Journal of Engineering, Science, and Technology
ISSN: 2714-2396 | Volume 6, Issue 1

FT-IR AND GC-MS CHARACTERIZATION OF CRUDE OIL AND BIODIESEL FROM KHAYA SENEGALENSIS SEEDS.

Zamani D. Ishaya[1, a], Samuel Joseph[1], and Hauwa Ndaji[1].

[1] Department of Mechanical Engineering, Ahmadu Bello University, Zaria, Kaduna State, Nigeria.
[a] Corresponding E-mail: waidungiz@gmail.com

ABSTRACT. Globally, for energy sufficiency, incentives are put in place to drive more sustainable alternative energy sources that are renewable and eco- friendly. Biodiesels production from varied feedstocks and application in engines are encouraged due to the fuel higher lubrication quality, excellent combustion properties and low greenhouse gas emission. Khaya senegalensis seeds, a nonedible, readily available feedstock was used to extract its bio-oil. This research explored the seeds oil feasibility as biodiesel, so the crude oil was pretreated by esterification and trans-esterified to biodiesel. The specific objectives of this research were to establish the specific chemical characteristics - compositions, the essential components, the percentiles, and the quantitative transformation of desired components from the crude oil to the biodiesel as processed from Khaya senegalensis seeds. The crude and biodiesel oils produced were characterized using Fourier Transform Infrared (FT-IR) set at scanning range of 650 to 4000 cm^{-1} and Gas Chromatography - Mass Spectrometry (GC-MS)-QO2010 Shimadzu Japan machines. The evolution of the results from the FT-IR test on the Khaya senegalensis crude oil to biodiesel oil, indicates that the functional groups increased from 10 to 11, with the addition of C=C weak stretch (alkene) at the wavelength of 1654 cm^{-1}. The GC-MS analyses confirmed that the predominant fatty acid is Oleic acid having 43.81 % by area, other esters are Nonadecanoic acid and Hexadecanoic acid. Also, the GC-MS validates the transesterification from 10 chemical compounds in the crude oil to 12 chemical compounds in the biodiesel oil. The major compounds found were fatty acids methyl esters as required for biodiesel. The natural occurring and transformed esters accounted for 95.39 % of total mass spectral area. Oleic acid ($C_{18}H_{34}O_2$) has the largest mass spectra area of 43.81 % that elute at retention time of 18.170 minutes.

Keywords: Renewable energy, Khaya senegalensis, crude oil, Biodiesel oil, Fatty acids, Esters.

1.0 INTRODUCTION

In recent times, efforts have been directed towards inventing sustainable alternative energy sources that are renewable and eco- friendly. Biodiesel has great potential; it is a renewable energy resource and can serve as alternate to conventional fossil fuels.It has a higher lubrication quality and low greenhouse gas emission [1]. Biodiesel is a fuel obtained from plant oil and is characteristically similar to petro-diesel, but it is non-toxic, renewable, sustainable, and environmentally harmless [2, 3]. Biodiesel is biodegradable. It is also used as aviation fuel, and when used in diesel engines, it presents excellent combustion properties due to its low sulfur and aromatic content, and high cetane number [4], [5]. Non-edible and microbial oil such as Khaya senegalensis, from nonedible feedstock are used in the production of biodiesel [6, 7, 8]. Most biodiesel production in the world is from edible oils, which either currently or in the future would lead to a food shortage that may create a global disparity in the food market [9]. Therefore, the use of non-edible oils such as from Khaya senegalensis has been recommended for this purpose to avoid the food-versus-fuel crisis within the country.

The established pathways for biodiesel production are pyrolysis (thermal cracking), microemulsion, transesterification, and blending processes [10], of which transesterification is the most adopted industrial method. This is due to its simple process route and its viscosity lowering potential [2, 11].

Transesterification is the reaction of triglycerides in oil or fat and alcohol in the presence of a catalyst (either an enzyme or a base or an acid) to form alkyl esters [2, 4, 12]. It is the most common method of biodiesel production from any oil resources, where triglycerides are converted into fatty acid esters through interchange of alkoxy moiety using homogeneous (acid and alkaline) or heterogeneous catalysts [13 – 14]. Homogeneous catalysts are widely used for biodiesel due to their excellent catalytic activity with faster and higher yield conversions under mild reaction condition [15]. Transesterification process plays an important role in the biodiesel production as it greatly influences higher yields and cost of production. The operating conditions like, catalyst type, temperature, methanol amount, and reaction time are significantly attributed to biodiesel production [2, 3, 16, 17].

Ogwuche [18]characterized the leaves of Pandanus Candalabrum using FT-IR and GC-MS analyses. The results showed that Kaur-16-ene was the most abundant component with 84.62 % percentage abundance. Carboxylic, alkenes and renes functional group were identified at 2958.08 cm^{-1}, 1598.10 cm^{-1} and 1457.68 cm^{-1} wavenumber respectively. Odo [19]carried out FT-IR and GC-MS analyses on Brenania brieyi root extract. The results showed the presence of pentadecanoic acid (17.04 %), 9, 12-hexadecanoic acid (10.18 %), 9-Octadecanoic acid (60.53 %) in methanol extract, and hexadecanoic acid (11.29 %), 9-Octadecanoic acid (54.85%), 9, 12-Octadecanoic (3.90 %) in chloroform extracts. Kavipriya[20] used FT-IR and GC-MS to analyze the leaf extract of Cassia Alata. The FT-IR results showed sulfates, sulfonamides, sulfones, aromatic, alkane, alkenes, nitrile, and amide functional groups. The GC-MS results showed 1-Butanol, 3-methyl-1,6-Anhydro-beta-D-glucopyranose, Oxirane, and Oleic acid. Also Bolade [21] carried out FT-IR and GC-MS analyses on Canna indica. The FT-IR results showed presence of aromatic O-H stretch (3300cm^{-1}) and aromatic C=C stretch (1451 and 1640 cm^{-1}). The GC-MS result showed presence of di-alpha-tocopherol.

In this research work, production of biodiesel from khaya senegalensis with methanol through homogeneous catalyzed transesterification was reported. The quality of the purified biodiesel obtained in optimal conditions was characterized by FT-IR and GC–MS. Khaya senegalensis was investigated in this research due to regional availability, and limited previous studies on this subject was conducted on the feedstock. Principally, this research established the chemical characteristics - compositions, the essential components, the percentiles, and the quantitative transformation of desired components from the crude oil to the biodiesel as processed from Khaya senegalensis.

2.0 MATERIALS AND METHODS

2.1 Collection of Samples

Khaya senegalensis originate from tropical Africa, Madagascar. Khaya senegalensis is a flowering plant in the family Meliaceae, of the Angiosperms group in the Sapindales order. Khaya senegalensis is a medium size, deep root drought resistant tree, often buttressed at the base. The barks of the tree changes from dark grey to a grey-brown pigment at maturity. Its leaves are arranged in a spiral formation of 4-6 pairs without terminal leaf. The tree has deciduousevergreen leaves. It has white, sweet-scented flowers and the matured fruit is globose, four or five-valved woody capsule of 4–6 cm diameter, containing numerous winged seeds. The tree can also grow well in subtropical conditions.

The key material for this characterization is the Khaya senegalensis winged seed kernels collected in Kagoro and environs of Kaura Local government area of Kaduna State in Nigeria. The oil was extracted from the seed kernels using mechanical expeller press, where the yield varies from 14 % to 43 % by weight [2, 4, 16]. This implies that crude oil from this feedstock has high commercial viability for biodiesel production. Other materials used in this research are analytical grades solutions of methanol (CH_3OH), propan-2-ol (C_3H_7OH), sulphuric acid (H_2SO_4), 0.1 Mole sodium hydroxide (NaOH) pellets and 0.1 Mole potassium hydroxide (KOH) pellets as the major reagents employed for this experiment

2.2 Preparation of Biodiesel

Part of the methods of the characterization includes the biodiesel production, which involves pretreatment method of the oil, followed by the transesterification method. The procedure was to use 10.0 g of the extracted oil added to 240.0 g of propan-2-ol. The mixture was titrated using 0.1 Mole of potassium hydroxide and a blank titration was conducted. The FFA value and acid value (AV) of the oil were evaluated using Equations 1 and 2. The process cycle was repeated until the AV value of ≥ 0.5 % was achieved [16].

$$\text{Free Fatty Acid (FFA)} = \frac{\text{KOH Titre Value x Molarity of KOH x Molar Mass of KOH}}{\text{Mass of oil}} \tag{1}$$

% FFA = (v x 0.1 M x 56.1 g /mol) / 10.0g Where, v is the titre value.

$$\text{Acid Value (A.V.)} = \frac{\%\ \text{FFA}}{2} \tag{2}$$

Normally, the stoichiometric reaction requires a mole of oil (Triglyceride) and three moles of alcohol (Methanol) as expressed in Equation 3.

$$\underset{\text{Triglyceride}}{R\text{-}CO\text{-}OR'} + \underset{\text{Alcohol}}{R''\text{-}OH} \xleftrightarrow{H^+ \ or\ R''O^-} \underset{\text{Methyl Esters}}{R\text{-}CO\text{-}OR''} + \underset{\text{Glycerol}}{R'\text{-}OH} \tag{3}$$

The transesterification was conducted using 240 g of the esterified oil with 52.08 g of methanol for an oil to alcohol molecular mass ratio of 1:6 in excess times two, and 1.8 g (0.75 % m/m of oil) of NaOH as catalyst loaded times three the normal weight, for 1-hour at 60 ^{o}C to produce the Khaya senegalensis biodiesel. Similar process may be conducted for biodiesel production at higher oil to methanol ratios.

The required reagents used were methanol (CH_3OH), sulphuric acid (H_2SO_4), 0.1M potassium hydroxide (KOH) pellets, propan-2-ol (C_3H_7OH) and sodium hydroxide (NaOH) pellets. The reagents were of analytical grade (Assay 99.5 %) manufactured by BHD Chemical Ltd, Polle, England, and sourced from Cardinal Nig. Ltd, Kwagila in Zaria, Nigeria and were used as received.

2.3 Fourier Transform Infrared (FT-IR) Spectroscopy

Agilent technologies FT-IR Spectrophotometer was used to take the infrared spectrum of samples. Potassium bromide disc method was used to obtain the IR spectra of samples. 650 to 4000 cm^{-1} scanning range was used. In this research, functional groups were identified in the crude oil and biodiesel samples using the FT-IR spectra.

2.4 Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS-QO2010 Shimadzu Japan was used in the analysis. A fused silica column, filled with Elite-5MS (95 % dimethylpolysiloxane 5 % biphenyl, 30 m × 0.25 mm ID × 250 μm df). Helium as carrier gas flowing constantly at 1 mL/min was used to separate the components. Temperature of injector was set at 250 °C for the chromatographic run. The extract (1 μL) was injected into the instrument and the temperature was 70 °C (0 min); followed by 280 °C at the rate of 10 °C min^{-1}; and 280 °C, where it was kept for 5 minutes. The mass detector conditions were set at transfer line temperature at 250 °C; ion source temperature of 200 °C; and ionization mode electron impact of 70 eV, a scan time of 0.5 seconds and scan interval of 0.1 seconds respectively. In this research, compounds were detected in the crude oil, its biodiesel, and their respective quantities with GC-MS spectrometer.

2.5 Compounds Identification

National Institute Standard and Technology (NIST) was used as a reference to compare and interpret the results. Compounds were identified according to molecular mass, structure, and fragments. The unknown components spectrum was compared with that of the NIST library, and the closest match is taken.

3.0 RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared Spectroscopy (FT-IR) Analysis

3.1.1 Crude Khaya senegalensis oil

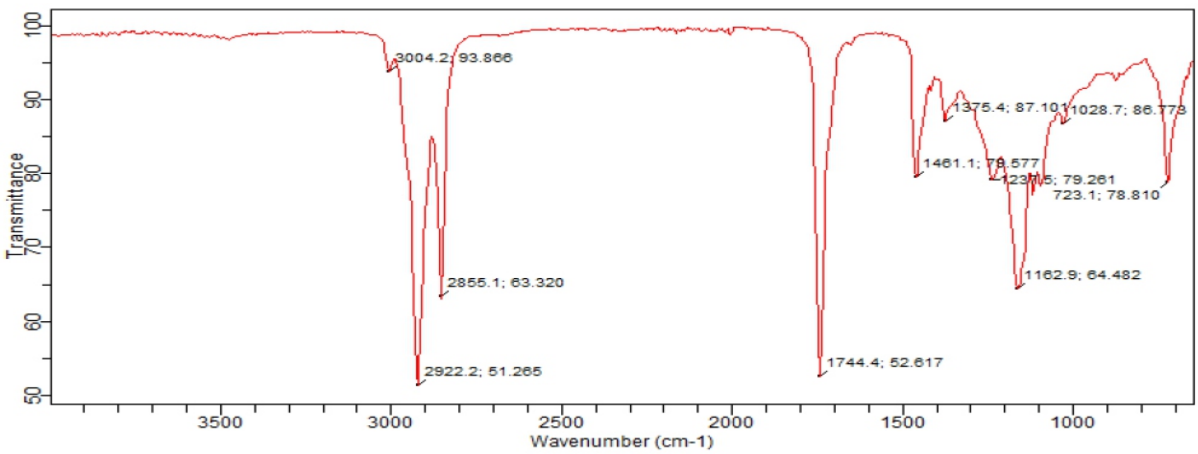

Fig. 1. FT-IR spectra of crude Khaya senegalensis oil.

Table 1. Functional groups in crude Khaya senegalensis oil.

S/N	Wavelength (cm^{-1})	Functional group
1	**3004**	**=C-H weak stretch (alkene)**
2	**2922, 2855**	**C-H strong stretch (alkanes)**
3	**1744**	**C=O strong stretch (alkanoic & ester)**
4	**1461**	**C-H medium stretch (terminal alkane bend)**
5	**1375**	**C-O weak stretch (alkanoic)**
6	**1103-1236**	**C-O medium stretch, (esters)**
7	**1028**	**C-O-C weak stretch (alkanoate)**
8	**723**	**C=C-H medium stretch (alkene bending)**

Tulashie [22] assigned 3000 cm^{-1} to -CH bond, 2853.09 and 2922.27 cm^{-1} to $–CH_2$ stretching vibrations, 1741.91 cm^{-1} to –C=O bond stretching, 1436.22 cm^{-1} to $–CH_3$ asymmetric stretching and 1196.28 cm^{-1} due to $O-CH_3$ stretching. In the present research, as indicated in Fig. 1 and Table 1; =C-H bond was observed at 3004 cm^{-1}, C-H bond at 2855-2922 cm^{-1}, C=O at 1744 cm^{-1}, C-H terminal alkane bond at 1461 cm^{-1} and C-O-C at 1028 cm^{-1}. Cheah [23] assigned peaks at 1059 cm^{-1} to C-O stretching, 2900 cm^{-1} to alkane C-H stretching, 1700 cm^{-1} to ester C=O stretching, 1650 cm^{-1} due to alkene C=C stretching. Similarly, Mohammed [24] identified 2853.78 cm^{-1} as methyl C-H group, and 1741.78 cm^{-1} as ester. The research identified C-O ester group at 1103-1236 cm^{-1}, C-O carboxylic group at 1375 cm^{-1} and C-H bend at 723 cm^{-1}. This research is consistent with [22, 23, 24].

Based on the FT-IR result in Fig.1 in the research, it was observed that: alkanes, alkenes, carboxylic and esters functional groups were present in Khaya Senegalensis crude oil. The alkanes and alkenes functional groups were due to the presence of long fatty acid chain in the triglyceride compound. The chain usually consists of saturated and unsaturated sites. The carboxylic functional group indicates suitable site for transesterification reaction to occur. The carboxylic group is normally attached to the end of the long fatty acid chain away from the triglyceride backbone. The ester functional group was due to naturally occurring esters in plant oils.

3.1.2 Khaya Senegalensis Biodiesel

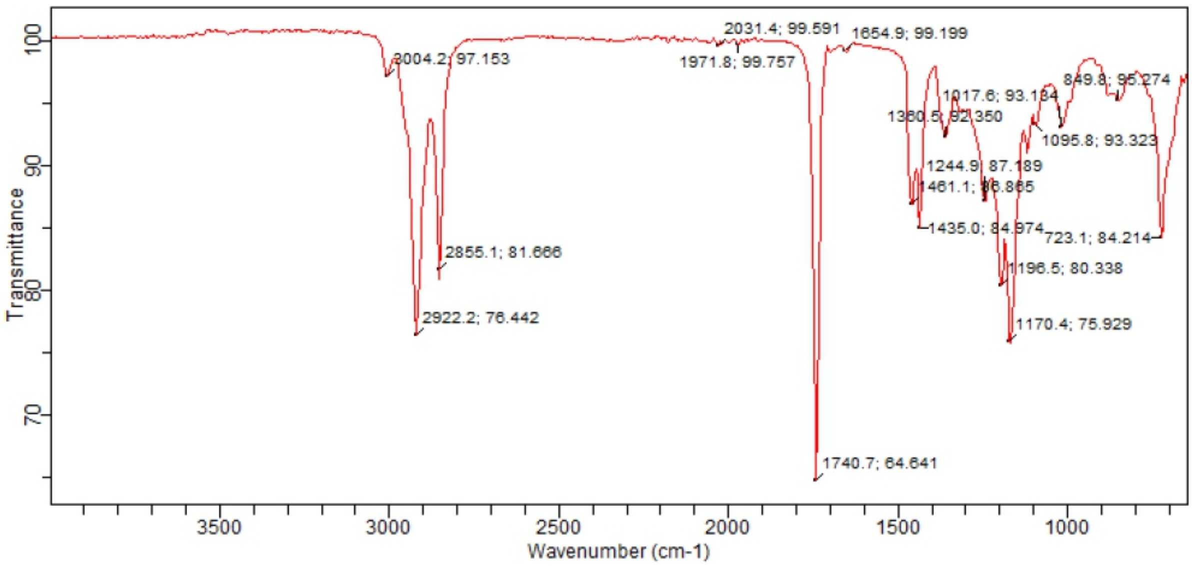

Fig. 1. FT-IR spectra of Khaya senegalensis biodiesel.

Table 2. Functional groups in Khaya senegalensis biodiesel.

S/N	Wavelength (cm^{-1})	Functional group
1	**3004**	**=C-H weak stretch (alkene)**
2	**2922, 2855**	**C-H strongh stretch (alkanes)**
3	**1744**	**C=O strongh stretch (alkanoic & ester)**
4	**1654**	**C=C weak stretch (alkene)**
5	**1461, 1435**	**C-H medium stretch (terminal alkane bend)**
6	**1380**	**C-O weak stretch (alkanoic)**
7	**1170-1244**	**C-O medium stretch, (esters)**
8	**1095**	**C-O-C weak stretch (alkanoate)**
9	**723-850**	**C=C-H medium stretch (alkene bending)**

Dass [26]observed that 1744.4, 1461.1, 1237.6, 1162.9 and 723.1 cm^{-1} were marched to C=O, C=C, C-O, C-C stretch, and C-H bending respectively in a biodiesel produced from Mahogany fruit shell. Alhassan [24] Attributed 1709, 1248 and 1611 cm^{-1} to C=O, C-O and C=C groups respectively. In this research as indicated in Fig. 2 and Table 2, C-H bond was identified at 2855-2922 cm^{-1}, C=O at 1744 cm^{-1}, C=C at 1654 cm^{-1}, C-H terminal alkane bond at 1435-1461 cm^{-1},

Journal of Engineering, Science, and Technology
ISSN: 2714-2396 | Volume 6, Issue 1

C-O at 1380 cm^{-1}, C-O-C at 1095 cm^{-1} and =C-H bending at 723-850 cm^{-1}. The present functional grouping in this research are in agreement with other research works [25 - 26].

Based on the FT-IR result in Fig. 2 in the research, it was observed that: alkanes, alkenes, alkanoic, and esters functional groups are clearly visible in the Khaya senegalensis biodiesel. The alkanes and alkenes functional groups were due to the presence of long fatty acid chain and mono-unsaturation in the fatty acid chain attached to the ester end in the biodiesel. The presence of alkanes implies the biodiesel will have high energy content and short ignition delay, but it will be faced with low pour and cloud point. Alkenes/mono-unsaturation presence implies the biodiesel will have good fluidity property, even though it will have to deal with oxidation instability and lower heating value. The long fatty acid chain will support high heating value and short ignition delay for the biodiesel, but it will contribute to high viscosity challenges. Due to the transesterification reaction, the long fatty acid chain has been detached from the triglyceride backbone to form the fatty acid methyl esters (FAME). The presence of esters in the biodiesel indicates good fuel properties like low viscosity, improved pour and cloud points, more energy per unit volume. Esters also implies the alkanoic functional group in the crude oil have undergone transesterification reaction. The remaining carboxylic group in the biodiesel may indicate incomplete transesterification reaction and results in high total acid number of the biodiesel.

3.2 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

3.2.1 Crude Khaya senegalensis oil

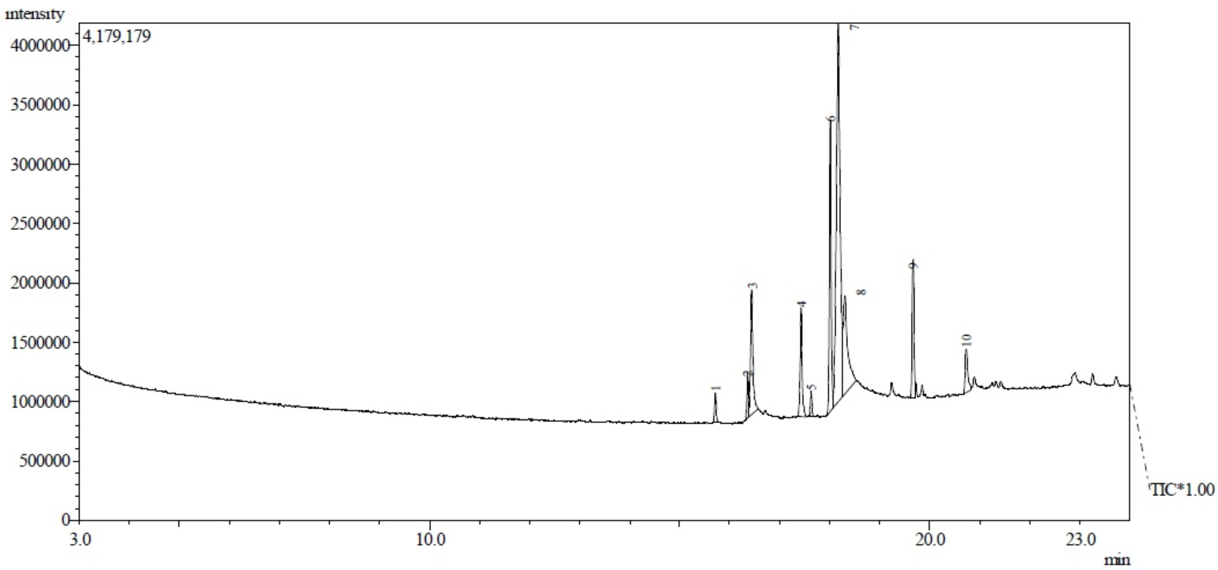

Fig. 3. GC-MS spectra of crude Khaya senegalensis oil.

Table 3. Compounds present in crude Khaya senegalensis.

*PK	*RT	Area %	Library - ID*	Formula
1	**15.713**	**1.41**	**Methyl 6-methyl heptanoate**	$C_9H_{18}O_2$
2	**16.357**	**1.98**	**Hexadecanoic acid, ethyl ester**	$C_{18}H_{36}O_2$
3	**16.440**	**9.05**	**Hexadecanoic acid**	$C_{16}H_{32}O_2$
4	**17.427**	**6.45**	**11-Octadecenoic acid, methyl ester**	$C_{19}H_{36}O_2$
5	**17.628**	**1.23**	**Methyl 8-hydroxyoctanoate**	$C_9H_{18}O_3$
6	**18.013**	**14.79**	**9-Octadecenoic acid, ethyl ester**	$C_{20}H_{38}O_2$

7	**18.170**	**43.81**	**Oleic acid**	**$C_{18}H_{34}O_2$**
8	**18.304**	**11.54**	**Nonadecanoic acid**	**$C_{19}H_{38}O_2$**
9	**19.665**	**6.60**	**cis-Oleic acid**	**$C_{18}H_{34}O_2$**
10	**19.665**	**3.15**	**10-Undecenal**	**$C_{11}H_{20}O$**

*PK= Peak, RT = Retention Time, ID = Identification.

The GC-MS analysis of crude Khaya senegalensis oil showed presence of 10 compounds as shown from the 10 Peaks (PK) in Fig. 3 and the data in Table 3. It was observed that major compounds were fatty acids, natural occurring esters and accounted for 95.39 % of total mass spectral area. Major compounds include: Oleic acid ($C_{18}H_{34}O_2$) at retention time of 18.170 min occupying the largest mass spectra area of 43.81 %, 9-Octadecenoic acid, ethyl ester ($C_{20}H_{38}O_2$) at retention time of 18.013 min with a mass spectra area of 14.79 %, Nonadecanoic acid ($C_{19}H_{38}O_2$) at retention time of 18.304 min with a mass spectra area of 11.54 %, Hexadecanoic acid ($C_{16}H_{32}O_2$) at retention time of 16.440 min occupying 9.05 % mass spectra area, 11-Octadecenoic acid, methyl ester ($C_{19}H_{36}O_2$) at retention time of 17.427 min with a mass spectra area of 6.45 %, cis-Oleic acid ($C_{18}H_{34}O_2$) at retention time of 19.665 min with a mass spectra area of 6.60 %, 10-Undecenal ($C_{11}H_{20}O$) at retention time of 20.727 min occupying 3.15 % mass spectra area.

The GC-MS result indicates that Oleic acid is the predominant fatty acid present in the crude Khaya senegalensis oil. The Oleic acid is a monounsaturated fatty acid with 18 carbon atoms. Mono-unsaturation implies the intended biodiesel will have good fluidity property. The long fatty acid chain will contribute high heating value and short ignition delay for the intended biodiesel. The presence of fatty acids indicates possible conversion to fatty acids methyl esters (FAME) that possesses good fuel properties.

3.2.2 Khaya senegalensis biodiesel

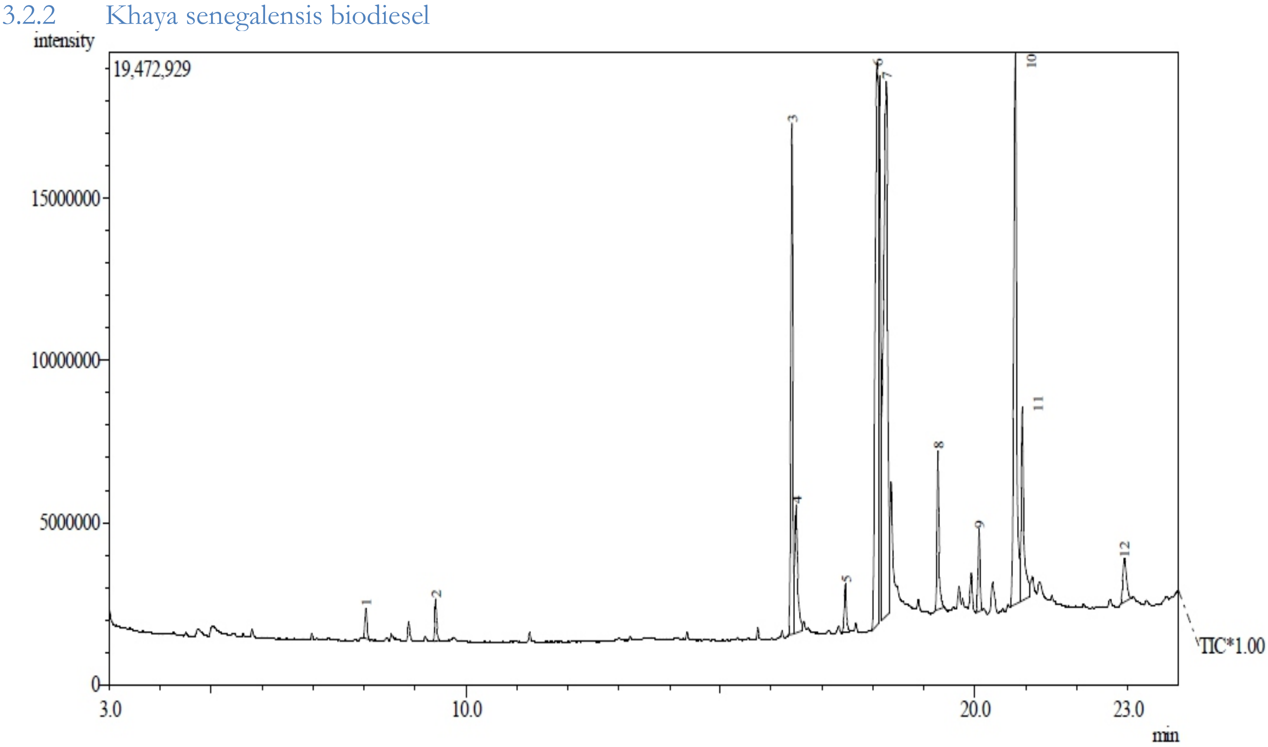

Fig. 4: GC-MS spectra of Khaya senegalensis biodiesel.

Table 4: Compounds present in Khaya senegalensis biodiesel.

*PK	*RT	Area %	Library - ID*	Formula
1	**8.037**	**0.65**	**2-Decenal, (E)**	$C_{10}H_{18}O$
2	**9.412**	**0.93**	**2-Undecenal**	$C_{10}H_{20}O$
3	**16.406**	**12.05**	**Hexadecanoic acid, ethyl ester**	$C_{18}H_{36}O_2$
4	**16.494**	**4.26**	**Palmitic acid**	$C_{16}H_{32}O_2$
5	**17.455**	**1.29**	**11-Octadecenoic acid, methyl ester**	$C_{19}H_{36}O_2$
6	**18.076**	**22.00**	**9-Octadecenoic acid, ethyl ester**	$C_{20}H_{38}O_2$
7	**18.267**	**26.50**	**Stearic acid, ethyl ester**	$C_{20}H_{40}O_2$
8	**19.275**	**3.66**	**2-Butyloctanol alcohol**	$C_{12}H_{26}O$
9	**20.080**	**1.82**	**Oleic acid**	$C_{18}H_{34}O_2$
10	**20.793**	**18.80**	**9-octadecenal**	$C_{18}H_{34}O$
11	**20.928**	**6.09**	**Stearyl vinyl ether**	$C_{20}H_{40}O$
12	**22.937**	**1.93**	**Oleic acid, 2-hydroxyethyl ester**	$C_{20}H_{38}O_3$

*PK= Peak, RT = Retention Time, ID = Identification.

The GC-MS analysis of the Khaya senegalensis biodiesel showed presence of 12 compounds as indicated from the 12 Peaks (PK) in Fig. 4 and the details in Table 4. It was observed that major compounds were esters, small fatty acids and accounted for 71.9 % of total mass spectral area. These compounds include: Stearic acid, ethyl ester ($C_{20}H_{40}O_2$) at retention time of 26.50 min occupying the largest mass spectra area of 26.50 %, 9-Octadecenoic acid, ethyl ester ($C_{20}H_{38}O_2$) at retention time (RT) of 18.1 min with a mass spectra area of 22.0 %, 9-octadecenal ($C_{18}H_{34}O$) at retention time of 20.8 min with a mass spectra area 18.80 %, Hexadecanoic acid, ethyl ester ($C_{18}H_{36}O_2$) at retention time 16.4 min occupying 12.05 % mass spectra area, Stearyl vinyl ether ($C_{20}H_{40}O$) at retention time of 20.9 min with a mass spectra area of 6.09 %, Palmitic acid ($C_{16}H_{32}O_2$) at retention time of 16.5 min with a mass spectra area of 4.26 %, 2-Butyloctanol alcohol ($C_{12}H_{26}O$) at retention time of 19.3 min occupying 3.66 % mass spectra area. Dass[26]reported 9-octadecenoic acid (Z), methyl ester at 7.678 RT, Hexadecanoic acid (Z) methyl ester at 13.790 RT, 12-Octadecenoic acid (Z), methyl ester at 15.425 RT and Oleic acid, 3-hydroxypropyl ester at 15.723 RT for a biodiesel produced from Mahogany fruit shells.

The GC-MS results indicates that the transesterification reaction has greatly reduced the percentage area of the Oleic acid in the crude oil from 43.81 % to 1.82 % in the biodiesel, with the formation of more esters. This implies the biodiesel will have low total acid number and good fuel properties such as high energy per unit volume, low viscosity, and short ignition delay. The percentage of esters increases 9-Octadecenoic, ethyl ester from 14.79 % to 22.0 % area, Hexadecanoic acid, ethyl ester from 1.98 % to 12.05 % area. New product like Stearic acid, ethyl ester occupying 26.50 % area was formed. The presence of fatty acid methyl esters (FAME) indicates successful transesterification reaction and good fuel properties. Biodiesels are the esters of fatty acids.

4.0 CONCLUSIONS

In this research, FT-IR and GC-MS characterization of crude oil and biodiesel oil from Khaya senegalensis seeds as investigated, establishes its chemical conformity as biodiesel fuel. Also, the following conclusions are drawn from the results:

1. The FT-IR analyses of the crude Khaya Senegalensis oil showed the presence of alkanes, alkenes, alkanoic and esters functional groups. The GC-MS analyses confirmed the presence of fatty acids having most of the functional groups identified in the FT-IR. The predominant fatty acid was Oleic acid having 43.81 % area. Others are Nonadecanoic acid and Hexadecanoic acid. The fatty acids are the reactive species in the oil for transesterification reaction to occur when reacted with alcohol.

2. The FT-IR analyses of Khaya Senegalensis biodiesel indicates that the functional groups increased from 10 to 11, with the addition of C=C weak stretch (alkene) at the wavelength of 1654 cm^{-1}. The alkanoic functional group in the crude oil underwent transesterification reaction.
3. The GC-MS analyses validates the transformation from 10 chemical compounds in the crude oil to 12 chemical compounds in the biodiesel oil. The major compounds were fatty acids, naturally occurring and transformed esters that accounted for 95.39 % of total mass spectral area. Oleic acid ($C_{18}H_{34}O_2$) has the largest mass spectra area of 43.81 %, elute at retention time of 18.170 min.
4. Other predominant FAME was Stearic acid, ethyl ester having 26.50 % area. More esters products with lower percent are 9-Octadecenoic acid, ethyl ester, Hexadecanoic acid, ethyl ester. The FAME are identified as biodiesel molecules.

ACKNOWLEDGEMENT.
The authors acknowledge the support of the Laboratories for the provision of the equipment used in conducting these experiments at the National Research Institute for Chemical Technology (NARICT), Zaria, Department of Chemistry and Department of Mechanical Engineering, Ahmadu Bello University, Zaria. Nigeria. Special thanks to Professor G.Y. Pam for his time and assistance in this research.

REFERENCES

[1] Elango, R. K., Sathiasivan, K., Muthukumaran, C., Thangavelu, V., Rajesh, M. and Tamilarasan, K. (2019). Transesterification of castor oil for biodiesel production: Process optimization and characterization. *Microchemical Journal, 145*, 1162–1168. doi:10.1016/j.microc.2018.12.039.

[2] Ishaya Z. D., Pam G.Y., Kulla D.M., Ngbale E. O. (2017). Non-Conventional Feedstock for Biodiesel Production for a Diversified Economy: Canarium Schweinfurtii and Thevetia Peruviana Seed oils Case. *Proceeding of Nigerian Society of Engineers (NSE) Conference on Diversification of Nigerian Economy: Engineering Perspective* at Chida Event Centre, Utako - Abuja 20th – 24th November 2017 (Technical Book of Abstracts Pg 10 and 50).

[3] Olagbende, O. H., Falowo, O. A., Latinwo, L. M. and Betiku, E. (2021). Esterification of Khaya senegalensis seed oil with a solid heterogeneous acid catalyst: Modeling, optimization, kinetic and thermodynamic studies. *Cleaner Engineering and Technology, 4, 100200.* doi:10.1016/j.clet.2021.100200.

[4] Yahuza I., **Ishaya Z. D.,** Dandakouta H. (2013).A Review of Ethanol-Diesel Blend as a Fuel in Compression-Ignition Engine.*Proceeding of the 3rd National Engineering Conference and Annual General Meeting of the Automotive Engineers Institute* at the NUT Conference Hotel, Kaduna, 26th – 27th September 2013.Pg 17 – 27.

[5] Chaudhary P., Verma A., Kumar S. and Gupta, V. K. (2018). Experimental design and optimization ofcastor oil transesterification process by response surface methodology. *Biofuels 9*:7-17.

[6] Bateni H. and Karimi K. (2016). Biodiesel production from castor plant integrating ethanol production via a biorefinery approach. *Chem Eng Res Des. 107*: 4-12.

[7] Sukkasi, S., Chollacoop, N., Ellis, W., Grimley, S. and Jai-In, S. (2010). Challenges and considerations for planning toward sustainable biodiesel development in developing countries: Lessons from the Greater Mekong Subregion. *Renew Sustain Energ Rev 14*: 3100-7.

[8] Dai, Y. M., Chen, K.T. and Chen, C. C. (2014). Study of the microwave lipid extraction from microalgae for biodiesel production. *Chem Eng J 250*: 267-73.

[9] Falowo, O.A., Ojumu, T.V., Pereao, O. and Betiku, E. (2020). Sustainable biodiesel synthesis from honne-rubber-neem oil blend with a novel mesoporous base catalyst synthesized from a mixture of three agrowastes. *Catalysts 10* (2), 190. doi: 10.3390/catal10020190.

[10] Olagbende, H.O., Aransiola, E.F., Ogunsina, B.S., Sanda, O. and Shonibare, D. (2016). Modification of a fixed bed reactor system for pyrolytic conversion of royalpoinciana pods into alternative fuels. *Int. J. Renew. Energy Resour. 6* (4),1350–1360.

[11] Juan, J.C., Kartika, D.A., Wu, T.Y. and Hin, T.-Y.Y. (2011). Biodiesel production from jatropha oil by catalytic and non-catalytic approaches: an overview. *Bioresour. Technol. 102* (2), 452–460. doi: 10.1016/j.biortech.2010.09.093.

[12] Olatundun, E.A., Borokini, O.O. and Betiku, E. (2020). Cocoa pod husk-plantain peel blend as a novel green heterogeneous catalyst for renewable and sustainable honne oil biodiesel synthesis: a case of biowastes-to-wealth. *Renew. Energy 166*, 163–175. doi: 10.1016/j.renene.2020.11.131.

[13] Baskar, G., Gurugulladevi, A., Nishanthini, T., Aiswarya, R. and Tamilarasan, K. (2017). Optimization and kinetics of biodiesel production from Mahua oil using manganese doped zinc oxide nanocatalyst. *Renew Energ. 103*: 641-6.

[14] Dai, Y. M., Wang, Y. F. and Chen C.C. (2018). Synthesis and characterization of magnetic LiFe5O8-LiFeO2as a solid basic catalyst for biodiesel production. *Catal Commun 106*: pp.20-4.

[15] Roschat, W., Phewphong, S., Thangthong, A., Moonsin, P., Yoosuk, B., Kaewpuang, T. and Promarak, V. (2018). Catalytic performance enhancement of CaO by hydration-dehydration process for biodiesel production at room temperature. *Energ Convers Manage 165*:1-7.

[16] Ishaya, Z. D., Pam, G.Y., Kulla, D. M., Jumare, A. I. (2020). "Fatty Acid Alkyl Esters Profiles of African Mahogany Biodiesel Produced at 0.25% NaOH Catalyst Concentration and Varied Molar Ratios". *African Journal of Renewable and Alternative Energy (AJRAE). 2020. Vol. 5 No. 1: 32 – 40*. (ISSN: 2006 0394). June 2020. A publication of Renewable and Alternative Energy Society of Nigeria (RAESON).

[17] Muthukumaran, C., Praniesh, R., Navamani, P., Swathi, R., Sharmila, G., and Kumar, N. M. (2017). Process optimization and kinetic modeling of biodiesel production using non-edible Madhuca indica oil. *Fuel 195*:217-25.

[18] Ogwuche C. E., Edema M. O. (2020). *GC-MS and FTIR characterization of essential oil from the fresh leaves of Pandanus candalabrum obtained from Bayelsa state, Nigeria*. Nigerian Journal of Chemical Research *25*(1).

[19] Odo, F., Ezeanyika, U. S., Ogugua, N., Joshua, E., & Okagu, U. (2017). *FTIR and GC-MS Spectroscopic Analysis of Methanol and Chloroform Extracts of Brenania brieyi Root Bark*. *5*(2013), 44–54.

[20] Kavipriya, K., Chandran, M., & Estuningtyas, A. R. I. (2018). *FTIR and GCMS Analysis of Bioactive Phytocompounds in Methonalic Leaf Extract of Cassia Alata Are Mangiferin and Mangiferin-Containing Plant Extracts Helpful for Iron- Loaded Transfusion-Dependent and Non-Transfusion-Dependent Thalassaemia Patients ?11*(1), 141–147.

[21] Bolade O. P., Akinsiku A. A., Adeyemi A. O. Jolayemi G. E., Williams A. B., Benson N. U. (2019). *Qualitative analysis, total phenolic content, FT-IR and GC-MS characterisation of Canna indica : bioreducing agent for nanoparticles synthesis Qualitative analysis , total phenolic content , FT-IR and GC-MS characterisation of Canna indica : bioreducing agent for nanoparticles synthesis*. https://doi.org/10.1088/1742-6596/1299/1/012135.

[22] Tulashie, S. K., Kalita, P., Kotoka, F., Segbefia, O. K., & Quarshie, L. (2018). Biodiesel production from Shea Butter: a suitable alternative fuel to premix fuel. *Materialia*. https://doi.org/10.1016/j.mtla.2018.08.038.

[23] Cheah, M. Y., Ong, H. C., Zulkifli, N. W. M., Masjuki, H. H., & Salleh, A. (2020). Physicochemical and tribological properties of microalgae oil as biolubricant for hydrogen-powered engine. *International Journal of Hydrogen Energy*, *45*(42), 22364–22381. https://doi.org/10.1016/j.ijhydene.2019.11.020.

[24] Mohammed, I. A., Sadiq, M. M., Aberuagba, F., Olurinde, A. O., & Obamina, R. (2015). *SYNTHESIS AND CHARACTERIZATION OF TRIMETHYLOLPROPANE-BASED Proceedings of the 45 th Annual Conference of NSChE , 5 th Nov – 7 th Nov ., 2015 , Warri , Nigeria P 024 Synthesis and Characterization of Trimethylolpropane-Based Biolubricants from Castor Oil D. November.*

[25] Alhassan, A. J., Muhammad, I. U., Sule, M. S., Wudil, A. M., & Imam, A. A. (2017). *Characterization and Anti-diabetic Activity of Dihydrophenantherene Isolated from Khaya senegalensis Stem Bark*. *17*(2), 1–17. https://doi.org/10.9734/ARRB/2017/35908.

[26] Dass P. M., Louis H., Alheri A., Amos P. I., Bifam M., Ago M. A. (2018). *Production of Biodiesel Oil from Desert Dates (Balanites aegyptiaca) Seeds Oil Using a Hetrogeneous Catalyst Produced from Mahogany (Khaya senegalensis) Fruit Shells*. Analytical Chemistry: An indian Journal *18*(1), 1–12.