

Production and Characterization of Chemically Activated Carbon from *Khaya senegalensis* Shell Waste

Mikyitsabu Ago Atoshi and Ataitiya Hyelalibiya

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Abstract: Despite the numerous and important applications of activated carbon, not much work has been done on its application as a supporting activated carbon for the production of biodiesel. In this study, an activated carbon was developed through chemical activation of *Khaya senegalensis* shell waste. The effect of pH, conductivity, ash content, carbon yield and moisture content were investigated. From the results, the yield of the activated carbon decreases gradually as the temperature increases from 500°C to 900°C. The activated carbon yield decreases progressively as the impregnation ratio of K_2SO_3 increases from 2 to 10 gdm^{-3} . The optimum temperature for carbonization was observed at 800 °C. The effect of temperature on the ash content of the activated carbon at 500 to 700 °C were observed to be 6.2 to 11.2. However, when the temperature was raised from 700 to 900 °C, the ash contents also increased from 11.2 to 15.3. Moisture content also shows a decrease with increasing reaction temperature. At 500 °C the moisture content was observed to be 7.9 % and the lowest moisture content was observed to be 3.0% at 900 °C. The external surface of the

activated carbon that was produced at 3:6 carbon impregnated with K_2CO_3 displays some crystals on the surface, which also displayed some pores and cavities on the surface. The FT-IR spectrum showed band that was attributed to C-C stretching. Other peaks were observed at corresponds C=O, 1442.5 is associated to C-O stretching and a C-H bending at $700.7cm^{-1}$.

Keywords: Activated, carbon, Heterogenous, Shell, Waste

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1.0 Introduction

The shell of *Khaya Senegalensis* fruit popularly known as the Mahogany fruit shell is a spherical woody capsule (4-6 cm in diameter), this has always been thrown away as waste (Dass *et al.*, 2018). Usually, each capsule contains at least six seeds. *Khaya senegalensis* seeds have about 53 % by weight oil (Eromosele *et al.*, 1998). However, it is used in traditional medicine of several African communities (Bamaiya *et al.*, 2006). The high preparation cost of alkaline heterogeneous

activated carbons poses a challenge that hinders their usage in the biodiesel industry (Gohla *et al.*, 2021). Ogungbenro *et al.* (2020), synthesized from seeds of local date fruits in the United Arab Emirates (UAE) and investigated for carbon dioxide (CO₂) adsorption. Activated carbon (AC) synthesis was completed by furnace activation in the temperature range (600, 700, 800, 900 °C) after infusion with chemical compounds. Two reagents (Potassium Hydroxide- KOH and

Sulfuric acid- H_2SO_4) were mixed with the date seed samples in varying impregnation ratios (KOH- 3:1, 4:1, 5:1 and H_2SO_4 - 0.5:1, 1:1, 2:1) and directly heated to activation wherein the removal of volatile matter content leads to formation of activated carbon porous structure. The activated carbon yield was found to be inversely proportional to the activating temperature and impregnating ratio (Sadiq & Hussian 2013).

Shima & Ajay (2009) optimized and characterized highly porous activated carbon from algal-derived hydrochar. The chemically prepared activated carbons at optimum process conditions of $T = 675\text{ }^\circ\text{C}$, $R = 1.5$ and $F = 267\text{ cm}^3/\text{min}$, using potassium carbonate or potassium hydroxide as a chemical agent, revealed high surface area ($\geq 2100\text{ m}^2/\text{g}$) with the maximum yield of 61.3 wt%, pore volume in the range of $(1.2\text{--}1.5\text{ cm}^3/\text{g})$ and average pore size of $(5.9\text{--}8.3\text{ nm})$. Almost complete removal of methylene blue removal was achieved from a solution with methylene blue concentration of 250 mg/L, with chemically activated carbon dosage of 1 g/L within 5 min at room temperature.

The present study is aimed at producing an activated carbon as a heterogeneous activated carbon from indigenous agricultural wastes (Mahogany – *Khaya senegalensis*) shells impregnated with K_2CO_3 .

2.0 Material and Methods

2.1 Sample collection and reagents

The *Khaya senegalensis* fruit shells were collected from the premises of the Federal University Wukari, Wukari LGA, Taraba State, Nigeria. The chemicals used for the study were analytical grade and included K_2CO_3 , K_2CO_3 , HCl, and CH_3OH .

2.2 Pre-treatment of raw materials

The shells of *Khaya senegalensis*, was washed, sun-dried to a constant weight. The crushed samples were thoroughly washed with distilled water till the wash water became colorless which was then dried at $110\text{ }^\circ\text{C}$ in an oven for 8 hours to get rid of moisture and other

volatiles. The pretreated materials were kept in a sample container for the carbonization and activation process (Girgis & Ishak 2019).

2.4 Carbonization of *Khaya senegalensis* shell.

Five hundred grams of the pre-treated shell was placed in a crucible and carbonized at a temperature of $800\text{ }^\circ\text{C}$ for 2 hours. At the end of the 2 hours, the sample was allowed to cool to room temperature and preserved in a desiccator, ensuring that little or no oxygen was present during the carbonization and activation process. It was removed, grounded and sieved with a $300\text{ }\mu\text{m}$ before storage and labelling. sieve

2.5 Chemical activation and carbonization

Chemical activation was carried out according to the methods described by Ajayi and Olawale, (2009). The aqueous solution of potassium carbonate (K_2CO_3) was prepared with initial concentration of $100\text{ g}/\text{dm}^3$, 100 g of the produced *Khaya senegalensis* carbon were weighed into separate beakers, and 300 cm^3 of the prepared solution of potassium carbonate was added and the solution was agitated for 1 hour and kept for 24 hours to allow the sample to soak which was placed in an oven at $110\text{ }^\circ\text{C}$ to form a paste.

The carbonization of sample was carried out by charging 30 g of the paste in a crucible into a muffle furnace (Daud & Ali 2004). The carbonization temperature was varied from 500, 600, 700, 800 and $900\text{ }^\circ\text{C}$ with an activation duration of 1h. After carbonization, the sample was allowed to attain the room temperature and neutralized with HCl before re-wash with distilled water to a constant solution pH. The paste formed after washing was dried at $100\text{ }^\circ\text{C}$ to obtain the activated $06\text{ }\mu\text{m}$, put in an air tight bottle labeled K_2CO_3 activated carbon for *Khaya senegalensis* carbon and collectively called chemically prepared activated carbon (CPAC).

2.6 Determination of pH and conductivity



The pH and conductivity were determined according to ASTM D3838-80. 1.0 g of the activated carbon was weighed and transferred into a 250 ml beaker and 100 ml of distilled water was added and stirred for 1 hour. The sample was allowed to stabilize and then the pH was measured using a handheld pH/Conductivity meter, (Jenway 430 Model). The same sample was used for the electrical conductivity (EC) of the AC (Mikyitsabu & Johnson 2023).

2.7 Ash content determination

Ash content determination was done according to the ASTM D2866-94 method. 2g of AC sample was placed in a porcelain crucible which was weighed and transferred into a preheated muffle furnace set at a temperature of 1000°C. The furnace was left on for one hour after which the crucible and its content were transferred to desiccators and allowed to cool. The crucible and content were reweighed and the weight lost was recorded as the ash content of the AC sample (W_{ash}). Then the % ash content (dry basis) was calculated from the equation (Donni *et al.*, 2007).

$$Ash = \frac{W_{ash} \times 100}{W^{\circ}}$$

W° = initial weight of AC, W_{ash} = weight loss

2.8 Carbon yield (ASTM D5373-99)

The total yield (%) was determined after sample processing in terms of raw material mass. The dried weight, W° of pre-treated sample was determined using Metler balance and the carbon yield calculated as

$$Y(\%) = \frac{W \times 100}{W^{\circ}}$$

where Y_{ch} = Carbon yield (%); W = final weight of AC prepared; W° = initial weight of the sample used in the carbonization and activation processes (Donni *et al.*, 2007).

2.9 Moisture content determination

AC sample (1g) was weighed and dried in an oven set at 110°C. The drying sample was constantly reweighed at 10-minute intervals until a constant weight (W_p) was obtained. The

crucible and its content were retrieved and cooled in a desiccator. The difference in weight was recorded and the moisture content (MC) was calculated from the equation as loss in weight on drying divided by initial weight of activated carbon multiplied by 100 (ASTM D2867-99).

$$MC = \frac{W_f - W_i \times 100}{W_o}$$

where W_f = weight of Carbon retrieved from the oven, W_i = weight of crucible and AC and W_o = initial dry weight of the AC sample (Verla *et al.*, 2012)

2.10 Characterization of activated carbon.

2.10.1 The morphology of the activated carbon

Oven dried porous samples will be mounted on an adhesive carbon tape attached to an aluminum-stub and subsequently sputter coated with platinum for 5 minutes in a JFC-1100 sputter coater. SEM magnification will be selected at 1000, 5000, and 10000 (Guo & Lua 2003). The properties of the activated carbon from the scan result were used to determine the surface area and porosity of each of the activated carbon prepared.

2.10.2 FT-IR analysis

The FTIR of the sample was obtained after scanning through a wave number range of 400 to 4000 cm^{-1} using a PERKIN-ELMER FTIR machine.

3.0 Results and Discussions

3.1 Effect of temperature (°C) On the Yield (%) of Activated carbon

Fig. 1: shows the effect of temperature on the yield (%) of the carbonized *Khaya senegalensis* shell when the temperature was varied from 500 to 900 °C (at constant concentration of K_2CO_3 and time). The result shows that the yield of the activated carbon decreases with an increase in temperature. Increase in temperature degressively affect the yield of the activated carbon. The optimum yield was



82.96% at 500°C. this was in agreement to Ogungbenro *et al.*, (2020) where the activated carbon yield was found to be inversely

proportional to the activating temperature and impregnation.

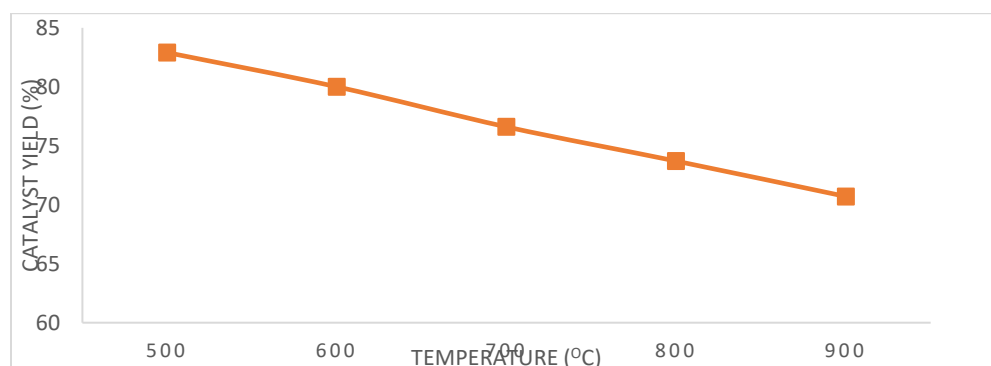


Fig.1: Plot showing the effect of temperature (°C) on the yield of the activated carbon yield at constant impregnation ratio (gdm³)

3.2 Effect of impregnation ratio K₂CO₃ (gdm⁻³) on the yield (%) of the Activated carbon

Fig. 2: The impregnation ratio was varied from 3:2 to 3:10 and the observed results showed that the yield of the activated carbon decreases with an increase in the impregnation ratio. There was a minimal decrease in the yield of the activated carbon with the highest yield

observed at 3:2 with 86.63%. Luo *et al.*, (2019) indicate the relationship between product yield and impregnation ratio that activated carbon yield decreases with increasing the number of activating agents at a fixed activation temperature. The effect of impregnation ratio may be attributed to increasing in effectiveness of activation process by the chemical agents due to burn-off (Sugumaran *et al.*, 2012).

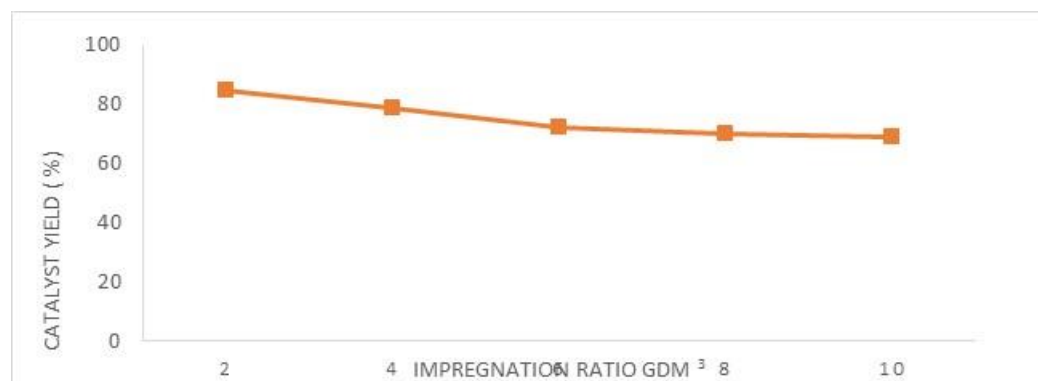


Fig 2: A plot showing the effect of impregnation ratio (gdm³) on activated carbon yield at constant temperature (°C) and time (h).

3.3 Dependency of the pH of the activated carbon on temperature

Fig. 3: shows that the pH is affected by the constant impregnation of k₂CO₃ and time. As the temperature of the reaction increases the pH

from 9.9 to 11.0. A sharp rise in the pH was observed from temperature of 500 °C to 820 °C This shows that temperature effect the alkalinity of the activated carbon. The optimum temperature for carbonization was observed at pH of 10.6 for the activated carbon.



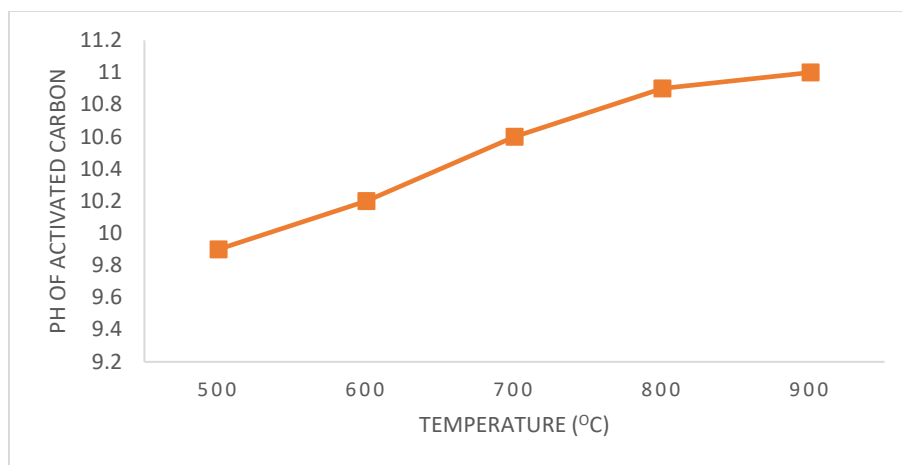


Fig 3: Variation of the pH of the activated carbon with temperature

3.4 Dependent of pH on calcination temperature

Fig. 4 shows the dependence of pH on calcination temperature. The result shows that the pH of activated carbon increases with an increase in the concentration of K_2CO_3 . The impregnation ratio shows a progressive increase between 4 to 6 and 8 to 10 gdm^{-3} .

Jun'ichi *et al.* (2002) Prepared activated carbon from chickpea husk by K_2CO_3 activation and obtained highest surface area as $1778 m^2/g$ with an impregnation ratio of 1.0 (100 wt%) and activation temperature of $800^\circ C$ which was kept constant. Jun'ichi *et al.* (2002) observed that BET surface areas of both KOH and K_2CO_3 activated carbon increased to some extent by increasing impregnation ratio.

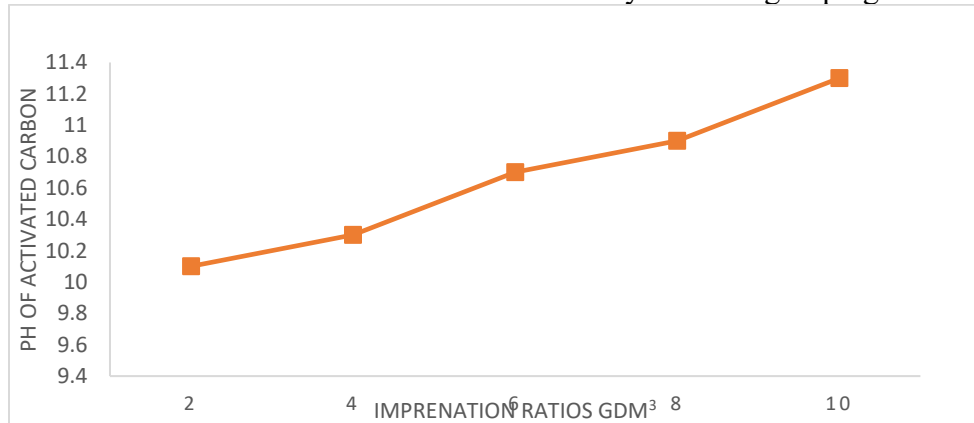


Fig. 4: pH of activated carbon at different impregnation ratio (gdm^3), constant temperature ($^\circ C$) and time (h).

3.5 Determination of Ash Content (%) of Activated carbon at Different Reaction Temperature ($^\circ C$)

Fig. 5, shows the ash content of *Khaya senegalensis* shell activated carbon which was activated at different temperature range and at

constant concentration of K_2CO_3 and time. The effect of activation temperature as seen on Figure 5, the higher the temperature the higher the ash content. Activated carbon at 500 to $700^\circ C$ shows a gradual increase in ash content from 3.2 to 3.7% and as the temperature increases from 700 to $900^\circ C$ the percentage ash content was observed to be 3.7 to 4.8. % .



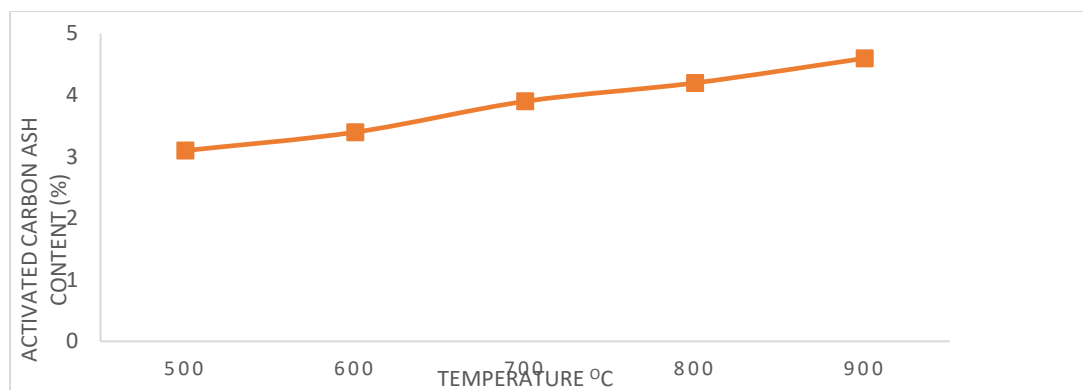


Fig. 5: Ash content (%) of activated carbon at different reaction temperature (°C), constant impregnation ratio (gdm^3) and time (h).

3.6 Determination of Ash content (%) of activated carbon at varying impregnation ratios

Fig. 6: shows Ash content (%) of activated carbon at different impregnation ratio (gdm^3), constant reaction temperature (°C), and time (h). It was observed that an increase in impregnation ratio increases the percentage ash content. The adsorption capacity of activated

carbon depends on many factors, such as activation temperature and species of activating agents (Ahmed & Theydan, 2012). This may be due to the enhanced activation reactions which cause burn-off of the structure to form larger pores (González *et al.*, 2009).

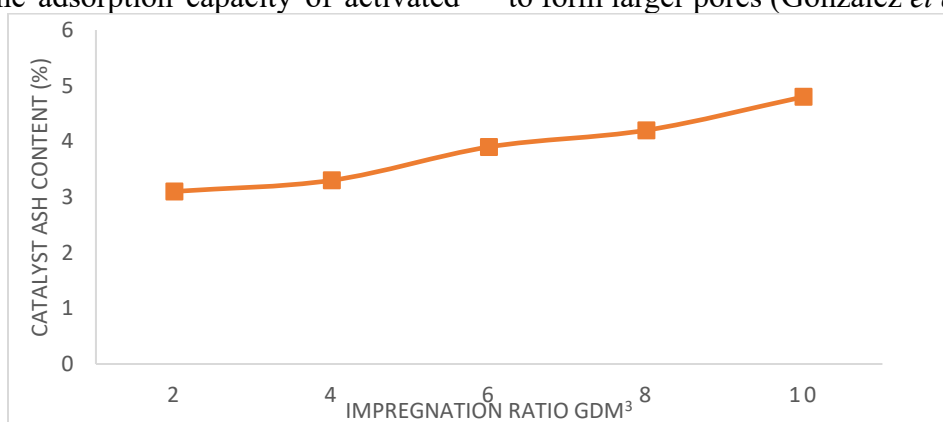


Fig. 6: Ash content (%) of activated carbon at different impregnation ratio (gdm^3), constant reaction temperature (°C), and time (h).

3.7 Determination of moisture content (%) of activated carbon at different reaction temperature

Fig. 7: At different reaction temperature, the moisture content in *Khaya senegalensis* shell activated carbon moisture content decreases with increasing reaction temperature at 500 °C. The moisture content was observed to be 7.9 %

and the lowest moisture content was observed to be 3.0% at 900°C.

Menya *et al.* (2020) & Janković *et al.* (2019) observed that biomass suitable for carbonization should have a moisture content not exceeding 30wt%, as this reduces the need for heat energy and time that would otherwise be employed to vaporize the high moisture content in the biomass.



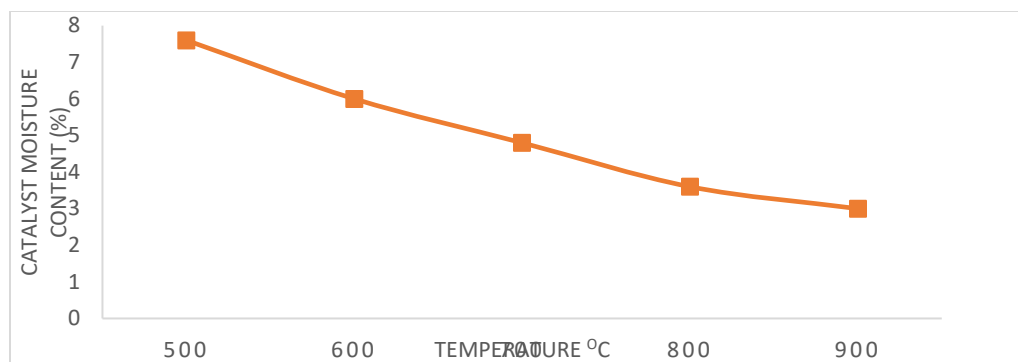


Fig. 7: Moisture content of activated carbon at different reaction temperature (°C), constant impregnation ratio (gdm^3) and time (h).

3.8 Determination of Moisture content (%) of activated carbon at different impregnation ratios

Figure 8: shows a gradual increase in moisture content as the impregnation ratio increases from 2 to 10 gdm^3 . Moisture content increases from 4.8 to 8.9 in *Khaya senegalensis* shell activated carbon.

Ridzuan *et al.*, (2021) observed the effect of H_3PO_4 : precursor impregnation ratio (3, 4, and 5) and activation temperature (300, 400 and 500 °C) on the yield of mangrove based activated carbon. At an activation temperature of 300 °C, a gradual decrease in the yield of activated carbon (45–41%) was observed as the impregnation ratio was increased from 3 to 5. It should be noted that a decrease in the yield of the activated carbon with respect to an increase in the impregnation ratio could be due

to the reaction between the H_3PO_4 with the char and volatile matter that occurred during the activation process. At high H_3PO_4 content, more phosphoric acid could react with the carbon and volatile matter and diffuse out of the surface of the particles faster during the activation process. Therefore, the gasification of surface carbon atoms became major thus led to an increase in weight loss and contributed to low yield of activated carbon (Kumar *et al.*, 2016 & Yakout *et al.*, 2016). Prahas *et al.* (2008) in which a gradual reduction in the yield of activated carbon was observed as the ratio of activating agent to precursor had been increased. However, at an activation temperature of 400 °C, the yield of activated carbon decreased slightly from 40 to 38% at an impregnation ratio of 3 and 4, respectively before experienced a sudden enhancement in the yield of activated carbon to 41% at an impregnation ratio of 5.

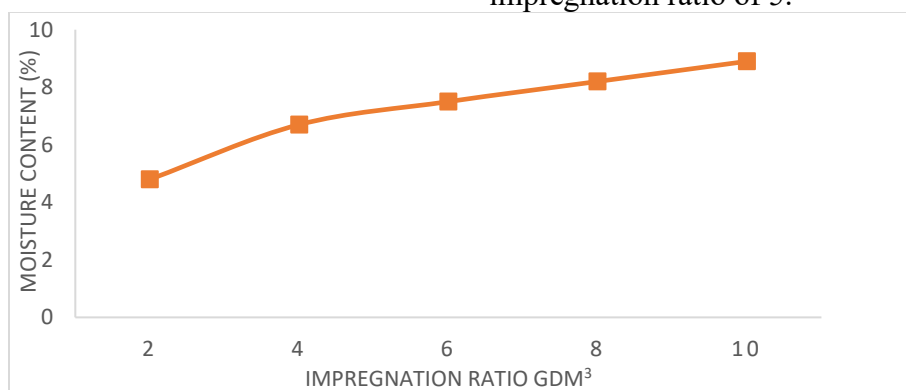


Fig. 8: Moisture content of activated carbon at different impregnation ratio (gdm^3), constant reaction temperature (°C), and time (h)



3.9 Morphology of activated carbon

Fig. 9 shows the SEM micrograph of the activated carbons produced from *Khaya senegalensis* activated carbon produced at 700 °C. The Figure 9 shows some crystals with pores and cavities of different shapes and sizes on the external surface of chemically prepared *Khaya senegalensis* activated carbon, the crystal which are most likely to be the potassium compounds which was used as activating agent during activation. Figure 10 shows a similar micrograph, the external surface of the activated carbon produced at 3:6 K_2CO_3 and carbon impregnation displays some crystals on the surface with some pores and ities on the surface. The crystal is also

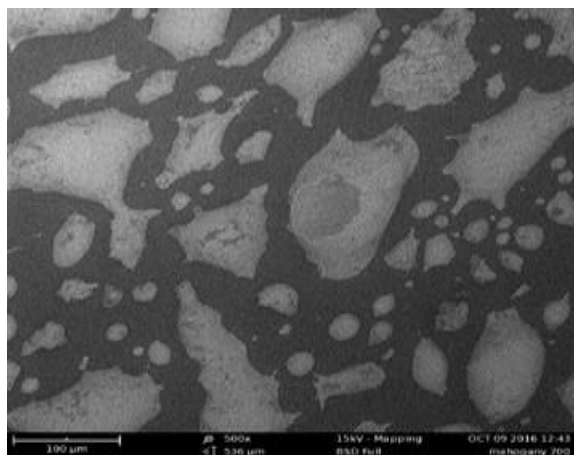


Fig. 9: SEM micrographs of the activated carbons produced at 700 °C.

3.10 Fourier Transformed Infrared (FT-IR) for the Activated carbon

Fourier transform infrared (FTIR) spectroscopic analysis was used to analyzed the functional groups on the surface of the prepared activated carbons from *Khaya senegalensis* shell.

Fig. 11, shows the spectrum of *Khaya senegalensis* shell activated carbon produced at 700°C the observable spectra indicate C-H stretching at 3134.7cm^{-1} , an -OH (ester) at $2482,4\text{cm}^{-1}$, the Wavelength at 2117.1cm^{-1} can

presumed to be the potassium compound. Paryanto *et al.* (2019) observed the surface morphology of activated carbon from Mangrove Waste, the increasing of concentration of KOH encouraged the alteration of pores, indicating that the volatile matter was released during the activation process. The releasing of volatile matter enhanced the pore structures of activated carbon (Özhan *et al.*, 2014). Moreover, a higher concentration of KOH not only produce pores but also dramatically destroys the carbon structure as reported by Budi *et.al.* 2016). During the activation process, the reaction mechanisms are given as follows Luo *et al.*, 2019).

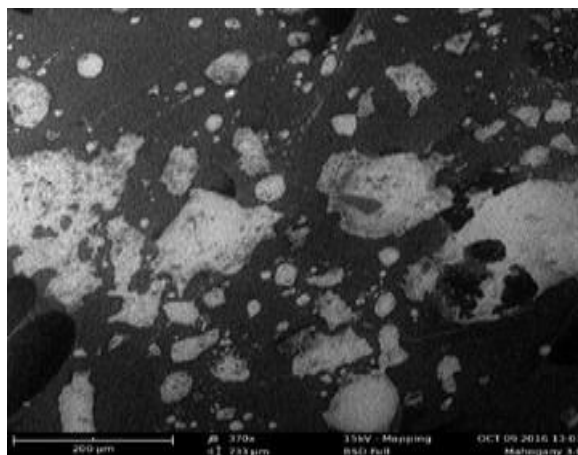


Fig.10: SEM micrograph for the chemically produced activated carbon produced from K_2CO_3

be associated to C-C stretching, 1759.3cm^{-1} corresponds to C=O, 1442.5 could be associated to C-O stretching and a C-H bending at 700.7cm^{-1} .

Yakout *et al.*, (2016) observed FTIR spectra of the synthetic carbons obtained by phosphoric acid activation at different concentrations. All spectra show a wide transmittance band at $3200\text{--}3600\text{ cm}^{-1}$ with a maximum at about $3420\text{--}3440\text{ cm}^{-1}$. This band can be assigned to the O-H stretching mode of hydroxyl groups and adsorbed water.



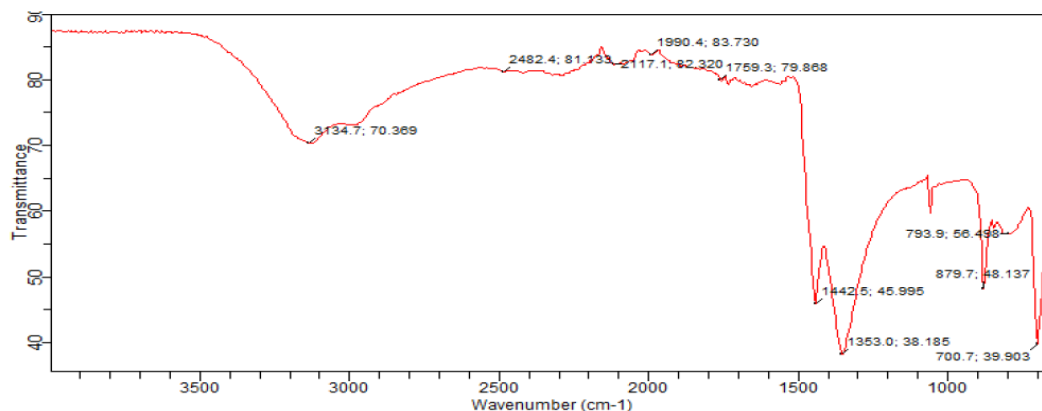


Fig. 11: Fourier transform infrared for *Khaya senegalensis* activated carbon prepared at 800°C

Other bands were found at 1625.1 cm^{-1} which matches C=O, 1360.5 cm^{-1} corresponding to C-O stretching and 704.5 cm^{-1} corresponding to C-H bending. Assignment in this region is difficult because absorption bands are overlapped. The peak at 1190–1200 cm^{-1} may be also assigned to the stretching mode of hydrogen-bonded PO, to O–C stretching vibrations in P–O–C (aromatic) linkage and to POOH (Puziy *et al.*, 2002). The shoulder at 1100 cm^{-1} was ascribed to ionized linkage P–

O– in acid phosphate esters, and to symmetrical vibration in a P–O–P chain (Bourbigot *et al.*, 1995). To conclude on IR characterization, the most important changes introduced by the increase of the acid concentration are the development of C–H vibrations (possibly because of the loss of oxygen at the surface of the carbon material) as well as the increase of phosphorous group content ($\sim 1100 \text{ cm}^{-1}$).

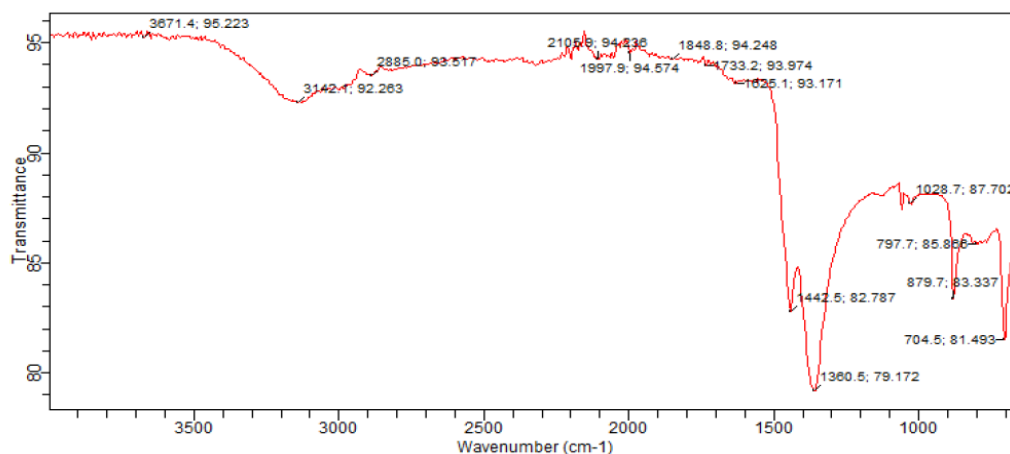


Fig. 12: Fourier transform infrared for *Khaya senegalensis* shell activated carbon produced at 3:6 K_2CO_3

4.0 Conclusion

At the end of these studies, the effect of temperature on the percentage yield of activated carbon produced, effect of pH, Fourier Transform Infrared analysis,

morphology, ash content, moisture of activated carbon produced by chemical activation of *Khaya senegalensis* shell was studied. It identifies non-edible potential raw materials



and produced heterogeneous activated carbon (Activated supported on K_2CO_3) based on indigenous sourced raw materials. It shows that, heterogenous activated carbon can be produced from *Khaya senegalensis* shell which is an agricultural waste and can be further used as activated carbon for substrate biodiesel production.

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Declarations

The authors declare that they have no conflict of interest.

Data availability

All data used in this study will be readily available to the public.

Consent for publication

Not Applicable.

Availability of data and materials

The publisher has the right to make the data public.

Competing interests

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Authors' contribution

Both authors were involved in the design of the work and in the experimental and reporting aspects.

