was performed on the Gmelina wood activated carbon in order to determine the crystallinity or amorphous nature. Proximate analysis was carried out to determine the moisture content, free carbon, bulk density and ash content. The following values were obtained during characterization of the produced activated carbon: moisture content was 8.2%, Ash content of 11%, free carbon of 53%, bulk density of 0.5 g/cm^3 iodine number of 1050 mg/g and BET of 1000 m^2/g. The X-Ray Diffraction (XRD) result confirmed that the produced activated carbon has less crystallinity (9%) and high amorphous (91%). It was then concluded that the activated carbon produced from Gmelina wood met all the requirements of the Industrial Standard.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed

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