



ORIGINAL RESEARCH ARTICLE

SYNCHRONIZED ANALYSIS OF BIODIESEL FROM KHAYA SENEGALENSIS OILS
USING FTIR, GC-MS AND NMR AT TOP PARAMETRIC CONDITIONS.

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ARTICLE INFORMATION

Received: 24th April 2025

Revised: 19th May 2025

Accepted: 20th May 2025

Keywords:

Khaya senegalensis

Fatty methyl esters

FTIR

GC-MS

NMR

Functional groups

ABSTRACT

Alternatives to costly fossil fuel have witnessed rekindled interest because of the depletion of fossil reserves and environmental concerns. In this study, non-edible vegetable oil from Khaya Senegalensis (KS), which grows well in semi-arid zones was converted to biodiesel via transesterification under the reaction condition of 1.25 % NaOH catalyst and 1:6 Oil-methanol molar ratio. FTIR, GC-MS and NMR techniques were concurrently used to monitor common or similar peaks in the conversion process in an extensive cross comparison to validate other techniques' results. Furthermore, the composition of conventional diesel with the vegetable biodiesel produced were simultaneously analyzed. The results showed that conversion peaks were aligned, and successful delivered 82.02 % yield. The results from FTIR, GC-MS and NMR investigations all agreed, on the conversion of triglyceride molecules in the crude KS oil to fatty methyl esters in the biodiesel. Both produced biodiesel and conventional diesel were observed to contain alkanes and alkenes functional groups, that impart good fuel properties.

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1.0 Introduction

Fossil liquid fuel in the form of expensive gasoline and diesel, is mostly utilized in the transport and electrical power sectors to power internal combustion engines. The exhaust products from these engines are not problem-free. The exhaust emissions comes along with environmental pollutants that contaminate the air, also the emissions are responsible for climate change and coronary disease (Ishaya et al., 2020a). Carbon (IV) oxide (CO₂) concentration in the atmosphere has reached alarming levels due to the production and consumption of fossil fuels. According to Banik et al., (2018), 86 % of the world energy consumption, 20 % of CO₂ emissions and almost all fuel needed in the transport sector are from fossil sources. With the increasing trends of human population and global demand for motorized mobility, there shall be increased fossil fuel consumption, pollutants emission and environmental sustainability challenges. Biodiesel is a fuel obtained from plant oil, and it is characteristically similar to petrol-diesel, but it is nontoxic, renewable, sustainable, and environmentally harmless (Olagbende et al., 2021). When biodiesel is used in diesel engines, it presents excellent combustion properties due to its low sulfur, low aromatic content and high cetane number (Chaudhary et al., 2018). Non-edible oil from cheap tropical feedstock such as Khaya senegalensis (KS) that are easily cultivated in arid or semi-arid conditions, are used as alternatives and in the sustainable production of biodiesel (Sukkasi et al., 2010 and Dai et al., 2014).

Some studies in the area of biodiesel production include: Alahmar et al., (2025) that investigated the efficient way of producing biodiesel from waste cooking oil by the use of a bifunctional catalyst. Similarly, Rao et al., (2025) studied the synthesis and optimization of biodiesel production from Trichosanthes cucumerina seed using Prosopis juliflora catalyst and reported the optimized conditions as 60 mg of biomass at 70 °C for a reaction time of 60 min to yield 46.31% catalyst, that was used in transesterification reaction to yield 97.42% biodiesel. Additionally, Farouk et al., (2024) worked on the optimized production of biodiesel from waste cooking oil using nano-catalyst and reported an optimized conditions of 7:1 methanol to oil ratio, 50 °C reaction temperature and 60 minutes reaction time using response surface methodology. Weldeslase et al.,

(2023) investigated the optimized production of biodiesel from waste cooking oil using a CaO catalyst and reported 96.5% biodiesel conversion at 14:1 methanol to oil molar ratio, 57.5 °C reaction temperature, 5% wt catalyst loading and 2 hours reaction time. Zubairu et al., (2021) produced synthesized biodiesels and hybrid oil blends from *Jatropha curcas* and *Thevetia peruviana* seeds oil, and reported high biodiesel yield of 90.0%, 86.1%, 90.7%, 92.2% and 88.2% for five hybrid blends at 60 °C reaction temperature and 1 hour reaction time with 1% (w/w) KOH catalyst. Kaisan et al., (2018) Carried out a comparison of *Jatropha* and *Neem* seed biodiesel and reported that *Jatropha* biodiesel contained 91.9 % methyl ester while *Neem* biodiesel contained 70.2 %. Nahadi & Atadashi, (2018) Studied the effects of variation of catalyst concentration on the production of biodiesel from coconut oil. The results showed that 96 % biodiesel was obtained at 1 % w/w catalyst concentration, 1:6 oil to methanol ratio, 55 °C reaction temperature and 60 minutes reaction time. Furthermore, Ofoegbu & Kelle, (2013) determined the edibility of *Thevetia peruviana* seed oil using GC-MS, FTIR and UV-VIS techniques. The results showed that the oil has nutritional benefits provided the traces of cyanide are removed.

The study of the biodiesel production process using solid acid catalyst was investigated by Gebremariam & Marchetti, (2021), the results showed that variation in oil cost and the price of biodiesel strongly affects the viability of the production process. Similarly, Alhassan et al., (2017) characterized *Khaya senegalensis* stem bark, the 1H NMR results indicated a carbon-carbon double bond, aromatic protons, and protons are attached directly to electronegative atoms. Additionally, Bolale et al., (2019) carried out FTIR and GC-MS analyses on *Canna indica*, the FTIR results showed the presence of aromatic $O-H$ stretch (3300 cm^{-1}) and aromatic $C=C$ stretch (1451 and 1640 cm^{-1}), and the GC-MS result showed the presence of di-alpha-tocopherol. Also, the chemical modification of *Camelina* oil was investigated by Sharma et al., (2020), its 1H NMR spectra showed CH- protons and secondary alcohols at 2.9 – 3.2 and 4.25 – 3.35 ppm, respectively. A research was conducted by Odo et al., (2017) using FTIR and GC-MS analyses on the *Brenania brieyi* root extract, and reported the presence of pentadecanoic acid (17.04 %), 9, 12-hexadecanoic acid (10.18 %), 9-Octadecanoic acid (60.53 %) in the methanol extract, and hexadecanoic acid (11.29 %), 9-Octadecanoic acid (54.85 %), 9, 12-Octadecanoic (3.90 %) in the chloroform extracts. Similar studies by Hossain et al., (2021) investigated triglyceride conversion of waste frying oil, and reported that the 1H NMR showed glycerine peaks, $\alpha-CH_2$ protons and a 98.48 % biodiesel conversion.

For this study, the aim is to convert *Khaya senegalensis* crude oil to biodiesel at 1.25 wt. % NaOH catalyst loading and 1:6 alcohol to methanol molar ratio. Other researchers have considered the alcohol-to-methanol ratio between 1:6 to 1:12 for different feedstock and catalyst loading between 0.5 to 1.0 wt%. The *Khaya senegalensis* feedstock was investigated in the present study because it is non-edible, it is commonly available within the semi-arid region of northern Nigeria, and no previous studies were conducted on the feedstock under this defined conditions. The FTIR, GC-MS, and 1H NMR techniques used in the present study is to indicate the justifications for the conversion of triglyceride molecules to fatty acid methyl esters (biodiesel).

2. Materials and Methods

The *Khaya senegalensis* winged seed kernels were collected from the trees in Samaru-Zaria in Nigeria and the oil was extracted using mechanical expeller press. The reagents used for the conversion of crude oil to biodiesel were of analytical grade (Assay 99.5%). The reagents details are outlined as follows: Sodium Hydroxide (NaOH) pellets, Propan-2-ol (CH_3CH_2OH), Sulphuric acid (H_2SO_4), 0.1M Potassium Hydroxide (KOH) and Methanol (CH_3OH). The first stage was to pretreat the crude oil using propan-2-ol, then followed by the determination of free fatty acid and acid value. The crude oil was titrated with 0.1M of KOH until the acid value reduces to $\leq 0.5\%$. The second stage transesterification reaction to convert the crude oil to biodiesel was carried out at an oil-to-alcohol molar ratio of 1:6 using NaOH catalyst for 1 hour at 60 °C. The details of the procedure are contained in an earlier study by Ishaya et al., (2020a).

2.1 Fourier Transform Infrared (FTIR) Spectroscopy

Agilent technologies FTIR Spectrophotometer (54VA Agilent technologies Cary 630 FTIR Spectrophotometer Part G8043-64002, Serial No. MY14470031, made in Malaysia) was used to take the infrared spectrum of samples. The potassium bromide disc method was used to obtain the IR spectra of samples. 650 to 4000 cm^{-1} scanning range was used in this study, functional groups were identified in the crude oil, biodiesel and diesel samples using the FTIR spectra.

2.2 Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS-QO2010 Shimadzu Japan was used in the analysis. A fused silica column was filled with Elite-5MS (95 % dimethylpolysiloxane 5 % biphenyl, $30\text{ m} \times 0.25\text{ mm ID} \times 250\text{ }\mu\text{m df}$). Helium as carrier gas, flow constantly at 1 ml/min , was used to separate the components. The temperature of the injector was set at $250\text{ }^{\circ}\text{C}$ for the chromatographic run. The extract ($1\text{ }\mu\text{l}$) was injected into the instrument, and the temperature was at $70\text{ }^{\circ}\text{C}$ (0 min), followed by an increase to $280\text{ }^{\circ}\text{C}$ at the rate of $10\text{ }^{\circ}\text{C min}^{-1}$, subsequently the temperature was kept at $280\text{ }^{\circ}\text{C}$ for 5 min . The mass detector conditions were set: the transfer line was at $250\text{ }^{\circ}\text{C}$; ion source temperature was at $200\text{ }^{\circ}\text{C}$; and ionization mode electron impact was at 70 eV . Also, the mass detector scan time of 0.5 sec was set, at scan interval of 0.1 sec . In this study, chemical compounds were detected in the crude oil, biodiesel and their respective quantities as reported by the GC-MS spectrometer. The National Institute Standard and Technology (NIST) library was used as a reference to compare and interpret the GC-MS results. Compounds were identified according to molecular mass, structure and fragments calculated. The unknown components spectrum was compared with that of the NIST library, and the closest match was taken.

2.3 NMR Spectroscopy

All the ^1H -NMR spectra were recorded with an Agilent Technologies 400/S4/PC + spectrometer (850 Watts Agilent technologies UK Ltd, 400MHz Nuclear Magnetic Resonance VNMRJ Premium Compact+ AR, Part G8504-64002, Serial No. MRY0023179, made in England) operating at 2.38 bar Helium vessel and 2.5 bar Nitrogen vessel. The reference standard was tetramethyl-silane (TMS), and chemical shifts were reported relative to it. All experiments were conducted at room temperature.

3. Results and Discussion

3.1 Crude Khaya Senegalesis oil FTIR

From Figure 1 and Table 1, the $=\text{C}-\text{H}$ bond was observed at 3004 cm^{-1} , $\text{C}-\text{H}$ alkyl symmetric and asymmetric strong stretches at $2855-2922\text{ cm}^{-1}$, $\text{C}=\text{O}$ very strong stretch at 1744 cm^{-1} , $\text{C}-\text{H}$ terminal alkane bond at 1461 cm^{-1} and $\text{C}-\text{O}-\text{C}$. Figure 1 indicated that the alkanes, alkenes, carboxylic and esters functional groups were present in Khaya Senegalensis crude oil. As reported by Hellier *et al.*, (2018) and Karmakar *et al.*, (2021), the fatty acids attached to the glycerol backbone are usually long chains containing 14 – 22 carbon atoms. Some of the attached carbon atoms are either methyl, methylene, carbonyl, carboxylic or esters in structure. This however, is consistent with the findings by Hellier *et al.*, (2018) and Karmakar *et al.*, (2021) because it shows the presence of a triglyceride molecule with alkane carbon atoms, alkene carbon atoms, carboxylic carbon atoms and esters carbon atoms.

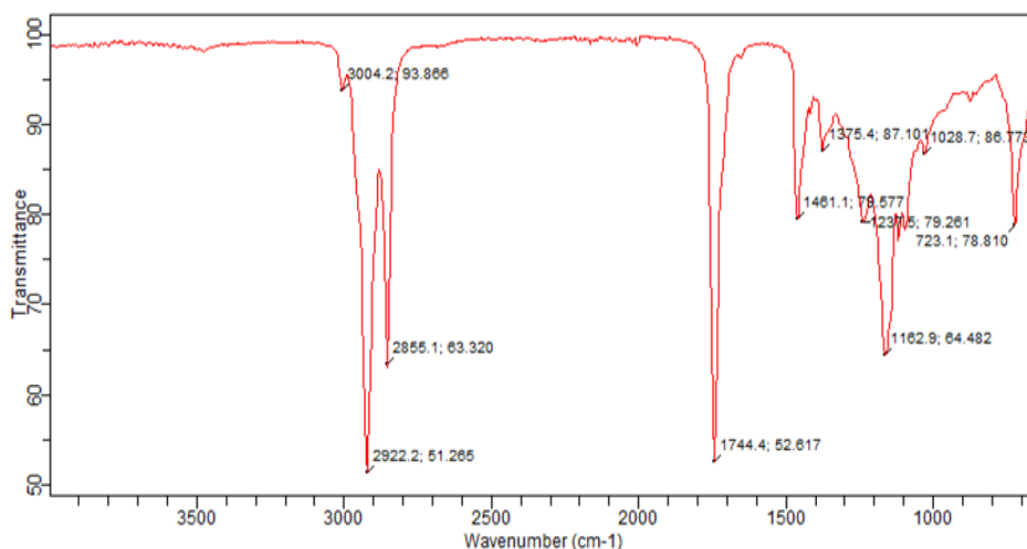


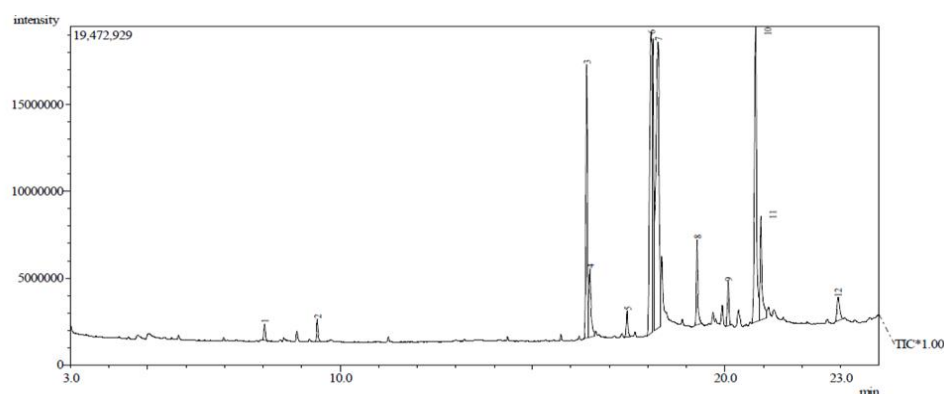
Figure 1: FTIR spectra of crude Khaya senegalesis oil.

Table 1: Functional groups in crude *Khaya senegalensis* oil.

S/N	Wavelength (cm^{-1})	Functional group
1	3004	$\text{C} - \text{H}$ weak stretch (alkene)
2	2922, 2855	$\text{C} - \text{H}$ strong stretch (alkanes)
3	1744	$\text{C} = \text{O}$ strong stretch (alkanoic & ester)
4	1461	$\text{C} - \text{H}$ medium stretch (terminal alkane bend)
5	1375	$\text{C} - \text{O}$ weak stretch (alkanoic)
6	1162	$\text{C} - \text{O}$ medium stretch, (esters)
7	1028	$\text{C} - \text{O} - \text{C}$ weak stretch (alkanoate)
8	723	$\text{C} = \text{C} - \text{H}$ medium stretch (alkene bending)

This study results from Table 1 conforms with Cheah *et al.*, (2020), which observed $\text{C} - \text{O}$ stretching at 1059 cm^{-1} , alkane $\text{C} - \text{H}$ stretching at 2900 cm^{-1} , ester $\text{C} = \text{O}$ stretching at 1700 cm^{-1} , and alkene $\text{C} = \text{C}$ stretching at 1650 cm^{-1} . Also, the study is consistent with (Tulashie *et al.*, 2018), who observed $-\text{CH}_2$ stretching vibrations at 2853.09 and 2922.27 cm^{-1} and $-\text{C} = \text{O}$ bond stretching at 1741.91 cm^{-1} .

3.2 Crude *Khaya Senegalesis* oil GC-MS

**Figure 2:** GC-MS spectra of crude *Khaya senegalesis* oil.**Table 2:** Compounds present in crude *Khaya senegalesis*.

PK	RT	Area %	Library/ID	Formula
1	15.713	1.41	Methyl 6-methyl heptanoate	$\text{C}_9\text{H}_{18}\text{O}_2$
2	16.357	1.98	Hexadecanoic acid, ethyl ester	$\text{C}_{18}\text{H}_{36}\text{O}_2$
3	16.440	9.05	Hexadecanoic acid	$\text{C}_{16}\text{H}_{32}\text{O}_2$
4	17.427	6.45	11-Octadecenoic acid, methyl ester	$\text{C}_{19}\text{H}_{36}\text{O}_2$
5	17.628	1.23	Methyl 8-hydroxyoctanoate	$\text{C}_9\text{H}_{18}\text{O}_3$
6	18.013	14.79	9-Octadecenoic acid, ethyl ester	$\text{C}_{20}\text{H}_{38}\text{O}_2$
7	18.170	43.81	Oleic acid	$\text{C}_{18}\text{H}_{34}\text{O}_2$
8	18.304	11.54	Nonadecanoic acid	$\text{C}_{19}\text{H}_{38}\text{O}_2$
9	19.665	6.60	cis-Oleic acid	$\text{C}_{18}\text{H}_{34}\text{O}_2$
10	19.665	3.15	10-Undecenal	$\text{C}_{11}\text{H}_{20}\text{O}$

From Figure and Table 2; the GC-MS analysis of crude *Khaya Senegalensis* oil showed the presence of 10 compounds. Major compounds include: Oleic acid $\text{C}_{18}\text{H}_{34}\text{O}_2$ at retention time of 18.170 min occupying the largest mass spectra area of 43.81 %, 9-Octadecenoic acid, ethyl ester ($\text{C}_{20}\text{H}_{38}\text{O}_2$) at retention time 18.013 min with a mass spectra area of 14.79 %, Nonadecanoic acid ($\text{C}_{19}\text{H}_{38}\text{O}_2$) at retention time 18.304 min with a mass spectra area 11.54 %, Hexadecanoic acid ($\text{C}_{16}\text{H}_{32}\text{O}_2$) at retention time 16.440 min occupying 9.05 % mass spectra area, 11-Octadecenoic acid, methyl ester $\text{C}_{19}\text{H}_{36}\text{O}_2$ at retention time 17.427 min with a mass spectra area of 6.45 %, cis-Oleic acid ($\text{C}_{18}\text{H}_{34}\text{O}_2$) at retention time 19.665 min with a mass spectra area 6.60 %, 10-Undecenal ($\text{C}_{11}\text{H}_{20}\text{O}$) at retention time 20.727 min occupying 3.15 % mass spectra area.

The GC-MS result showed that triglycerides with oleic fatty acid represent a larger portion of the KS vegetable oil. Oleic acid is a monounsaturated fatty acid with 18-carbon atoms. This GC-MS result conforms with the FTIR result in Figure 1, indicating similar compounds as discussed. The 18-carbon atoms in the KS triglyceride

molecule connotes a good parameter for biodiesel production because it will contribute to a higher heating value of the fuel and also a short ignition delay. Similarly, the un-saturation in the KS triglyceride molecule will enhance the fluidity of the fuel when the biodiesel is finally produced.

3.3 KS Crude oil NMR

Figure 3 show the chemical shift of the signals shown in Table 3.

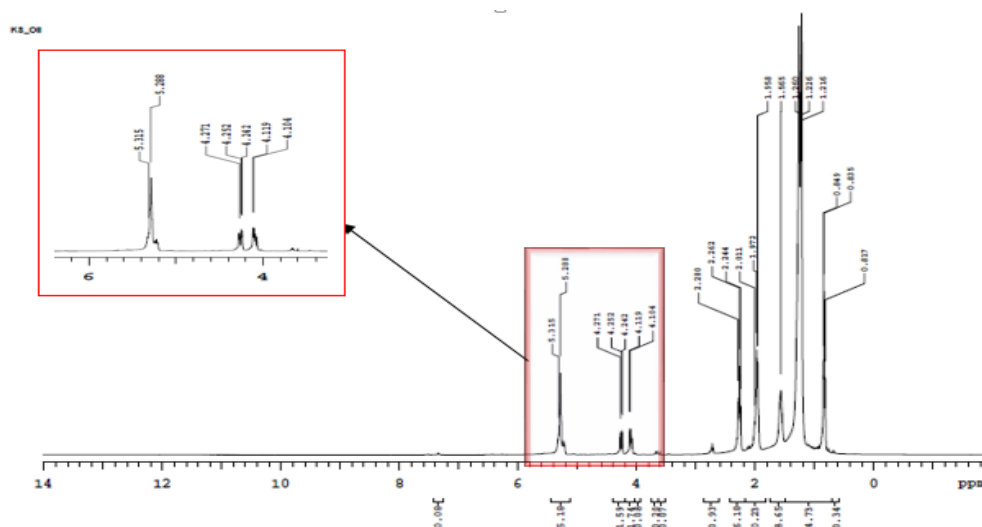


Figure 3: ^1H -NMR spectrum of crude KS oil

Table 3: Chemical shift of main resonances in KS.

S/N	Chemical Shift (ppm)	Multiplicity Nature of carbon
1	5.31 – 5.28	$\text{HC} = \text{CH}$
2	4.11 – 4.27	$-\text{CH}_2 \text{OCOR}$
3	2.26	$\alpha - \text{CH}_2$
4	1.22 – 1.97	$(\text{CH}_2)_n$
5	0.83	CH_3

The result shows that the duplets with peaks at 5.31 and 5.28 ppm correspond to protons attached to carbon-carbon double bonds in the KS triglyceride molecule. This is in agreement with the GC-MS result in Figure 2 and FTIR result in Figure 1 of KS crude oil. All results agree on the presence of an alkene functional group, and this is consistent with Fu *et al.*, (2021). Furthermore, the result indicates a triplet and a duplet centered at 4.25 and 4.12 ppm. This is mostly assigned to protons attached to the triglyceride backbone ($\text{CH}_2 - \text{COOR}$) in the sn 1' 3' position. This finding conforms with (Khosa, 2021), which shows a triglyceride as one molecule of glycerol attached to three molecules of fatty acid. This NMR observation is consistent with Truzzi *et al.*, (2021), and it also agrees with the GC-MS and FTIR result on the presence of carboxylic and ester functional groups. Figure 3 also shows a triplet at 2.26 ppm that corresponds to protons attached to α -carbon and $(\text{CH}_2)_n$ Protons in the region of 1.22 – 1.97 ppm. This shows the existence of alkanes in the triglyceride molecule and is consistent with (Sharma *et al.*, 2020). Protons of CH_3 are also observed at 0.83 ppm, which is in tandem with Fu *et al.*, (2021).

Based on the ^1H -NMR result in Figure 3, the study observed that: alkanes, alkenes, carbonyl and esters chemical environments are present in *Khaya senegalensis* crude oil. This result, together with the GC-MS and FTIR results, further confirms the triglyceride molecule with oleic acid as the major fatty acid in the KS vegetable oil.

3.4 FTIR of KS Biodiesel at 1.25 % Catalyst

From Figure 4 as interpreted in Table 4, the $=\text{C} - \text{H}$ bond was observed at 3004 cm^{-1} , $\text{C} - \text{H}$ alkyl symmetric and asymmetric strong stretches at $2922 - 2855 \text{ cm}^{-1}$, $\text{C} = \text{O}$ very strong stretch at 1740 cm^{-1} , $\text{C} - \text{H}$ terminal alkane bond at $1461, 1435 \text{ cm}^{-1}$ and $\text{C} - \text{O} - \text{C}$ at 1095 cm^{-1} . The study also identified the $\text{C} - \text{O}$ ester group at $1190, 1170 \text{ cm}^{-1}$, the $\text{C} - \text{O}$ carboxylic group at 1380 cm^{-1} , and the $\text{C} - \text{H}$ bend at 723 cm^{-1} .

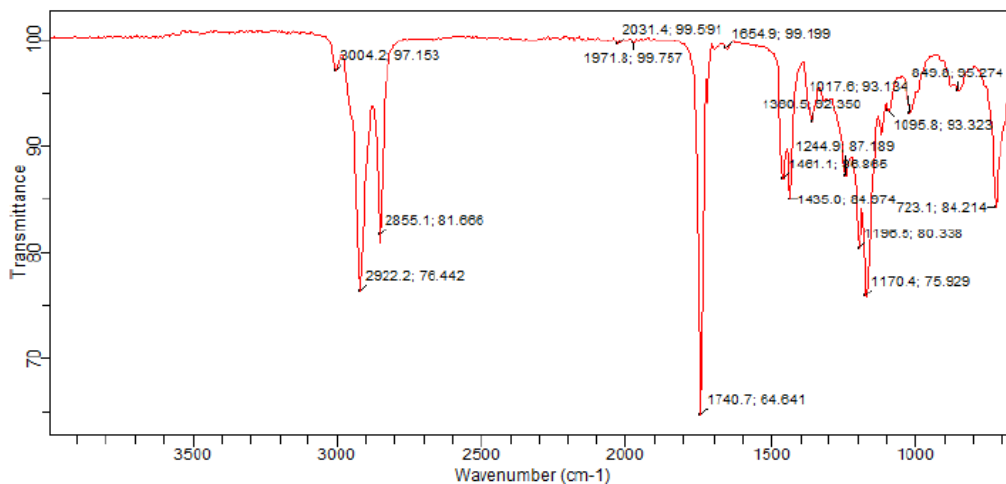


Figure 4: FTIR spectra of Khaya senegalensis biodiesel

Table 4: Functional groups in Khaya senegalensis biodiesel.

S/N	Wavelength (cm^{-1})	Functional group
1	3004	$\text{C} - \text{H}$ weak stretch (alkene)
2	2922, 2855	$\text{C} - \text{H}$ strong stretch (alkanes)
3	1740	$\text{C} = \text{O}$ strong stretch (alkanoic & ester)
4	1461, 1435	$\text{C} - \text{H}$ medium stretch (terminal alkane bend)
5	1380	$\text{C} - \text{O}$ weak stretch (alkanoic)
6	1190, 1170	$\text{C} - \text{O}$ medium stretch, (esters)
7	1095	$\text{C} - \text{O} - \text{C}$ weak stretch (alkanoate)
8	723	$\text{C} = \text{C} - \text{H}$ medium stretch (alkene bending)

Comparing Figure 4 with Figure 1 and Table 4 with Table I, the following observations were made: the alkanes and alkenes were unaltered during the conversion since peaks at 3004 cm^{-1} and $2922 - 2855 \text{ cm}^{-1}$, 1461 cm^{-1} and 723 cm^{-1} were observed in both crude oil and biodiesel of KS. However, the position of the $\text{C} = \text{O}$ shifted from 1744 cm^{-1} in the crude oil to 1740 cm^{-1} in the biodiesel, indicating some sort of transformation. From this, it can be said that the carboxylic functional group in the crude oil has been converted to the ester group during the transesterification reaction. Similarly, the occurrence of $1190, 1170 \text{ cm}^{-1}$ peaks in the biodiesel due to the $\text{C} - \text{O}$ bond indicates a formation of esters, since natural esters occurred at 1062 cm^{-1} in the crude KS oil. Based on this result, it is deduced that triglyceride molecules have been converted to fatty acid methyl esters (biodiesel).

This study is consistent with Dass *et al.*, (2018), who observed $\text{C} = \text{O}$, $\text{C} = \text{C}$, $\text{C} - \text{O}$, $\text{C} - \text{C}$ stretch and $\text{C} - \text{H}$ bending at $1744.4, 1461.1, 1237.6, 1162.9$ and 723.1 cm^{-1} , respectively, in biodiesel produced from a Mahogany fruit shell. In the same way Alhassan *et al.*, (2017) attributed $1709, 1248$ and 1611 cm^{-1} to $\text{C} = \text{O}$, $\text{C} - \text{O}$ and $\text{C} = \text{C}$ groups, respectively, that the present study is in agreement with.

3.5 GC-MS of KS Biodiesel at 1.25 % Catalyst

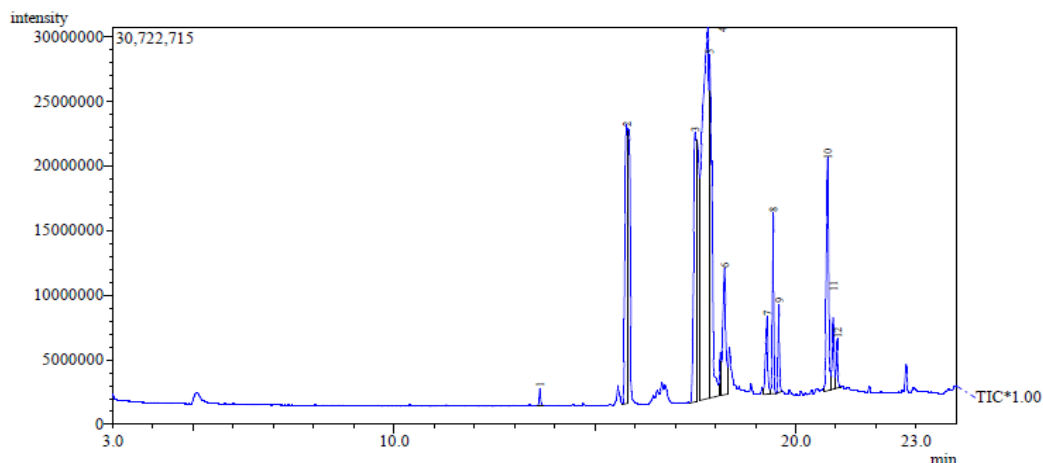


Figure 5: GC-MS spectra of Khaya senegalensis biodiesel at 1.25 % cat. Conc.

Table 5: Compounds present in Khaya senegalensis biodiesel at 1.25 % cat. Conc.

PK	RT	Area %	Library/ID	Formula
1	13.630	0.34	Tridecanoic acid, methyl ester	$C_{14}H_{28}O_2$
2	15.779	10.20	Palmitic acid, methyl ester	$C_{17}H_{34}O_2$
3	17.494	12.22	11-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$
4	17.815	38.13	9-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$
5	17.856	14.09	5-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$
6	18.230	5.81	Oleic acid	$C_{18}H_{34}O_2$
7	19.288	2.79	Octadecyl vinyl ether	$C_{20}H_{40}O$
8	19.442	3.96	Eicosanoic acid, methyl ester	$C_{21}H_{42}O_2$
9	19.583	1.98	Tetradecanoic acid methyl ester	$C_{16}H_{32}O_2$
10	20.796	7.51	9-Octadecenal	$C_{18}H_{32}O$
11	20.933	1.85	2-Butyloctanol	$C_{12}H_{26}O$
12	21.030	1.10	Eicosanoic acid, methyl ester	$C_{21}H_{42}O_2$

The GC-MS analysis of the Khaya senegalensis biodiesel showed the presence of 12 compounds. Major compounds include: 9-Octadecenoic acid, methyl ester at retention time of 17.815 *min* occupying the largest area of 38.13 %, 5-Octadecenoic acid, methyl ester at retention time 17.856 *min* with area of 14.09 %, 11-Octadecenoic acid, methyl ester at retention time 17.494 *min* with area 12.22 %, Palmitic acid, methyl ester at retention time 15.779 *min* occupying 10.20 % area, (-Octadecenal at retention time 20.796 *min* with area of 7.51 %, Oleic acid at retention time 18.230 *min* with area 5.81 %, Eicosanoic acid, methyl ester at retention time 19.442 *min* occupying 3.96 % area.

The percentage area shows the amount of each compound present in the biodiesel as a percentage. The higher the area it occupies the larger the compound percentage in the mixture. For a biodiesel it is expected that methyl ester occupies a larger area. From the result the total methyl esters accounted for 82.02%. The effect of methyl esters on biodiesel includes imparting high heating value and short ignition delay in the biodiesel.

Comparing Figure 2 with Figure 5 and Table 2 with Table 5, it was observed that 25.86 % ester in the crude oil has increased to 82.02 % in the biodiesel. This result is in agreement with Figure 4, which shows the conversion of triglyceride molecules to fatty acid methyl esters. The outcome of the present study conforms with Dass *et al.*, (2018), who reported 9-octadecenoic acid (Z), methyl ester, Hexadecanoic acid (Z) methyl ester, 12-octadecenoic acid (Z), methyl ester in biodiesel produced from Mahogany fruit shell.

3.6 NMR of KS Biodiesel

From Figure 6, the chemical shift of the signals is shown in Table 6 of KS biodiesel. As shown from the results, there are no peaks in the range 4.00 – 4.20 ppm, indicating that conversion of triglyceride molecules has taken place. This is further confirmed by the presence of peaks at 3.6 ppm, implying the formation of fatty acid methyl esters. This result further validates the FTIR result of biodiesel in Figure 4 and GCMS result of biodiesel in Figure 5. The NMR result of the present study is consistent with a study as reported elsewhere (Hossain *et al.*, 2021). Similarly, from Figure 6, a peak was observed at 5.3 ppm, corresponding to carbon-carbon double in the biodiesel. Other carbon-carbon single bonds were also identified in the range 2.3 – 0.9 ppm. These results are consistent with that of Fu *et al.*, (2021).

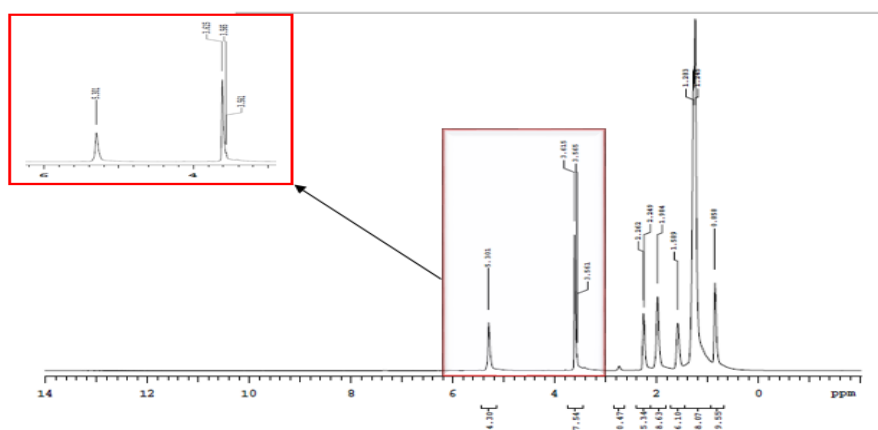
**Figure 6:** ^1H -NMR spectrum of KS biodiesel

Table 6: Chemical shift of main resonances in KS biodiesel.

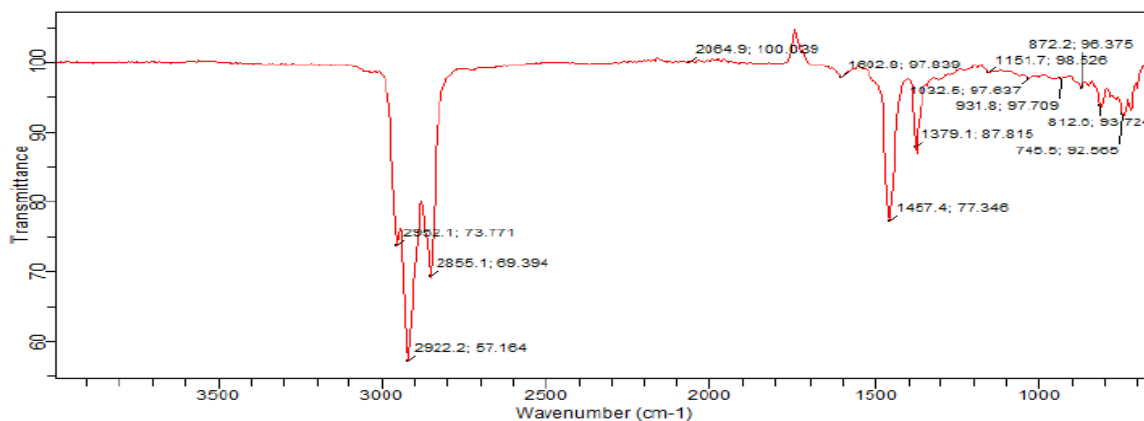
S/N	Chemical Shift (ppm)	Multiplicity	Nature of carbon
1	5.3		$HC = CH$
2	3.6		$H_3C - O$
3	2.3		$\alpha - CH_2$
4	1.9		$H_2C - C = C$
5	1.3 – 1.6		$(CH_2)_n$
6	0.9		CH_3

From Figure 6, the chemical shift of the signals is shown in Table 6 of KS biodiesel. As shown from the results, there are no peaks in the range 4.00 – 4.20 ppm, indicating that conversion of triglyceride molecules has taken place. This is further confirmed by the presence of peaks at 3.6 ppm, implying the formation of fatty acid methyl esters. This result further validates the FTIR result of biodiesel in Figure 4 and GCMS result of biodiesel in Figure 5. The NMR result of the present study is consistent with a study as reported elsewhere (Hossain *et al.*, 2021). Similarly, from Figure 6, a peak was observed at 5.3 ppm, corresponding to carbon-carbon double in the biodiesel. Other carbon-carbon single bonds were also identified in the range 2.3 – 0.9 ppm. These results are consistent with that of Fu *et al.*, (2021).

Based on the 1H -NMR result in Figure 6, the present study further observed: alkanes, alkenes, carbonyl and esters chemical environments present in *Khaya senegalensis* biodiesel. The 1H -NMR results in Figure 6 are consistent with the FTIR result of biodiesel in Figure 4 and GC-MS result of biodiesel in Figure 5.

3.7 Conventional Diesel FTIR

From Figure 7 and Table 7 of FTIR of the conventional or reference diesel, $C - H_2$ and $C - H_3$ asymmetric and symmetric bonds were observed at 2952 cm^{-1} and $2955, 2855\text{ cm}^{-1}$, respectively, $C = C$ at 1602 cm^{-1} , $C - H_2/C - H_3$ alkane bend at $1379 - 1457\text{ cm}^{-1}$, and $= C - H$ bending at $723 - 850\text{ cm}^{-1}$. The $C - H_2$ and $C - H_3$ asymmetric and symmetric bond observed in this study is consistent with observations by Qasim *et al.*, (2017), likewise the $C = C$ bond observed in this study is in concords with report by Ismail *et al.*, (2009), and lastly the $C - H_2/C - H_3$ alkane bend and $= C - H$ observed in the conventional diesel agrees with studies by Attia *et al.*, (2020).



3.8 Conventional Diesel NMR

From Figure 8 – the NMR graph of the conventional diesel, of which the correspondent chemical shift of the signals is represented in Table 8. The result has shown; protons in terminal methyl (CH_3) groups were observed between the range 0.71 – 0.9 ppm. Methylene (CH_2)_n groups in the range 1.09 – 2.0 ppm and $\alpha - CH_2$ protons in the range 2.0 – 2.8 ppm. The chemical shift of the terminal methyl group is consistent with the report by Vukovi et al., (2018), and that of methylene and $\alpha - CH_2$ groups are in accordance with findings by Sharma et al., (2020).

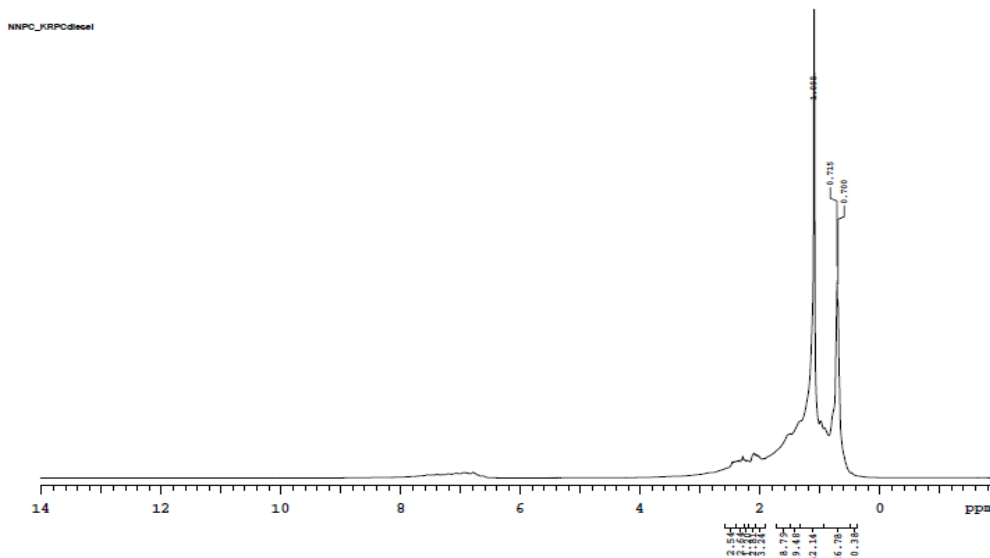


Figure 8: 1H -NMR spectrum of conventional diesel.

Table 8: Chemical shift of main resonances in conventional diesel.

S/N	Chemical Shift (ppm)	Multiplicity Nature of carbon
1	0.71 – 0.9	CH_3
2	1.09 – 2.0	$(CH_2)_n$
3	2.0 – 2.8	$\alpha - CH_2$

Based on the 1H -NMR result in Figure 8, the study observed that alkanes and alkenes chemical environments are present in the conventional diesel. The chemical environments identified by the 1H -NMR in Figure 8 are in agreement with the FTIR result of conventional diesel in Figure 7 that identifies alkane and alkenes functional groups. Comparing the produced biodiesel and conventional diesel, the FTIRs in Figures 1 and 4 both agree that both fuels contain alkanes and alkenes functional groups. The presence of alkanes contributes to the combustion efficiency and high cetane number of the fuels. Alkenes, on the other hand, contribute improved flow properties to the fuels, and this is in conformity to Ishaya et al., (2022). However, the 1H -NMR spectrum of KS biodiesel differs from that of conventional diesel in that it contains fatty acid methyl esters. These esters are long-chain hydrocarbons (between 12 – 22 carbon atoms). The existence of these long chains will impart high heating value and short ignition delay in the biodiesel.

Comparing earlier studies under the same experimental conditions by Ishaya et al., (2020a) at 0.50 % catalyst concentration, and Ishaya et al., (2020b) at 1.00 % catalyst concentration on the same feedstock, with the present study at 1.25 % catalyst concentration; the biodiesel yield are 55.99 %, 71.73 % and 82.02 %. The result shows that as the concentration of catalyst is increased, there is a corresponding increase in biodiesel yield (Zubairu et al., 2021). This trend is attributed to increased catalytic activity that results in lowering of the activation energy, thereby favoring biodiesel yield. This observation is consistent with studies by Ishaya et al., (2020a), Ishaya et al., (2020b).

4. Conclusion

In this study, triglyceride conversion in KS crude vegetable oil to fatty acid methyl esters at 1.25 % NaOH catalyst concentration and 1: 6 oil-methanol molar ratio. It was observed through the GCMS technique that 25.86 % ester in the crude oil was converted to 82.02 % in the biodiesel via transesterification reaction. The FTIR and 1H -NMR technique used in this study further supports and confirms the conversion of triglyceride molecules in the crude oil to fatty acid methyl esters in the biodiesel. The comparison between the biodiesel

produced and the conventional diesel by FTIR and ¹H-NMR technique shows that both fuels contain alkanes and alkenes functional groups. The present study is consistent with earlier previous studies in that 9-Octadecenoic acid, methyl ester was the major ester formed in the biodiesel. The present study also conforms with earlier studies such that, as catalyst concentration increases, the biodiesel yield increases also: 0.50 % catalyst concentration – 55.99 % biodiesel yield, 1.00 % catalyst concentration – 71.73 % biodiesel yield, and 1.25 % catalyst concentration – 82.02 % biodiesel yield.

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