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^{1}H-NMR and FTIR validation of chemical transformation from Khaya Senegalensis oil to biodiesel and comparison between the biodiesel and Conventional diesel

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Abstract

Low oxidation stability and poor flow are common characteristics of crude vegetable oil and in similar renewable energy sources, that negatively impact on the oil application directly as fuel in diesel engines. Some chemical transformation such as transesterification, epoxidation of the crude oil is carried out, and conversion from crude oil to biodiesel is achieved. To monitor the changes from crude oil to biodiesel, techniques like Fourier Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR) are carried out. This study further the FTIR investigation on an earlier Khaya Senegalensis sample of crude oil and biodiesel by Ishaya et al., (2022), by carrying out NMR investigations. The results showed that both FTIR and NMR agrees on the presence of carboxylic site in the crude oil to support transesterification reaction. ^{1}H-NMR spectrum (CH_3 alkane at 0.9 ppm, α- CH_2 alkane at 2.3 ppm, HC=CH alkene at 5.3 ppm, and H_3C-O ester at 3.6 ppm) *and FTIR spectrum* (C-H alkane at 2922-2855 cm^{-1}, C=C alkene at 1654 cm^{-1}, C-O esters at 1244-1170 cm^{-1}) *of the Khaya Senegalensis biodiesel both agrees on the presence of alkanes, alkenes and esters chemical environments, that suggest chemical transformation had occurred in the triglyceride crude oil* observed by (CH_2OCOR at 4.11-4.27 ppm in ^{1}H-NMR and C=O at 1744 cm^{-1}, C-O-C alkanoate at 1095 cm^{-1} in FTIR). *The ^{1}H-NMR spectrum of conventional diesel suggest presence of short chains as compared to biodiesel that is made up of long chains esters. The ^{1}H-NMR spectrum of Khaya Senegalensis biodiesel also identified esters that support low viscosity, high flash point and more energy per unit volume.*

Nomenclature and units

$H-NMR$	Proton Nuclear Magnetic Resonance
$FTIR$	Fourier Transform Infrared
$GC-MS$	Gas Chromatograph and Mass Spectrometry
ppm	Part Per Million
cm^{-1}	Wavelength

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1.0 Introduction

Fossil fuels are the main energy sources in the globe (Sani et al., 2018). According to IEA reports, global fossil fuel resources will face depletion by 2030. It is necessary to find alternative sources of energy that are sustainable and renewable (Bragadeshwaran et al., 2018). A significant portion of fuel is consumed by transportation, agriculture and power generation sectors. This translates to fast depletion of fossil reserves and pollution of the environment (Kaisan et al., 2017). Due to the close physico-chemical characteristics between biodiesel and diesel fuels, biodiesel are regarded as renewable alternative energy source to fossil diesel fuel (Pm et al., 2018, Kaisan et al., 2018). Biodiesel are majorly esters that are hydrolysis products of triglycerides (TG) and free fatty acids (FFA) in plant or animal oils and fats. An essential part of biodiesel development is to identify and quantify TG and FFA. This is done via various techniques such as Fourier transform infrared spectroscopy (FTIR), gas chromatography mass spectroscopy (GC-MS), high performance liquid chromatography (HPLC) or Nuclear magnetic resonance spectroscopy (NMR) (Enrica et al., 2020). The NMR technique is non- destructive, fast, direct, no need of sample pre-treatment test. It requires small amount of specimen, suitable for specimens sensitive to heat, and it is both qualitative and quantitative technique (Enrica et al., 2020).

Many studies on biodiesel such as Spanou et al. (2024) carried out the comparative studies on biodiesel production from acid oil using immobilized biolipasa. Bibi et al., (2024) worked on using recyclable and highly active nanocatalyst for the green synthesis of biodiesel. Sundarajan & Abdurahman, (2019) used ^{1}H NMR, FTIR, LC-MS and GC-MS to characterized the chemical compounds in Patchouli leaf extract. The results showed alcohol, carbonyl aldehyde, alkane, alkene, and aromatic rings. Hossain et al., (2021) investigated triglyceride conversion of waste frying oil. The ^{1}H NMR showed glycerine peaks, α- CH_2 protons and a 98.48% biodiesel conversion. Das et al. (2023) optimized the process of producing biodiesel using snail shell nanocatalyst. Biodiesel production using vigna mungo heterogenous catalyst from multiple oil mixture was investigated by (Brahma et al., 2024). Zohmingliana et al. (2024) produce biodiesel from Jatropha oil using a metal-organic solid catalyst. Chemical modification of camelina oil was investigated by (Sharma et al., 2020). The ^{1}H-NMR spectra showed CH- proton and secondary alcohols at 2.9-3.2 and 4.25-3.35 ppm respectively. Alhassan et al., (2017) characterized Khaya senegalensis stem bark. The ^{1}H-NMR results indicated carbon-carbon double bond, aromatic protons, and protons attached directly to electronegative atom. qNMR quantification of adulterated vegetable oils was investigated by (Truzzi et al., 2021), the ^{1}H-qNMR determined the adulterant with 91.81% accuracy. (Fu et al., 2021) investigated the physico-chemical and thermal properties of Moringa seed oil. The ^{1}H-NMR spectra show glycerylmethylenes and glycerylmethines protons at 4.14 and 5.27 ppm respectively. Extraction and transesterification of chlorella vulgaris for biodiesel production was investigated by (Adhiputra et al., 2021). The results from ^{1}H-NMR shows triglycerides peaks at 4.1 ppm and methoxy group at 3.66 ppm. (Enrica et al., 2020) carried out determination of free fatty acids present in vegetable oils using NMR. The ^{1}H-NMR showed =CH-, $-CH_2COOH$, and $–CH_3$ in the ranges 5.36-5.37, 2.53-2.36 and 0.89-0.98 ppm for oleic, linoleic and α-linolenic aids respectively. Combustion, performance and emission characteristics of biodiesel fuel blends was investigated by (Qasim et al., 2017). The FTIR of the conventional diesel showed alkanes peaks stretching at 2922 and 2852 cm^{-1} and bending at 1458 and 722 cm^{-1} respectively. (Domingos & Máquina, 2019) carried out NMR analysis of diesel and crambe biodiesel blends. The ^{1}HNMR spectra showed methoxy and olefinic protons at 3.7 and 5.4 ppm respectively. (Vukovi et al., 2018) investigated diesel fuel oxidation stability by ^{1}H NMR spectroscopy. The ^{1}H-NMR spectra showed polyaromatic, monoaromatic and paraffinic protons in the ranges 7.20-9.00, 6.60-7.20, 1.00-2.08 ppm respectively.

This study furthers carried out ^{1}H-NMR spectroscopy characterization on an earlier sample of biodiesel produced from khaya senegalensis oil by (Ishaya et al., 2022) to validate the earlier FTIR results. The study attempts to use FTIR and ^{1}H-NMR technique to compare the chemical composition between the khaya senegalensis (KS) biodiesel and the conventional diesel. The ^{1}H-NMR technique was selected for this characterization and validation study because it is both qualitative and quantitative.

2.0 Materials and Methods

All methods and materials for this study were published in (Ishaya et al., 2022), except for the Proton Nuclear Magnetic Resonance (^{1}H-NMR) Spectroscopy. Refer to the cited article.

2.1 Proton Nuclear Magnetic Resonance (^{1}H-NMR) Spectroscopy

All the ^{1}H-NMR spectra were recorded with Agilent Technologies 400/S4/PC+ spectrometer operating at 2.38 bar Helium vessel and 2.5 Nitrogen vessel. The reference standard was tetramethylsilane (TMS) and chemical shifts were reported relative to it. All experiment were conducted at room temperature.

3.0 Results and Discussions

3.1 FTIR Analysis of Crude Khaya Senegalesis oil

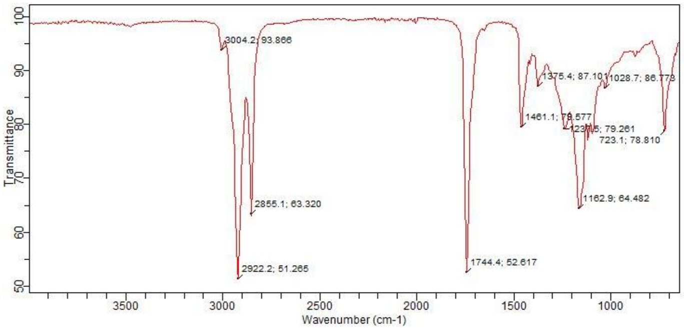

Figure 1: FTIR spectra of crude khaya Senegalensis oil
Source: Ishaya et al. (2022)

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 strongh stretch (alkanes)
3	1744	C=O strongh 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)

Source: Ishaya et al. (2022)

According to the FTIR spectrum of crude KS oil by Ishaya et al., (2022) ; the =C-H bond was observed at 3004 cm^{-1} and is consistent with (Tulashie et al., 2018), C-H bond was observed at 2855-2922 cm^{-1} in coherence with (Mohammed et al., 2015). The following functional groups were also observed; C=O at 1744 cm^{-1}, C-H terminal alkane bond at 1461 cm^{-1} and C-O-C at 1028 cm^{-1} that are consistent with (Cheah et al., 2020). As reported by Ishaya et al., (2022) the study observed: alkanes, alkenes, carboxylic and esters functional groups were present in Khaya Senegalensis crude oil.

3.2 NMR Analysis of Crude Khaya Senegalensis oil

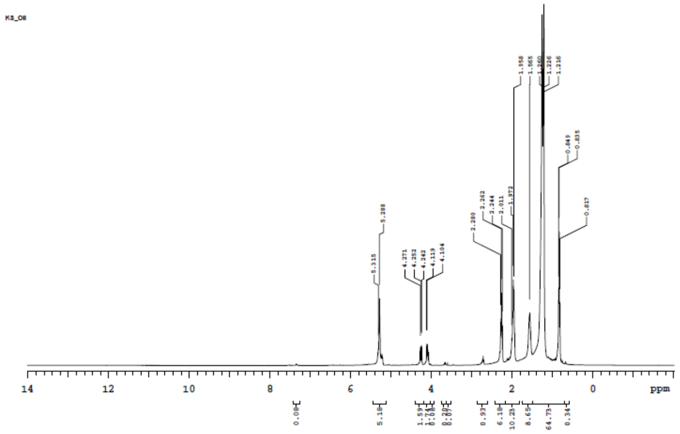

Figure 2: ^{1}H-NMR spectrum of crude Khaya Senegalensis oil.

Table 2: Chemical shift of main resonances in crude Khaya Senegalensis oil

S/N	Chemical Shift (ppm)	Nature of carbon
1	5.31- 5.28	HC=CH
2	4.11-4.27	–CH_2 OCOR
3	2.26	α- CH_2
4	1.22-1.97	$(CH_2)_n$
5	0.83	CH_3

As indicated from the ^{1}H-NMR spectrum of crude KS oil in Table 2; the duplets with peaks at 5.31 and 5.28 ppm corresponds to protons attached to carbon-carbon double bonds in the crude oil. This is consistent with (Fu et al., 2021). Also, the result shows a triplet and duplet centered at 4.25 and 4.12 ppm, this is attributed to protons attached to the triglyceride backbone (CH_2-COOR) in the SN 1' 3' position. This is consistent with (Truzzi et al., 2021). It also shows a triplet at 2.26 ppm that is corresponding to protons attached to α-carbon, and $(CH_2)_n$ protons in the region of 1.22 – 1.97 ppm. This is consistent with (Sharma et al., 2020). Protons of CH_3 are also observed at 0.83 ppm as supported by (Fu et al., 2021).

Based on the ^{1}H-NMR result, the study observed alkanes, alkenes, carbonyl and esters chemical environments present in Khaya Senegalensis crude oil similar to the results established by FTIR in the earlier studies by Ishaya et al. (2022). The ^{1}H-NMR results in figure. 2 shows the presence of glyceride molecules in the sample oil that further validates the study by (Ishaya et al., 2022). Due to the presence of these functional groups; their effect on the production of biodiesel from crude Khaya Senegalensis oil are as follows: The carbonyl group would provide suitable site for transesterification reaction, the esters will provide good fuel properties, the alkanes and alkenes will not change during the process.

3.3 FTIR Analysis of Khaya Senegalensis Biodiesel

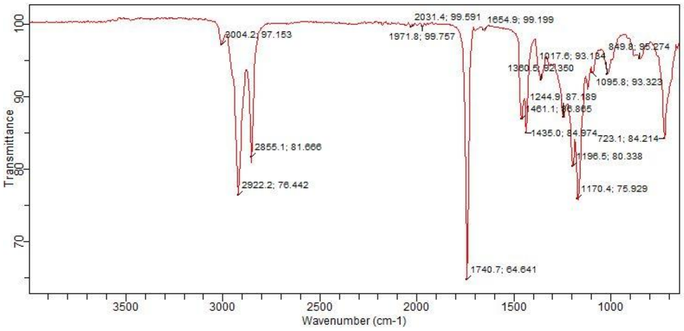

Figure 3: FTIR spectra of khaya Senegalensis Biodiesel
Source: Ishaya et al. (2022)

Table 3: Functional groups in Khaya Senegalesis 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)

Source: Ishaya et al. (2022)

According to the FTIR spectrum by Ishaya et al., (2022) in table 3; =C-H was detected at 3004 cm^{-1} that is coherent with (Attia et al., 2020), C-H bond was observed at 2855-2922 cm^{-1} and is consistent with (Augustina et al., 2020), C=O at 1744 cm^{-1}, C=C at 1654 cm^{-1} that is in agreement with (Singh & Abd, 2020), C-H terminal alkane bond at 1435-1461 cm^{-1}, C-O at 1380 cm^{-1}, C-O-C at 1095 cm^{-1} and =C-H bending at 723-850 cm^{-1} in accordance with (Dass et al., 2018). As reported by Ishaya et al., (2022) the study observed: alkanes, alkenes, alkanoic, and esters functional groups are visible in the Khaya Senegalensis biodiesel.

3.4 NMR Analysis of Khaya Senegalesis biodiesel

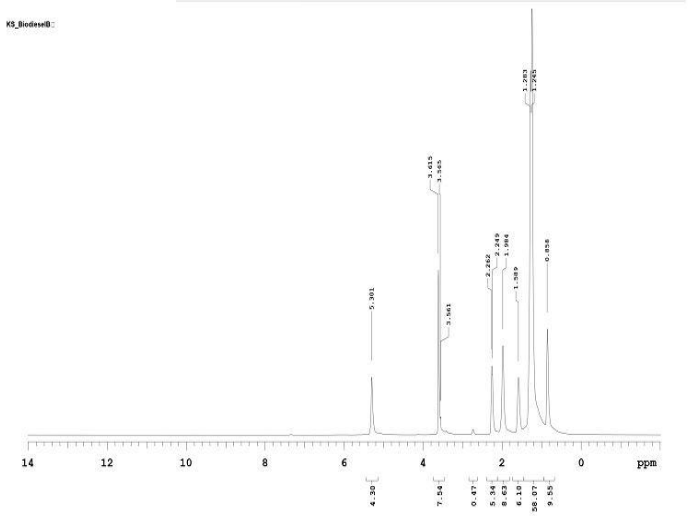

Figure 4: ^{1}H-NMR spectrum of Khaya Senegalensis biodiesel.

Table 4: Chemical shift of main resonances in Khaya Senegalensis biodiesel

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

According to the ^{1}H-NMR spectrum of KS biodiesel in Table 4; peaks observed in the range 4.00- 4.20 ppm, indicates conversion of glycerides. The presence of peaks at 3.6 ppm implies the formation of fatty acid methyl esters (FAME) and is consistent with (Hossain et al., 2021). Peak was observed at 5.3 ppm corresponding to carbon-carbon double in the biodiesel. Other carbon-carbon single bonds were identified in the range of 2.3-0.9 ppm; that is consistent with (Fu et al., 2021).

Based on the ^{1}H-NMR result, the study observed: alkanes, alkenes, carbonyl and esters chemical environments are present in Khaya Senegalensis biodiesel. The ^{1}H-NMR results also show the presence of esters molecules in the sample biodiesel (H_3C-O at 3.6 ppm) that further validates the study by Ishaya et al., (2022) that observed esters (C-O 1244-1170 cm^{-1}).

3.5 FTIR of Conventional Diesel

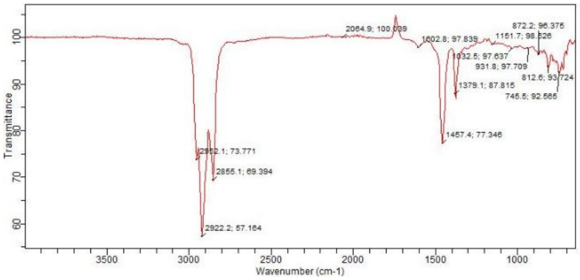

Figure 5: FTIR spectra of conventional diesel

Table 5: Functional groups in conventional diesel

S/N	Wavelength (cm^{-1})	Functional group
1	2952	C-H_2 medium stretch (alkene)
2	2922, 2855	C-H_3 asym./sym. strongh stretch (alkanes)
3	1602	C=C weak stretch (conjugated alkene)
4	1379- 1457	C-H_2/ C-H_3 medium stretch (alkane bending)
5	740-812	C=C-H weak stretch (alkene bending)

From the results of the FTIR spectrum in figure 5; C-H_2 and C-H_3 asymmetric and symmetric bond were observed at 2952 cm^{-1} and 2855-2922 cm^{-1} respectively and is consistent with (Qasim et al., 2017), C=C at 1602 cm^{-1} that is in agreement with (Ismail et al., 2009), C-H_2/C-H_3 alkane bond at 1379-1457 cm^{-1}, and =C-H bending at 723-850 cm^{-1} that is in agreement with (Attia et al., 2020).

Based on the FTIR result, the study observed: alkanes and alkenes functional groups are present in the conventional biodiesel.

3.6 NMR Analysis of Conventional Diesel

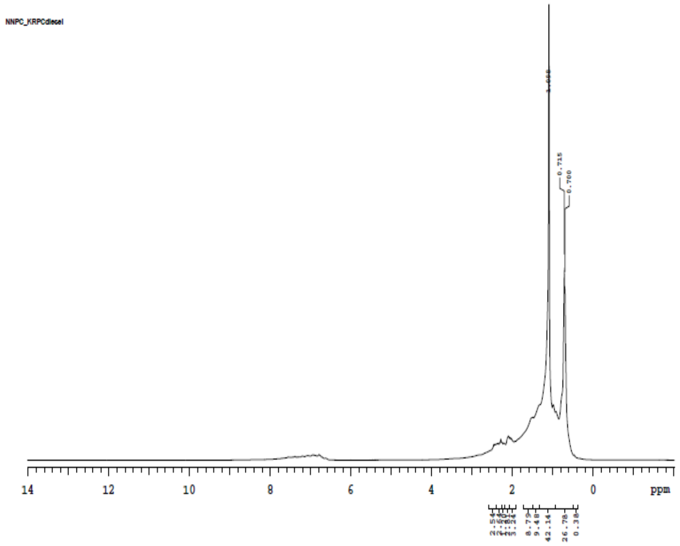

Figure 6: ^{1}H-NMR spectrum of conventional diesel.

Table 6: Chemical shift of main resonances in conventional diesel

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

According to the ^{1}H-NMR spectrum of KS biodiesel in figure 6; protons in terminal methyl (CH_3) groups were seen in the range 0.71-0.9 ppm which is consistent with (Vukovi et al., 2018). Methylene $(CH_2)_n$ groups were also seen in the range 1.09-2.0 ppm and α-CH_2 protons in the range 2.0-2.8 ppm, this is in agreement with (Sharma et al., 2020).

Based on the ^{1}H-NMR result, the study observed alkanes and alkenes chemical environments present in the conventional diesel. The functional groups observed in the FTIR of figure 5 are consistent with the chemical environments identified by the ^{1}H-NMR of figure 6 which validates the functional groups present in the conventional diesel.

3.7 ^{1}H-NMR Validation

Based on the FTIR spectrum observations made by Ishaya et al., (2022) on the Khaya Senegalensis crude oil, that study established that alkanes, alkenes, and esters functional groups are present in the crude oil.

Table 7: Comparing FTIR and H-NMR of KS crude oil

FTIR Results		**H-NMR Results**	
Wavelength (cm^{-1})	Functional group	Chemical Shift (ppm)	Nature of carbon
3004	=C-H alkane	5.31-5.28	HC=CH alkene
2922, 2855	C-H alkane	0.83	CH_3 alkane
1744	C=O alkanoic/ester		
1461	C-H alkane bend	2.26	α- CH_2 alkane
1375	C-O alkanoic		
1103-1256	C-O esters	4.11-4.27	-CH_2OCOR ester
1028	C-O-C alkanoate	4.11-4.27	-CH_2OCOR ester
723	C=C-H alkene bending	5.31-5.28	HC=CH alkene

The ^{1}H-NMR result compared with FTIR in table 7 also indicated the presence of alkanes, alkenes, carbonyl and esters chemical environments in the crude oil. The ^{1}H-NMR further shows that the increasing order of relative abundance of functional groups in the sample crude oil is carbonyl < alkene < alkane marked by the peak intensities as shown in figure 2.

Based on the FTIR observation made by Ishaya et al., (2022) on the Khaya Senegalensis biodiesel, the study established that alkanes, alkenes, carboxylic, and esters functional groups are clearly visible.

Table 8: Comparing FTIR and H-NMR of KS biodiesel

FTIR Results		**H-NMR Results**	
Wavelength (cm^{-1})	Functional group	Chemical Shift (ppm)	Nature of carbon
3004	=C-H alkane	1.9	$H_2C-C=C$
2922, 2855	C-H alkane	0.9	CH_3 alkane
1744	C=O alkanoic/ester		
1654	C=C alkene	5.3	HC=CH alkene
1461, 1435	C-H alkane bend	2.3	α- CH_2 alkane

1380	C-O alkanoic		
1170-1244	C-O esters	3.6	H_3C-O ester
1095	C-O-C alkanoate		
850-723	C=C-H alkene bending	5.3	HC=CH alkene

Chemical environments corresponding to alkanes, alkenes, carbonyl and esters in the biodiesel were also observed by the ^{1}H-NMR spectrum in table 8. The ^{1}H-NMR suggest the relative abundance in order of alkene < esters < alkanes as illustrated by peaks in figure. 4.

Both FTIR and ^{1}H-NMR agrees that conventional diesel contains alkanes and alkenes functional groups. Based on the resonances in ^{1}H-NMR, the methylene group has the highest abundance in the conventional diesel.

3.8 Functional Groups Comparison between Khaya Senegalensis biodiesel and Conventional Diesel

Based on the FTIR and ^{1}H-NMR spectra of Khaya Senegalensis biodiesel and conventional diesel carried out in this study; It was observed that both fuels have alkanes and alkenes functional groups in common. The terminal methyl in conventional diesel is higher due to higher intensity in figure 6 compared to biodiesel in figure 4. This is indicative of the presence of short chains hydrocarbons in conventional diesel. The intensities of methyl groups in both fuels are similar. This is because the long chains in biodiesel are compensated by the plenty short chains in the conventional diesel. The presence of alkanes supports high energy content and short ignition delay in both fuels. Alkenes presence improves the fluidity property of both fuels. The presence of ester functional group in biodiesel improves fuel properties such as low viscosity, high flash point and more energy per unit volume as supported by (Ishaya et al., 2022).

4.0 Conclusion

Based on this study; ^{1}H-NMR validation of chemical transformation from Khaya Senegalensis oil to biodiesel and comparison between the biodiesel and conventional diesel. The following conclusion can be drawn:

The ^{1}H-NMR spectrum of the Khaya Senegalensis crude oil identified the presence of glyceride molecules (CH_2OCOR ester at 4.11-4.27 ppm in ^{1}H-NMR compared to C=O alkanoic/ester at 1744 cm^{-1}, C-O-C alkanoate at 1095 cm^{-1} in FTIR) as supported by the earlier study (Ishaya et al., 2022). The ^{1}H-NMR spectrum of the Khaya Senegalensis biodiesel also confirmed the presence of alkanes, alkenes and esters chemical environments (CH_3 alkane at 0.9 ppm, α- CH_2 alkane at 2.3 ppm, HC=CH alkene at 5.3 ppm, and H_3C-O ester at 3.6 ppm respectively). This further confirms the chemical transformation from crude Khaya Senegalensis oil to biodiesel by FTIR (C-H alkane at 2922-2855 cm^{-1}, C=C alkene at 1654 cm^{-1}, C-O esters at 1244-1170 cm^{-1} respectively) as earlier reported by (Ishaya et al., 2022). The ^{1}H-NMR spectrum of conventional diesel suggest presence of short chains hydrocarbons due to the intensity of terminal methyl groups as compared to biodiesel that is made up of long chains esters. The ^{1}H-NMR spectrum of Khaya Senegalensis biodiesel shows esters (and H_3C-O ester at 3.6 ppm) which validates the FTIR study (C-O esters at 1244-1170 cm^{-1}) by (Ishaya et al., 2022). The esters are responsible for the low viscosity, high flash point and more energy per unit volume of the biodiesel. From this investigation; it was observed that ^{1}H-NMR spectroscopy further validates the FTIR spectroscopy study by (Ishaya et al., 2022) in the conversion of crude Khaya Senegalensis oil to biodiesel through the chemical transformation of triglyceride to fatty methyl esters. This study also established that; Khaya Senegalensis biodiesel has functional groups similar to those present in conventional diesel that supports good fuel properties.

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Declaration of Conflict of Interest

The authors of this paper declare no conflict of interest.

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