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## Production and Characterization of Active Carbon from Epicarp of Balanite Aegyptiaca and Detarium Mirocarpum Shells

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### **Abstract:**

Activated carbon was produced from Epicarp of Balanite aegyptiaca and Detarium mirocarpum shells with  $ZnCl_2$  and  $H_3PO_4$  as activating agents. The amount of Lignin and cellulose in the raw Balanite aegyptiaca shell were determined and found to be 30.12% and 10.40% respectively, while its content in Detarium mirocarpum seed-shell was 20.40% and 26.42% respectively. The % of carbon yield generally decreases as temperature of activation increases. The % yield of the carbon followed this order; ACBAZC > ACBAPA > ACDMZC > ACDMPA, while the SEM Micrographs shows the structural morphology of the carbon pores. The BET Surface area of ACDMPA, ACDMZC, CAC, ACBAPA and ACBAZC determined were  $857.99m^2/g$ ,  $799.43m^2/g$ ,  $785.35m^2/g$ ,  $774.76m^2/g$  and  $555.73m^2/g$  respectively, which are within the standard range of notable commercial activated carbon. The FTIR absorption peaks were observed in both precursors and the activated carbon produced. On the process of activation, new peaks were identified on the carbon, which were not in the precursors. There was no any functional group identified on the surface of commercial activated carbon. The characteristics property of the carbon shows that the adsorbent possessed good qualities when compared with standard commercial activated carbon. The carbon produced is highly recommended for water and wastewater treatment.

**Keywords:** Balanite egyptiaca, Detarium mirocarpum, surface area, SEM, and FTIR.

### **1. Introduction**

In developing Countries like Nigeria, activated carbon are obtained by purchasing in large quantity at a very expensive cost, whereas vast quantity of lignocellulosic biomass, which can be used for its production to meet our demands and for exportation, can be produced. The aim of this study is to create wealth from waste, by converting lignocellulosic biomass, which are regarded as by-products into suitable adsorbents, for water and wastewater treatment (Okibe et al., 2013).

One area of research interest is the applications of activated carbon in our environment for wastewater treatment such as wastewater purification, decolourization and the removal of toxic organics and heavy metal ions (Jabit, 2007, Lotfy, 2006; Dastgheib and Rockstraw, 2001; Wartelle and Marshall, 2001).

The requirement of activated carbon increased recently, and the market growth was estimated at 4.6 % per year (Alau, 2015; Jabit, 2007). This demand can be made available, by converting the large quantity of Lignocellulosic Biomass waste present in our environment for the production of activated carbon (Jabit, 2007). One of its most important fields is to remove hazardous organic compounds or those that impart odour or taste (Olafadehan and Susu, 2005; Olafadehan and Aribike, 2000; Tharapong et al., 1999), clean-up of off-gases containing volatile organic compounds, food processing, pharmaceutical and environmental remediation, amongst other uses. Activated carbon is a microcrystalline, non-graphite form of carbon that has been processed to develop internal porosity (Olafadehan and Jinadu, 2012), characterized by a very large specific surface area.

This study, therefore, focused on investigating the optimal conditions required for the production and characterization of active carbon from the Epicarp of Detarium *mirocarpum* and Balanite *aegyptiaca* shells.

## 2. Materials and Methods

### 2.1. Collection of Precursors and Pre-Treatment

Samples (*Balanite aegyptiaca* fruits and *Detarium macrocarpum* fruits) were purchased from Dutsin-ma Market in Dutsin-ma Local Government area of Katsina State. The edible parts of *Balanite aegyptiaca* and *Detarium macrocarpum* fruits were removed leaving behind the epicarp shells, which were washed thoroughly with distilled water to remove impurities. The samples were sundried for a week, washed again with distilled deionized water and then dried in a thermostatic Oven at 105<sup>0</sup>C for 48 hours which facilitated easy crushing and grinding. The dried samples were pulverised using mortar and pestle and sieved with a mechanical shaker into particle size of 850µm. The fine sieved particles were stored in a clean air tight plastic containers ready for further treatment.

### 2.2. Characterization of the Precursors

The composition of the raw material is an important criterion that dictates the selection of Precursors for activated carbon production. The chemical composition of the precursor material, mainly the percentages of cellulose and lignin present in the epicarp shell of *Balanite aegyptiaca* and *Detarium macrocarpum* were found by means of standard methods (Thimmaiah, 1999).

#### 2.2.1. Estimation of Cellulose and lignin

Standard methods were followed for the determination of the level of cellulose and lignin in the raw precursors, as exactly when Bael fruit shell was used for the production of activated carbon (Ramakrishna and Mishra, 2012).

### 2.3. Production of Activated Carbon

The activating agents used were H<sub>3</sub>PO<sub>4</sub> and ZnCl<sub>2</sub>. Standard methods were followed for the production of carbon as described by Alau et al., (2015).

### 2.4. Characterization of the Activated Carbon

The various properties of prepared activated carbons were characterized using standard procedures. The characteristics property determined include yield, SEM, BET Surface area and FTIR.

## 3. Results and Discussion

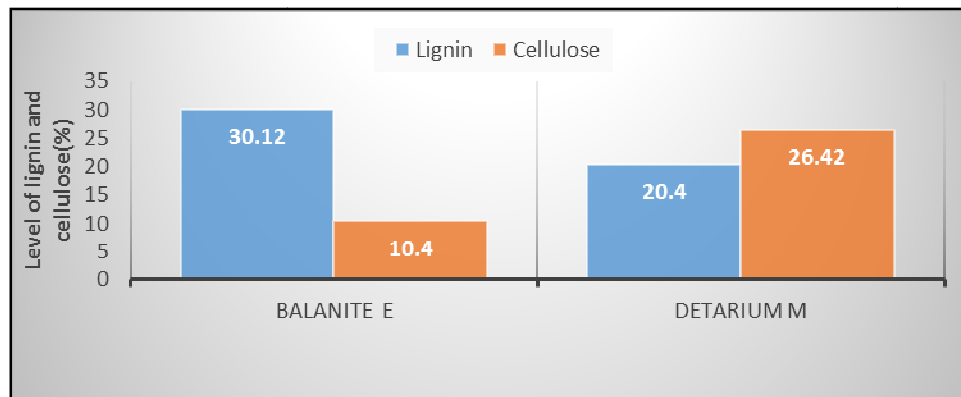


Figure 1: The level of Lignin and Cellulose in *Detarium microcarpum* and *Balanite egyptiaca* shells.

From Figure 1 above, the amount of Lignin and cellulose in *Balanite aegyptiaca* shell were found to be 30.12% and 10.40% respectively, while its content in *Detarium microcarpum* seed-shell was 20.40% and 26.42% respectively. Materials with high lignin content develops AC with high yield, whereas, materials with high cellulose yields AC with high BET surface area. The results obtained in this study is similar to research carried out when Bael fruit shell was used (Ramakrishna and Mishra, 2012).

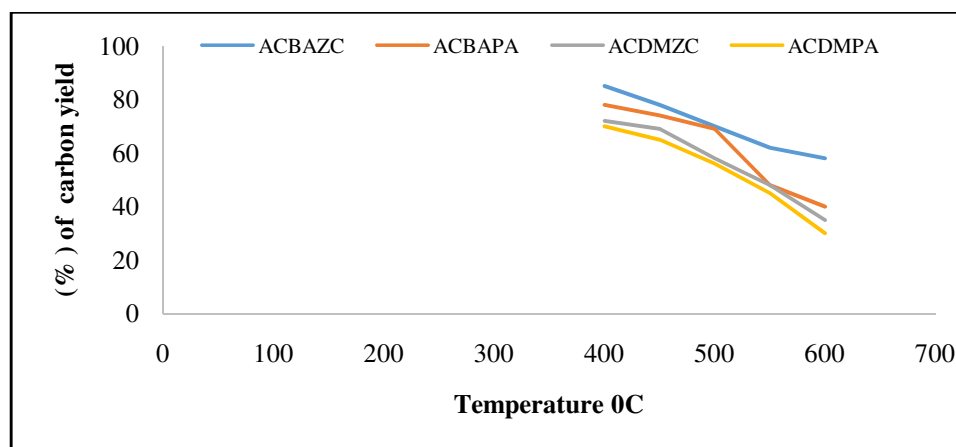


Figure 2: Effect of activation temperature on the yield of prepared activated carbon at optimized conditions.

➤ KEYS

- AC = Activated carbon
- ACDMPA = Activated carbon from *Detarium microcarpum* activated with H<sub>3</sub>PO<sub>4</sub>
- ACDMZC = Activated carbon from *Detarium microcarpum* activated with ZnCl<sub>2</sub>
- ACBAPA = Activated carbon from *Balanite aegyptiaca* activated with H<sub>3</sub>PO<sub>4</sub>
- ACBAZC = Activated carbon from *Balanite aegyptiaca* activated with H<sub>3</sub>PO<sub>4</sub>
- CAC = Commercial activated carbon.

Figure 2 illustrates the yield of the activated carbon produced with respect to temperature. The yield of carbon obtained from *Balanite aegyptiaca* was higher than that from *Detarium microcarpum*, because *Balanite aegyptiaca* has high level of lignin in the precursor when compared with *Detarium microcarpum*. Generally, the yield of the carbons decreases as temperature increases due to the loss of some organic compounds in the precursors during activation, which is in agreement with the research carried out when Neem cake and Neem husk was used (Alau et al., 2015).

### 3.1. SEM Micrographs of the Carbon

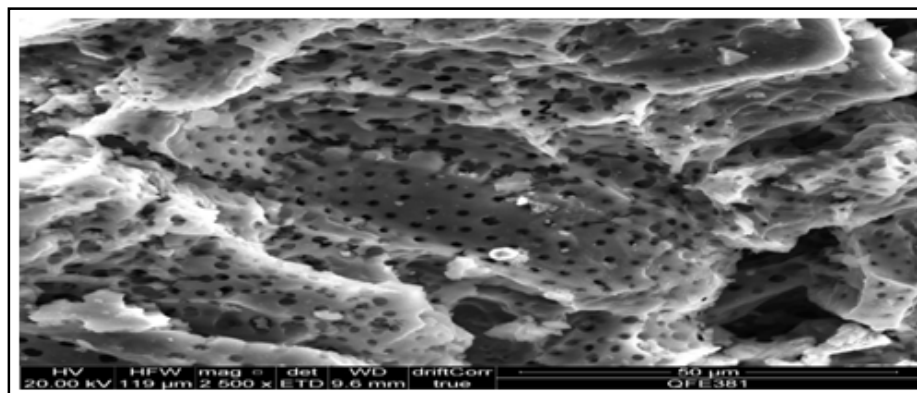


Figure 3: SEM of ACDMPA

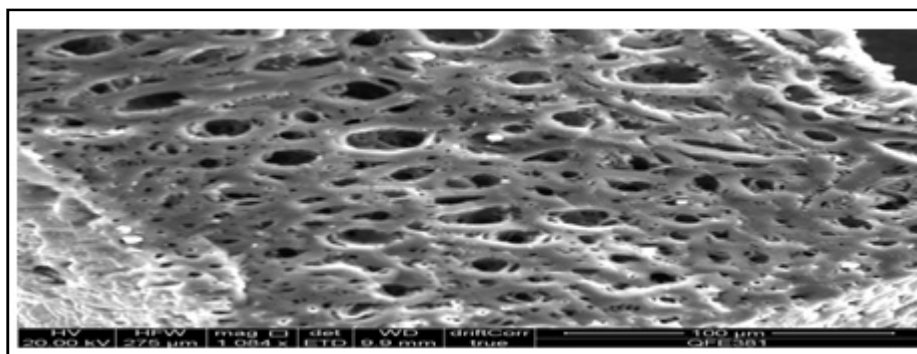


Figure 4: SEM of ACDMZC

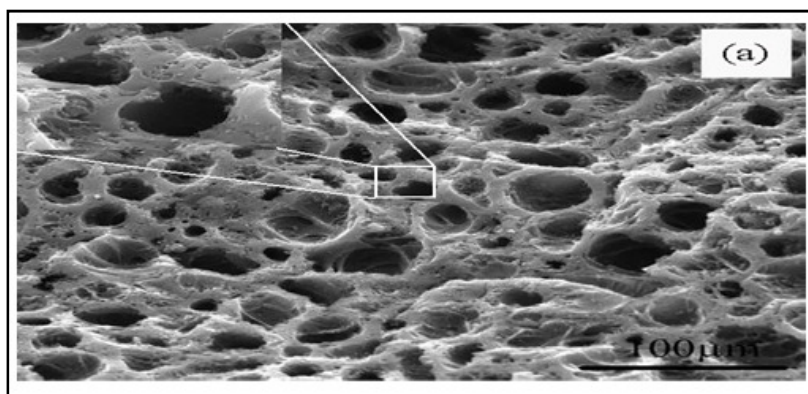


Figure 5: SEM OF ACBAPA

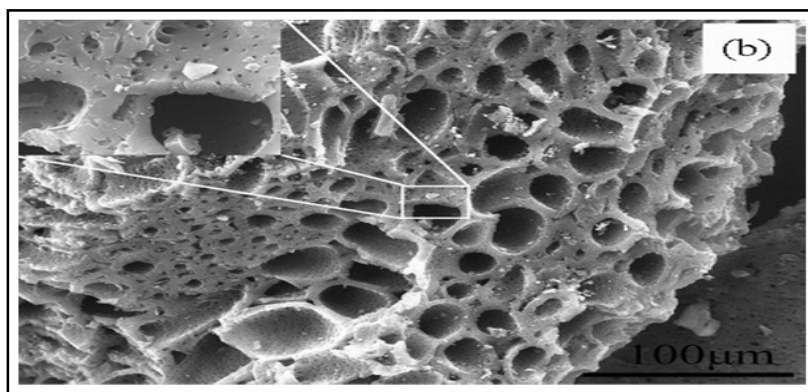


Figure 6: SEM OF ACBAZC

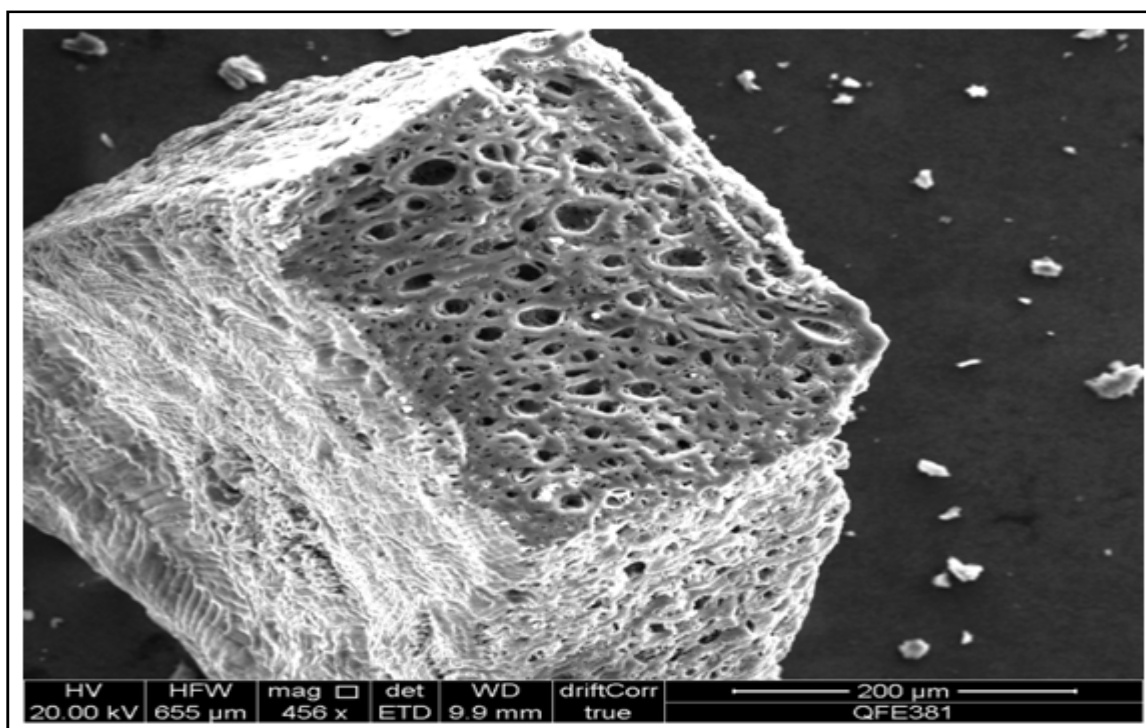


Figure 7: SEM of CAC

The micrographs of the carbon produced were presented in Figures 3 - 7. The micrographs revealed that the pore structures of the carbon were affected by the activation process. The pore structures of the activated carbon are a combination of micropores, mesopores and macropores which is an indication of more surface area available for adsorption and better performance during application.

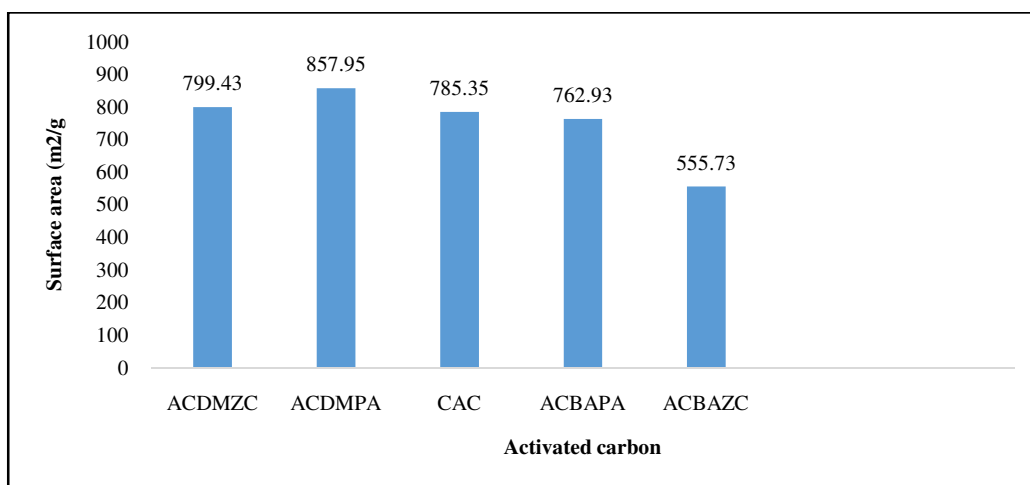


Figure 8: BET surface area of the produced carbons and commercial activated carbon.

Figure 8 above reveals the level of BET surface area of the carbon. It shows that ACDMPA and ACDMZC were the only produced carbon with BET surface area of 857.99m<sup>2</sup>/g and 799.43m<sup>2</sup>/g respectively, greater than that of commercial activated carbon, with 785.35m<sup>2</sup>/g, which may be attributed to the high level of cellulose in the precursor, while ACBAPA and ACBAZC possessed surface area of 774.76m<sup>2</sup>/g and 555.73m<sup>2</sup>/g respectively which is less than that of commercial activated carbon due to high level of lignin in the precursor. The BET Surface area of ACDMPA, ACDMZC, CAC, ACBAPA and ACBAZC were 857.99m<sup>2</sup>/g, 799.43m<sup>2</sup>/g, 785.35m<sup>2</sup>/g, 774.76m<sup>2</sup>/g and 555.73m<sup>2</sup>/g respectively. Most widely used commercial activated carbons have specific surface areas ranging from 750- 1500 m<sup>2</sup>/g (Okibe et al., 2013), hence the chemically produced activated carbons for this research possessed surface area that falls within the standard range of commercial carbon. However, the surface area of the carbon from *Detarium microcarpum* is greater than the ones produced from *Balanite aegyptiaca*. The nature of the precursors may be the reason for the differences.

### 3.2. FTIR of the Raw Precursors and Activated Carbon Produced

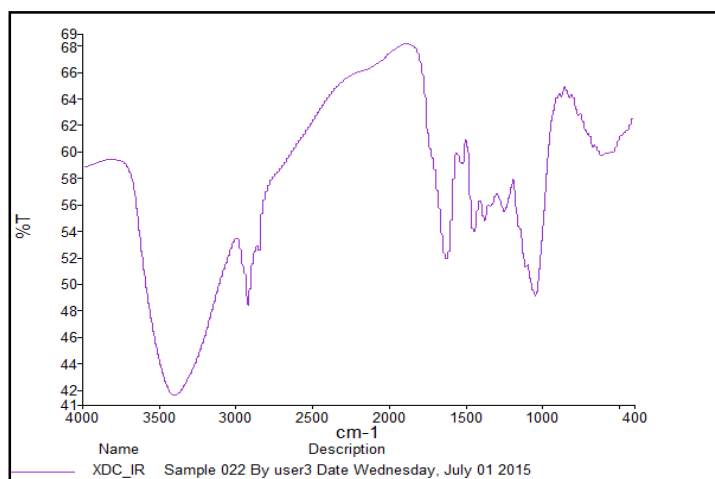


Figure 9: FTIR of raw *Detarium microcarpum* precursor

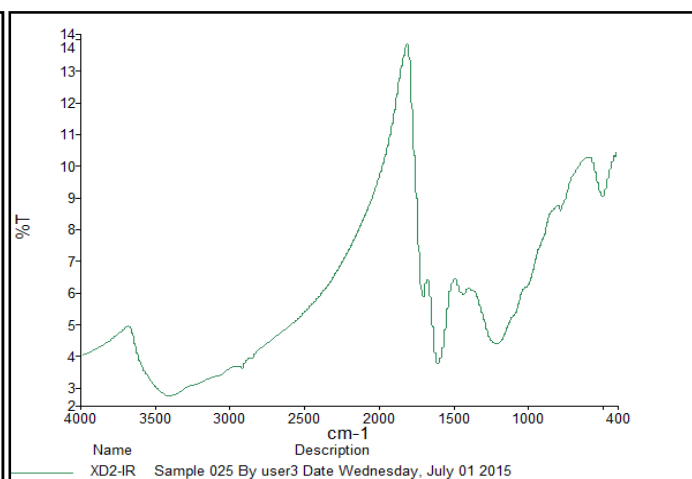


Figure 10: FTIR of ACDMPA

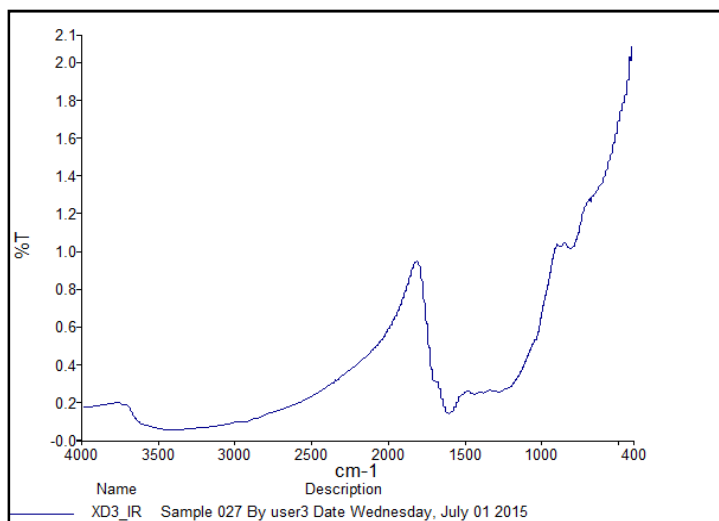


Figure 11: FTIR of ACDMZC

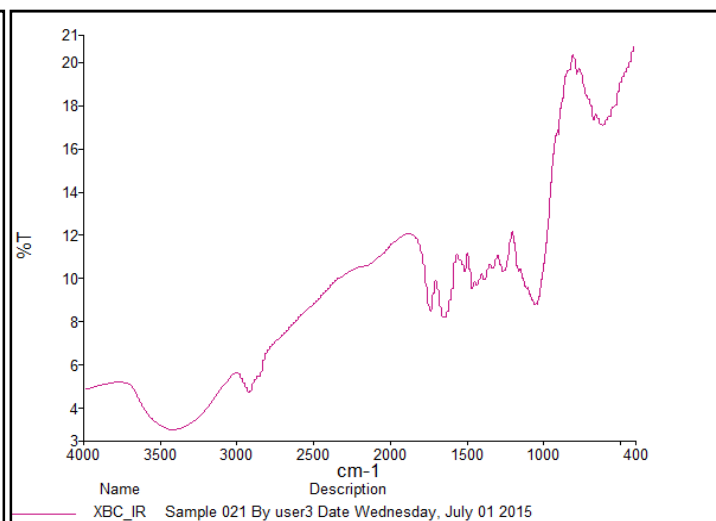


Figure 12: FTIR of raw Balanite egyptiaca precursor

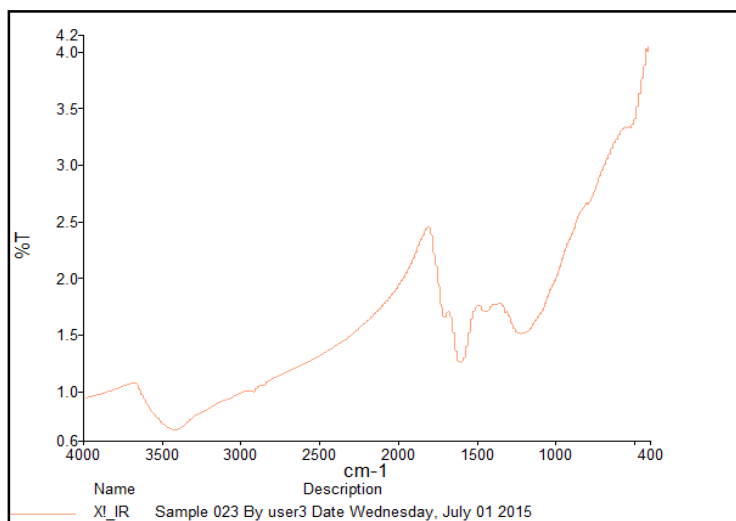


Figure 13: FTIR of ACBAPA

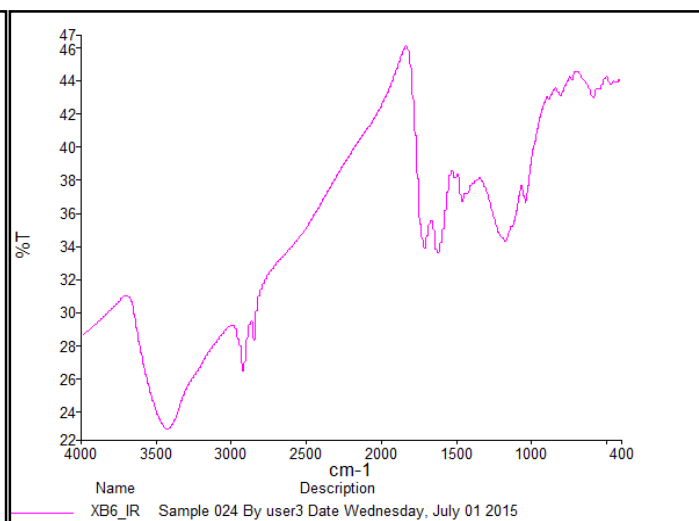


Figure 14: FTIR of ACBAZC

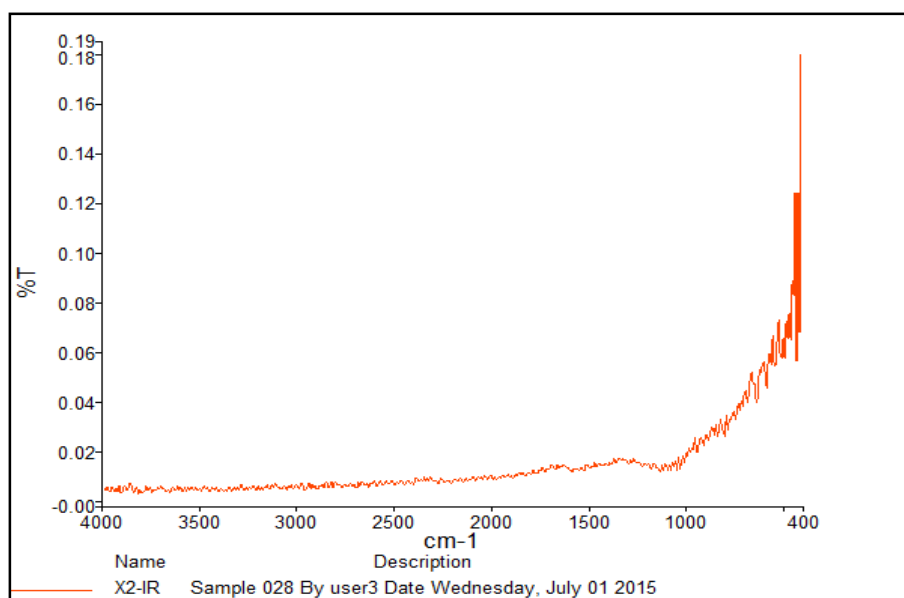


Figure 15: FTIR of CAC



The FTIR spectra of raw *Detarium microcarpum*, ACDMPA and ACDMZC were presented in Figures 9 - 11 respectively, identified absorption peaks with respect to some functional groups present. The raw *Detarium microcarpum* showed absorption within 3300-3400 $\text{cm}^{-1}$ , 2500-3000 $\text{cm}^{-1}$ , 1000-1250 $\text{cm}^{-1}$  and 1550-1650 $\text{cm}^{-1}$  due to N-H stretch vibration from 2<sup>o</sup>amines, strong O-H stretch vibration due to carboxylic acid, C-N stretch due to amine and medium NH<sub>2</sub> bending (scissoring) respectively. On the other hand, FTIR for ACDMPA shows absorption bands within 2500-2700 $\text{cm}^{-1}$ , 600-700 $\text{cm}^{-1}$  and 1000-1250 $\text{cm}^{-1}$  due to (O =) PO-H stretching vibration due to phosphonic acid as a result of using ortho-phosphoric acid as activating agent, C-H bending vibration due to alkyne and C-N stretching due to amine, which may be attributed to decomposition in the Furnace respectively. The only FTIR spectra for ACDMZC was observed at frequencies range of 1705-1720 $\text{cm}^{-1}$ , due to C-N stretching vibration from a carboxylic acid derivative.

The spectra for raw *Balanite aegyptiaca*, ACBAPA and ACBAZC presented in Figures 4.28-4.30 respectively. The raw precursor (*Balanite aegyptiaca*) shows absorption peaks within frequencies range of 3400-3500 $\text{cm}^{-1}$ , 2500-3300 $\text{cm}^{-1}$ , 1710-1740 $\text{cm}^{-1}$  and 600-700 $\text{cm}^{-1}$  due to N-H stretching from 1<sup>o</sup> amine, O-H stretch vibration due to carboxylic acid, strong C=O stretch due to saturated aldehyde and ketone, and C-H bending due to alkynes respectively.

On the other hand, ACBAPA shows new absorption peak at 2500-2700 $\text{cm}^{-1}$  due to (O=) PO-H stretch due to phosphonic acid as a result of using ortho-phosphoric acid as activating agent. The spectra of ACBAZC shows absorption peaks within frequencies range of 3300-3400 $\text{cm}^{-1}$  and 1785-1815 $\text{cm}^{-1}$  due to N-H stretch from 2<sup>o</sup> amine and C=O stretch due to acyl halide, which may be link to the use of Zinc chloride as activating agent. There was no functional group or absorption peaks observed with respect to commercial activated carbon (CAC).

#### 4. Conclusion

Activated carbon produced from epicarp of *Balanite aegyptiaca* and *Detarium microcarpum* shells with H<sub>3</sub>PO<sub>4</sub> and ZnCl<sub>2</sub> as activating, exhibited characteristics property of good adsorbents, which can be utilize for water and wastewater treatment. From the results of BET surface area of the carbon, it possessed quality surface area that falls within the standard limit of required commercial activated carbon. The SEM micrographs shows the morphological pores structure of the carbon that looks unique in quality. Consequently, the adsorbent produced from these wastes biomass can be used as adsorbents for various environmental applications including the removal of organic pollutants, colour and heavy metals from industrial effluents, and treatment of town water supply.

#### 5. References

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