

Production of biodiesel from *Balanite aegyptiaca* seed oil using chemical-activated catalyst produced from coconut shell

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ABSTRACT

Heterogeneous catalyst produced from coconut waste shell (CWS) via chemical activation was utilized for biodiesel production from *Balanite aegyptiaca* seed oil. The effects of impregnation ratios (2 – 10 g/cm³), activation temperatures (500 – 900 °C) and time (1 – 3 h) on the yield of catalyst were investigated. The chemical-activated catalyst produced from coconut shell was characterized by Scanning Electron Microscopy (SEM) and Fourier Transform Infra-Red (FT-IR) spectroscopy. The biodiesel was further characterized using GC-MS. The heterogeneous catalyst produced by chemical activation under the following optimum conditions: activation temperature (800 °C), impregnation ratio (3:6g/cm³), and reaction time (1 h) was of high yield. SEM images showed a highly porous characteristic, with lots of cavities and strands on the surface. The optimum conditions to achieve maximum yield of the biodiesel (82.9%) were reaction temperature (40 °C), methanol/oil molar ratios (9:1 cm³), catalyst concentration (1.0 g/cm³) and reaction time (60 min). The fuel properties of biodiesel produced by chemical activation catalyst were: kinematic viscosity (5.70 mm²/s), specific gravity (0.86), pour point (9 °C), flash point (185 °C), cloud point (9 °C), colour 1.0 and cetane number 62. The GC-MS analysis demonstrated the presence of decanoic acid methyl ester, hexadecanoic acid, methyl ester, 4-hexenoic acid methyl ester and pentadecanoic acid methyl ester. The study showed that chemically produced heterogeneous catalyst from coconut waste shell can be used for the production of biodiesel.

Key words: Catalyst, Coconut, Shell, *Balanite aegyptiaca*, Biodiesel.

INTRODUCTION

Balanites aegyptiaca is a tree, which belongs to the *Balanites* family of plants. Its English name is Desert date. It is popularly known and called ‘adua’ in Hausa [1]. It is one of the most drought-

resistance tree species in arid regions. From the top of the tree to its roots, the desert date is well adapted to survive the extreme conditions of the desert [2].

Guo and Lua [3] successfully converted palm shell into a well-developed activated carbon by thermal activation (physical activation) and chemical activation using carbon dioxide (CO_2) and H_2PO_3 . However, activated carbon prepared from palm shell using K_2CO_3 activation for carbonaceous precursors has not been thoroughly investigated. In this work K_2CO_3 will be used as an activating agent. The influence of carbonization temperatures and impregnation ratios on pore development and yield will be studied.

Donni *et al.* [4] reported that the yield of activated carbon produced from palm shell decreased as the activation temperature increased. When chemical activation with K_2CO_3 impregnation was used, increasing the carbonization temperature decreased the yield progressively due to the release of volatile products as a result of intensifying dehydration and elimination reaction. It also indicated that char of the palm shell was gasified by K_2CO_3 .

Most industrial chemical processes are catalytic in nature and most of the commercial catalysts are expensive, toxic and not environmentally friendly [5]. Most heterogeneous catalysts widely used are NaOH, KOH, CaO, CaO/NaOH [4]. Activated carbon (AC) are highly porous and adsorbent materials. They have wide applications in domestic, commercial and industrial settings. Plate 1 is the dried coconut shell gotten from a market.

Mikyitsabu and Ataitiya [6] studied the effect of temperature on the yield (%) of the activated carbon. The result shows that the yield of the activated carbon decreases with an increase in temperature. Increase in temperature digressively affect the yield of the activated carbon. Dass *et al.* [7] studied the morphology of catalyst produced from *Coco nucifera* fruit shells by physical activation at $900\text{ }^\circ\text{C}$. Cavities were visible on the surface of the catalyst of different sizes. Olivares-Marín *et al.* [8] reported that AC is also widely used on an industrial scale as an adsorbent mainly in the purification, separation of liquids and gases or as catalyst and catalyst support. The properties of AC make it advantageous for use as a catalyst support in transesterification reaction. It has a large surface area which allows the catalyst to disperse over it largely and effectively [9]. A study on AC supported over a range of support materials such as K_2CO_3 , MgO, SiO_2 demonstrate higher activity for rapeseed oil transesterification.

The purpose of this research is to produce activated carbon as a heterogeneous catalyst based on indigenous agricultural wastes (Coconut – *Cocos nuciferas*) shells impregnated with K_2CO_3 and to

produce biodiesel from seed oil of Desert date (*Balanite aegyptiaca*). Biodiesel production is undergoing rapid technological reforms in industries and academia. This has become more obvious and relevant since the recent increase in the petroleum prices and the growing awareness relating to the environmental consequences of petroleum overdependence [10]. Therefore, this study may serve as a tool to identify non-edible potential raw materials of *Balanite aegyptiaca* seed oils, for the production of biodiesel and the use heterogeneous catalyst (AC supported on K_2CO_3) based on indigenous sourced raw materials for possible optimization of bio-diesel production.

MATERIALS AND METHODS

Sample collection

Coconut shells was collected from coconut sellers in Girei market while *Balanite aegyptiaca* fruits were purchased at Girei market in Girei Local Government Area, Adamawa state, Nigeria.

Method

The coconut shell was washed, sun-dried for two days and crushed to small particle size (Fig.1). About 500 g of the crushed coconut shell was washed with distilled water till the wash water became colourless and then dried at 110 °C in an oven for 8 hours to get rid of moisture and other volatiles. The pre-treated material was kept in a sample container for carbonization and activation process [13, 14].



Plate 1: Crushed coconut shell

Chemical activation

This was carried out according to the methods of Ajayi and Olawale [9] by preparing aqueous solution of potassium carbonate (K_2CO_3) with initial concentration of 100 g/dm³. Exactly 100 g of ground coconut shell carbon was weighed into a beaker and 300 cm³ of the prepared solution of potassium

carbonate was added which was agitated for 1 hour and kept for 24 hours to allow the sample to soak. The sample was placed in an oven at 110 °C to form a paste [15]. The activation process was carried out by weighing 30 g of the paste in a crucible into a muffle furnace [16]. The activation temperature was varied from 500, 600, 700, 800 and 900 °C with activation duration of 1h. After activation, the sample was cooled to room temperature and neutralized with 1molar HCl (36.50g/dm³), then washed with distilled water until the pH was constant. The paste formed after washing was dried at 100 °C in an oven and removed to cooled at room temperature. The activated carbon produced was ground and sieved with 106 µm, and then put in an air tight bottle. The following parameters was determined on the catalyst prepared: percentage yield, pH, percentage ash and moisture content. The morphology and composition of the catalyst were also analyzed using FTIR.

Effect of temperature on the yield of catalyst produced by chemical activation of coconut shell (ASTM D5373-99).

The total yield (%) of coconut shell produced by chemical activation was determined after sample processing in terms of raw material mass. The dried weight, W^o of the pre-treated and carbonized samples were determined using Metler balance and the carbon yield calculated as

$$Y = \frac{W \times 100}{W^o}$$

Where Y = Carbon yield (%); W = final weight of catalyst prepared; W^o = initial weight of the sample used in the carbonization and activation processes [17].

pH of catalyst produced by chemical activation of coconut shell

The pH was determined according to ASTM D3838-80. About 1.0 g each of the catalyst produced by chemical activation of coconut shell was weighed and transferred into separate 250 ml beaker and 100 ml of distilled water was added and stirred for 1 hour. The samples were allowed to stabilize and then the pH was measured using a handheld pH meter (Jenway 430 model) [18].

Ash content (%) of catalyst produced by chemical activation of coconut shell

Ash content determination was done according to the ASTM D2866-94 method. About 2 g of each of coconut shell catalyst was placed in separate porcelain crucibles which were 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 desiccator to cool. The crucible and content

were reweighed and the weight loss was recorded as the ash content of the catalyst (W_{ash}). The % ash content (dry basis) was calculated from the equation [19].

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

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

Moisture (%) of catalyst produced by chemical activation of coconut shell

Moisture content was determined according to ASTM D2867-99. Catalyst produced from coconut shell (1g of each) was weighed and dried in an oven set at 110 °C. The drying sample was constantly reweighed at a 10 minutes' interval until a constant weight (W_p) was obtained. The crucible and its content were retrieved and cooled in 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 [19].

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

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.

Fourier Transform Infrared analysis of catalyst produced from coconut shell

Catalyst was analyzed using a PERKIN-ELMER spectrum One FT-IR spectrophotometer. Each sample was ground to fine powder and oven dried at 110 °C for 2 hours and turned into pellet hydrolytically. The pellet was analyzed immediately and the spectra produce were recorded. A pellet prepared with an equivalent quantity of pure KBr powder was used as control [20].

***Balanite aegyptiaca* seed preparation**

The seed of *Balanite aegyptiaca* was prepared by soaking the seed in water for 24 h to remove the glycoside pulp from the seed coats. The washed seeds were air dried. A metal hammer was used to crush the seed to obtain its seed nut. The resulting nuts were oven-dried at 60 °C for 2 hours and stored in a clean container [6, 20].

Oil extraction from *Balanite aegyptiaca* seeds

The dried nuts obtained were ground with (mortar and pestle) into powdery form to increase the extent of extraction of the oil. Oil was extracted from the seed by solvent extraction method using soxhlet

extractor and n-hexane (b.pt 67-68 °C) as the solvent. The solvent was distilled off from the mixture by distillation and the oil was recovered for further analysis and use [21].

Determination of oil yield (%) of *Balanite aegyptiaca* seeds

The percentage yield was carried out according to the method adopted by Manji *et al* [21]. *Balanite aegyptiaca* seeds oil extracted was purified to remove solvent. The oil was weighed and the percentage of oil yield was calculated using the equation:

$$Y_{oil} = \frac{W_{oil}}{W_{sample}} \times 100$$

Where, W_{oil} = weight of extracted oil, W_{sample} = weight of sample (g).

Determination of saponification value of *Balanite aegyptiaca* seeds oil

This was carried out according to the method adopted by Pei-Hsing *et al.* [22]. About 2.0 g of the crude oil was weighed into a 250 ml conical flask and 10% alcoholic KOH was added. It was refluxed over steam for 1 hour with occasional swirling. About 1ml of phenolphthalein indicator was added at the end of the refluxing time and solution titrated with 0.5 M HCl.

Determination of iodine value of *Balanite aegyptiaca* seeds oil

Two grams of the crude oil was measured into a 100 ml conical flask and 5 ml Dam's iodine was added. The flask was corked and placed in a dark cupboard for 5 min. About 5 ml of 10% KI was added followed by 20 ml of distilled water. The solution was titrated with 6.6% sodium thiosulphate in the presence of 1 ml of 1% starch indicator until the blue colour turned colourless [23].

Determination of free fatty acid of *Balanite aegyptiaca* seeds oil

This is the percentage by weight of specific fatty acid. The method used is as described by Seer *et al.* [24]. About 2.0 g of the crude oil was weighed into a conical flask and neutralized with 1:1 mixture of 95% ethanol and 10 ml diethyl ether was added and mixed thoroughly. The solution was titrated using 0.1 M NaOH and phenolphthalein indicator with constant stirring until a pink colour which persisted for 15 seconds was observed.

Determination of moisture content *Balanite aegyptiaca* seeds oil

Moisture content of oil is expressed as the weight loss (%) when the oil dried to a constant weight at 110 °C. Crucible and content were heated in an oven at 110 °C then cooled in a desiccator and weighed. This process was repeated until a constant weight was attained [25].

Determination of specific gravity of *Balanite aegyptiaca* seeds oil

This procedure was used to measure the specific gravity of the biodiesels. A clean dry empty 50 ml density bottle was weighed. It was then filled up with distilled water and weighed, the mass of the bottle and water were taken. *Balanite aegyptiaca* seeds oil was also weighed in a separate density bottle which was weighed, the weight of bottle and oil, the specific gravity were evaluated [9, 22].

Biodiesel production from *Balanite aegyptiaca* seeds oil

Methanol and *Balanite aegyptiaca* seed oil were used as raw materials to study the effect of varying reaction conditions using molar ratio of methanol to oil in ratios of 1:3, 1:6, 1:9, 1:12 and 1:15, temperature ranges from 40 to 80 °C, stirring time and catalyst concentrations of 0.50, 0.75, 1.00, 1.25 and 1.50. Single stage step by step trans-esterification was used in the production of biodiesel. The reaction mixture consists of *Balanite aegyptiaca* seed oil, methanol and catalyst produced from coconut shell. Exactly 30 ml of *Balanite aegyptiaca* seeds oil was measured and poured into 250 ml conical flask and heated to a temperature of 40 °C. About 90 ml of methanol was poured into a round bottom flask with soxhlet apparatus and heated for 5 min. This was done to purify the methanol. Exactly 0.5 g of catalyst was weighed. A solution of 0.5 g catalyst and 90 ml of methanol (i.e. mole ratio of oil to methanol of 1: 3) was prepared in a 250 ml beaker [24, 25].

Fourier Transform Infrared of biodiesel produced using catalyst prepared from coconut shell

The different functional groups in the biodiesel were analyzed. The instrument model used in these studies was Perkin Elmer model spectrum-I Pc. Lipid fraction was evaporated on the thalium bromide and FT-IR spectra (Resolution: 8 cm⁻¹, Scan Number: 3) were performed [24].

Gas chromatography and mass spectrophotometry of biodiesel produced using catalyst prepared from coconut shell

Sample injection tool was placed in the oven at a temperature of 50 °C maintained for 1 min. The temperature was increased to 325 °C at the heating rate of 10 °C/min and held for 2 min. The GC-MS interface temperature was set to 250 °C with helium used as a carrier gas with a constant flow of 1.2 ml/min. NIST Library was used to identify the compounds representing peaks in the GC spectrum [3, 5].

RESULTS AND DISCUSSION

Chemical analysis of coconut catalyst produced by chemical activation

Effect of temperature and impregnation ratio

For activation with chemical substance (K_2CO_3), the reaction temperature was varied at constant impregnation ratio and time. The effect of increasing the reaction temperature and impregnation ratio shows a gradual decrease in the yield (Fig.1). Optimum temperature was found to be 800 °C which yielded 78.33%.

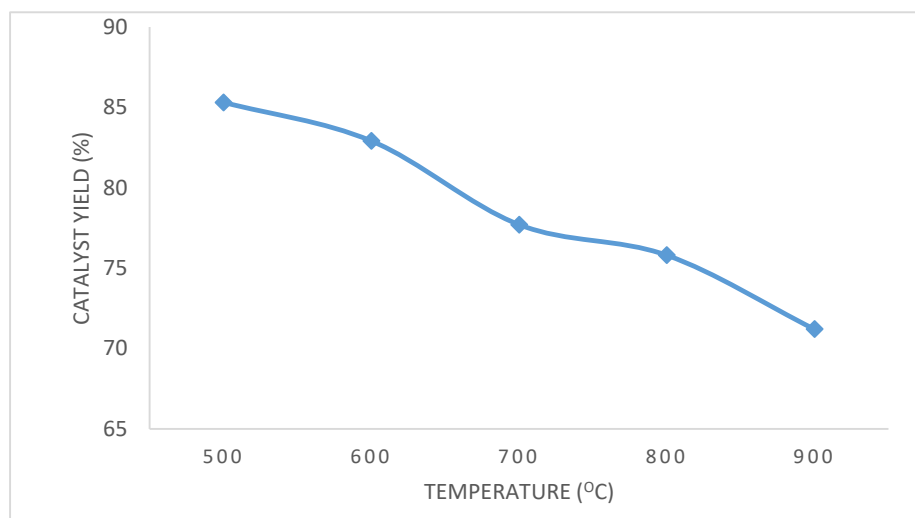


Figure 1: Effect of Temperature (°C) on the Yield of Catalyst Produced by Chemical Activation of Coconut Shell at Constant Impregnation Ratio (g/cm^3) and Time (h).

Figure 2 showed the different properties of the catalyst produced by chemical activation. The catalyst exhibited an alkaline nature for all the catalyst prepared at different activation temperatures and impregnation ratios. At 800 °C with impregnation ratio of 3:6 K_2CO_3 the pH of the catalyst was found to be 10.5. The value of pH obtained in this work is more effective because pH of catalyst obtained from lingo-cellulosic material is very effective at low pH than high pH [19].

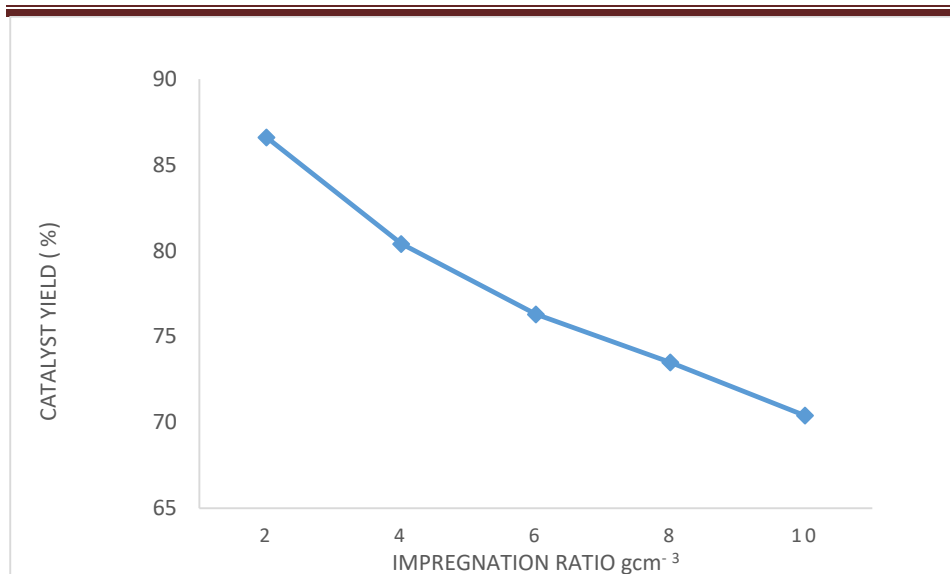


Figure 2: Effect of Impregnation Ratio (g/cm^3) on Yield (%) of Catalyst Produced by Chemical Activation of Coconut Shell at Constant Temperature $800\text{ }^\circ\text{C}$ respectively.

Moisture content (Table 1) of the catalyst produced at different activation temperatures and impregnation ratios was found to decrease with increase in activation temperatures and increase with increase in impregnation ratio. Catalyst produced at higher activation temperature 700 to $900\text{ }^\circ\text{C}$ show a lower moisture content of 5.4 and 3.2% respectively. This is due to the release of more volatile matter in the catalyst as a result of intensified dehydration and elimination reaction during the production of catalyst [26]. Lower moisture content increases the rate of adsorption. The catalyst shows a lower ash content of 3.8% (Table 1). High ash content is undesirable for catalyst since it reduces the mechanical strength of catalyst and affects adsorptive capacity.

Table 1: Analysis of catalyst produced by chemical activation of coconut shell

Parameter	Catalyst from Coconut Shell $800\text{ }^\circ\text{C}$, $3:6\text{ K}_2\text{CO}_3\text{ g}/\text{cm}^3$)
pH	10.5
Moisture content (%)	3.2
Ash content (%)	3.8

Spectroscopic analysis of catalyst produced from coconut shell by chemical activation

The morphology of the catalyst was carried out using Scanning Electron Microscopy. Morphology of catalyst produced by chemical activation of coconut shell is presented in Figure 3a. The external surface of the catalyst developed shows some cracks with some strands of crystals scattered on the surface of the catalyst. Figure 3b showed the type of particles present on the surface of the catalyst. The graph (Figure 3c) showed the cumulative (%) and volume (%) per area of the different particle per size. Due to carbonization and activation, volatiles and moisture content were removed producing a fixed carbon mass with widening of pore networks that are present in the catalyst sample which shows the adsorption capacity of a catalyst largely depends on the number of pores and the size of the surface area. Chemical activation process has successfully increased the surface area and porosity of the catalyst [27].

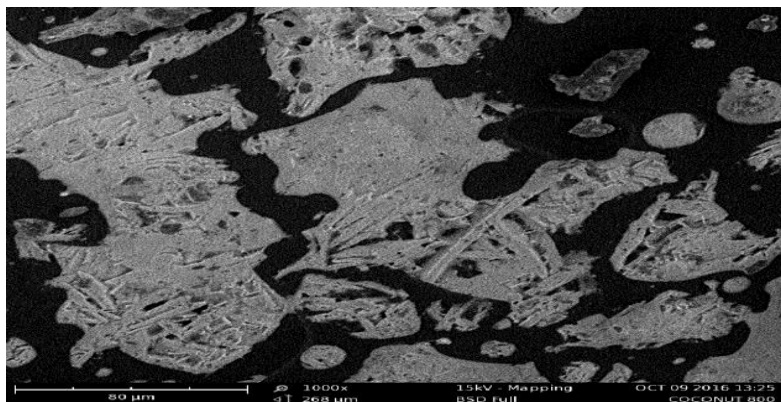


Figure 3a: Morphology of catalyst produced by Chemical activation of Coconut shell

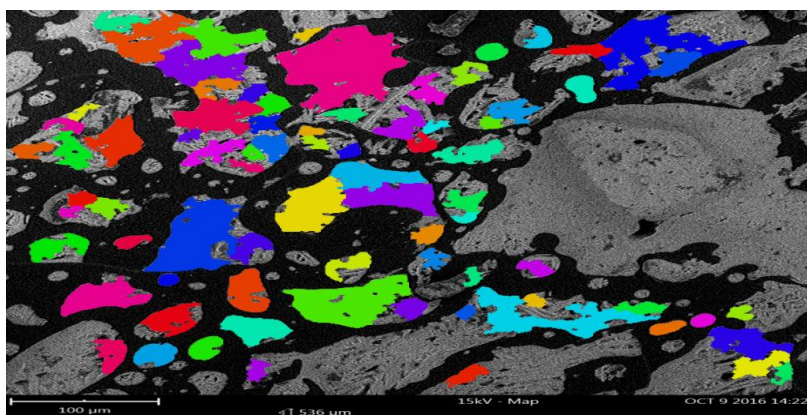


Figure 3b. Types of different particles on the of the catalyst produced by chemical activation of coconut shell

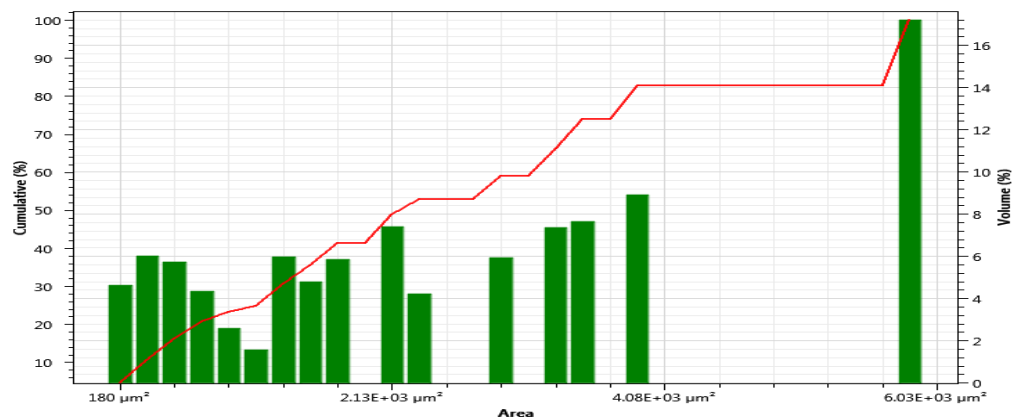


Figure 3c. Graph of cumulative (%) against area of particles of catalyst produced by chemical activation coconut shell

Fourier Transformed Infrared of catalyst produced by chemical activation coconut shell

Figure 4 showed the FTIR of catalyst produced by chemical activation of coconut shell. The spectra displayed broad band at 3138.4 cm^{-1} , 2288.6 cm^{-1} , 1654.9 cm^{-1} , 1442.5 cm^{-1} , and weak peak 700.7 cm^{-1} . The broad band at 3138.4 cm^{-1} is associated to $-\text{OH}$ stretching; 2288.6 cm^{-1} corresponds to C-C stretching and 1654.9 cm^{-1} corresponds to C=C stretch. The peak at 1442.5 cm^{-1} is attributed to C-O of carboxylic group and the weak peak at 700.7 cm^{-1} corresponds to C-H bending vibration of benzene rings. The adsorption capacity of the catalyst depends upon porosity as well as the chemical reactivity of functional groups at the surface of the catalyst [28].

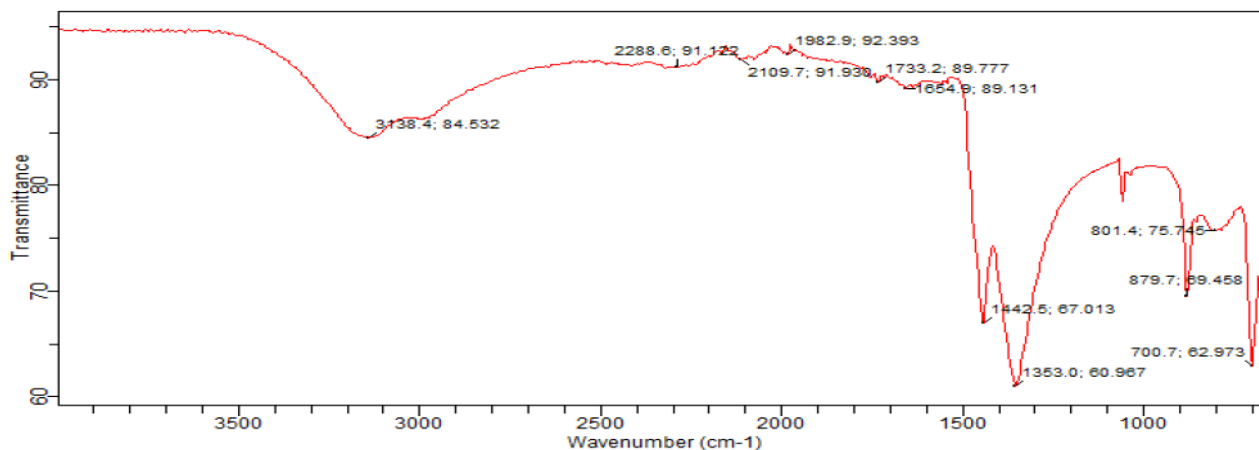


Figure 4: Fourier Transform Infrared of Catalyst Produced by Chemical Activation of Coconut Shell Produced at $800\text{ }^\circ\text{C}$.

Chemical analysis of *Balanite eagyptiaca* seed oil and biodiesel

Physico-chemical analysis was carried to study the potentiality of the oil as a biodiesel feed stock. The results obtained (Table 2) showed different properties of the seed oil which falls well within the range of biodiesel feed stock in terms of yield, saponification, free fatty acid, iodine value and specific gravity. The oil showed a lower moisture content. This indicates that the oil can have a long shelf live and a non-drying characteristic which can be considered suitable for biodiesel production because of less or no volatile compounds present [27, 28].

Table 2: Analysis *Balanite aegyptiaca* seed oil

Parameter	Value
Oil yield %	49.9
Saponification value (mgKOH/g)	168.6
Iodine value (g/100 g)	78.7
Free fatty acid (mgKOH/kg)	0.18
Moisture content (%)	0.27
Specific gravity	0.927
Colour	Pale yellow
Class of oil	Non drying

Biodiesel was produced at different reaction condition, the optimum condition for the production of biodiesel in this work gave the following parameters (Table 3) which falls within the specification of biodiesel according to ASTM D6751. The results show a considerable biodiesel yield with low specific gravity and kinematic viscosity. Fuel properties (Table 4) showed a higher flash point, cloud point, pour point and cetane number which falls within the range of biodiesel according to ASTM D6751.

Table 3: Analysis of biodiesel produced using coconut shell catalyst

Parameter	Biodiesel produced from coconut shell catalyst produced	ASTM D6751
Yield (%)	82.9	70.5 - 95
Specific gravity	0.86	0.86 – 0.90
Kinematic viscosity	5.7	4 – 6

Table 4: Fuel properties of biodiesel produced using catalyst produced by chemical activation of coconut shell

Parameter	Biodiesel produced from coconut shell catalyst produced	ASTM D6751
Flash point (°C)	185	100 – 190
Cloud point (°C)	9	-3 – 12
Pour point (°C)	9	-15 – 10
Colour	1.0	
Cetane number	62	48 – 65

FTIR of biodiesel produced using coconut shell catalyst

Biodiesel produced using catalyst produced by chemical activation of coconut shell (Fig. 5) displayed wavelengths of 3473.9 cm^{-1} which corresponds to $-\text{OH}$, 3004.2 cm^{-1} corresponds to C-H, 2922.2 cm^{-1} corresponds to $-\text{OH}$ (acids), 2855.1 cm^{-1} can be associated with C-H stretching, 1744.4 cm^{-1} corresponds to C=O (ester), 1461.1 cm^{-1} corresponds to C=C stretch, 1438.8 cm^{-1} corresponds to C-H bending, 1360.5 cm^{-1} corresponds to CH_3 bending, 1244.9 cm^{-1} associated to C-O, 1196.5 cm^{-1} corresponds to C-C stretching, and 879.7 cm^{-1} corresponds to C-H bending.

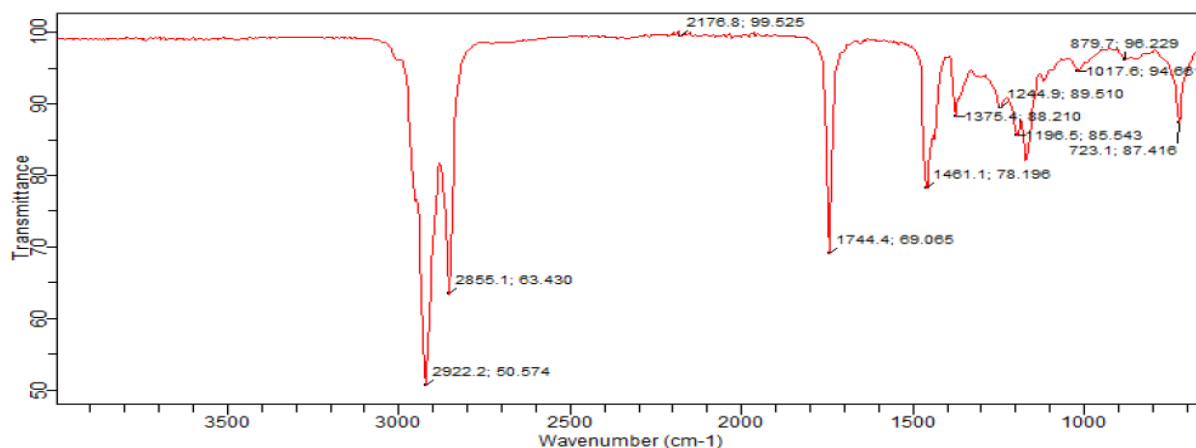


Figure 5. Fourier Transform Infrared of Biodiesel Produced using Coconut Shell Catalyst Produced by Chemical Activation

Gas Chromatography-Mass spectrometry analysis of biodiesel produced using coconut shell catalyst produced by chemical activation

Figure 6a and 6b showed the different microgram of biodiesel produced using coconut shell catalyst which was produced by chemical activation at different temperatures. Table 5 gave the different chemical compounds present at different retention time (s), percentage area and molecular weight of each compound present. Compounds represented by major peaks correspond to fatty acid methyl esters.

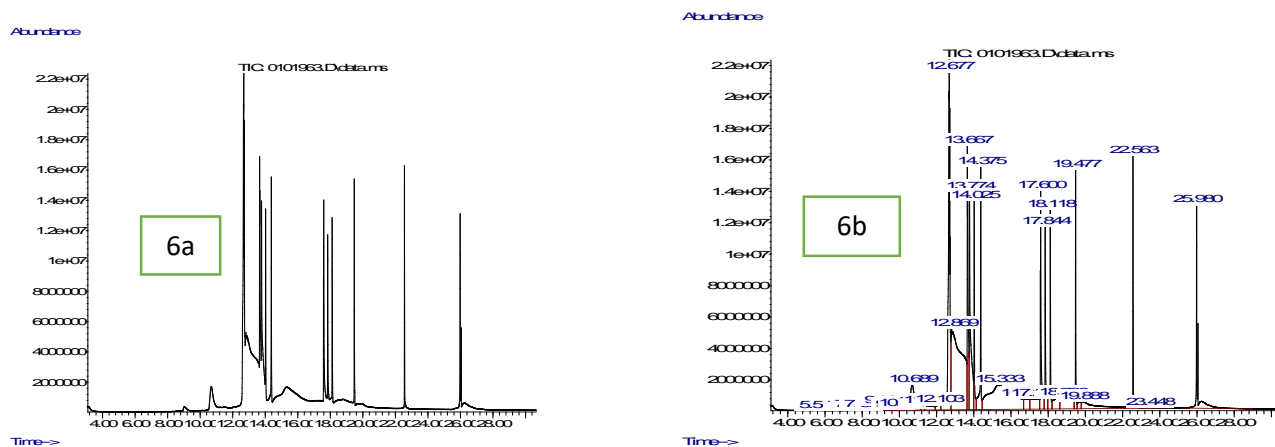


Figure 6a and 6b are the mass spectrometric analysis of biodiesel produced using catalyst produced by chemical activation of coconut shell at 800°C and at 3:6 K₂CO₃

Table 5: Different chemical compounds present at different retention time, percentage area and molecular weight of each compound present.

Peaks serial No.	Retention time (s)	Compounds	% Area	Molecular weight (g/mole)
1	5.541	13- Docosenoic acid, methyl ester (C ₂₃ H ₄₄ O ₂)	0.03	352.59
2	7.051	13- Octadecenal acid (Z), methyl ester (C ₁₉ H ₃₂ O ₂)	0.01	292.45
3	7.433	Decanoic acid, methyl ester (C ₁₁ H ₂₂ O ₂)	0.04	186.29
5	8.782	4-hexenoic acid, 3-methyl ester (C ₆ H ₁₀ O ₂)	0.12	114.14
6	9.0629	Dodecanoic acid, methyl ester (C ₁₁ H ₂₂ O ₂)	0.54	186.29
9	10.692	Tetradecanoic acid, methyl ester (C ₁₅ H ₃₀ O ₂)	0.31	242.39
13	12.679	Hexadecanoic acid, methyl ester (C ₁₇ H ₃₄ O ₂)	13.01	270.45
14	12.870	Pentadecanoic acid, 14-methyl, methyl ester (C ₁₆ H ₃₂ O ₂)	21.15	256.43
15	13.670	6- Octadecenoic acid, methyl ester (C ₁₈ H ₃₄ O ₂)	5.10	282.46
20	16.714	Heptadecanoic acid, 16-methyl, methyl ester (C ₁₉ H ₃₈ O ₂)	1.36	298.51
21	17.251	Octadecanoic acid, methyl ester (C ₁₉ H ₃₈ O ₂)	2.11	298.50
26	18.767	1-hexadecanol, 2-methyl ester (C ₁₆ H ₃₄ O ₂)	2.97	242.44
29	19.890	Hexadecanol, 2-methyl ester (C ₁₇ H ₃₂ O ₂)	2.60	268.43

CONCLUSION

Chemical activation attained an optimal condition at 800 °C, impregnation ratio of 3:6 K₂CO₃ g/cm³ and a duration of 1h. Production of heterogeneous catalyst by chemical activation of coconut shell was found to provide an alternative to the use of the costlier heterogeneous catalyst used in the production of biodiesel. This indicates that efficient heterogeneous catalyst can be obtained from cheap and easily accessible raw materials. Based on the study, it is possible to produce heterogeneous catalysts from agricultural waste and use the catalyst produced to generate bio-diesel from the seed oil of *Balanite aegyptiaca*. Therefore, the use of cheap and easily accessible heterogeneous catalysts as a means to facilitate biodiesel production both in small and industrial scales, and the search for non-edible potential raw material is an idea that is feasible and achievable.

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