

CHEMICAL COMPOUND TRANSFORMATION OF Canarium Schweinfurtii USING FTIR & GCMS TECHNIQUES IN BIODIESEL PRODUCTION



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 Received: September 21, 2022
 Accepted: November 12, 2022

ABSTRACT: This study is aimed at identifying the functional group of chemical compounds, then quantifying their active components and analyze the chemical transformations from Canarium Schweinfurthii (CS) crude oil to its biodiesel as evaluated using FTIR and GCMS techniques. The CS seed kernels were collected from the mature plants at Kagoro, Kaduna State, Nigeria. The oil from the seed kernels was extracted using mechanical expellers press method. Alkaline-catalysed transesterification process was used to convert other triacylglyceride to the desired fatty acid alkyl ester (FAAE). Agilent technologies FTIR Spectrophotometer using potassium bromide disc method, scanning range at 650 to 4000 cm⁻¹ was used to record the infrared spectrum of the samples. For the GCMS, 1µL oil were injected into a gas chromatography (GCMS-QO2010 Shimadzu Japan) fitted with a Mass Spectrometer (MS) detector and a fused silica column, packed with Elite-5MS (5% biphenyl 95% dimethylpolysiloxane, $30m \times 0.25mm$ ID $\times 250\mu m$ df). The unknown spectrum from the GCMS result were matched with known compounds of National Institute Standard and Technology library (NIST05s) as reference for identification. Consequently, the =C-H functional group in the crude oil was not affected by the transesterification reaction forming the biodiesel. However, it showed strong stretch at 1707cm⁻¹ in the crude oil due to C=O carboxylic group shifted to 1740cm⁻¹ in the biodiesel. The GCMS of the crude oil showed 9-Octadecenoic acid (Z)- with 56.71% area, while 9-Octadecenoic acid methyl ester with 22.23% area and 16-Octadecenoic acid methyl ester with 20.55% area were found in the biodiesel.

Keywords: Canarium Schweinfurthii, FTIR, Functional group, GCMS, Biodiesel, Methyl Esters.

Introduction

Heat engines uses heat derived from burning fuel to produce net or useful work. Internal combustion engines (ICE) are a type of heat engines. They burn gasoline or diesel fuel depending on the engine type, to produce work. Both gasoline and diesel fuels are fossil based. However, due to greater fuel economy and ease of operation, engines used for commercial transportation and mechanized agriculture are diesel based. Diesel fuel is more consumed than gasoline fuel (Chauhan et al., 2012; Ramadhas et al., 2005). Fossil fuel sources are faced with exhaustion in nearby future, rising prices, global warming, and environmental pollution (Samuel et al., 2019; Sani et al., 2018). Biodiesel is a renewable energy source that is a potential alternative for diesel fuel (Kaisan et al., 2018). Biodiesel are mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fat via transesterification, epoxidation reactions in the presence of a catalyst (Karaye et al., 2021; Ibeto et al., 2012). The advantages of biodiesel over diesel fuel are biodegradability, renewability, non-toxic, low emissions when used in ICE, easy blending with diesel, and high cetane number (Kaisan et al., 2018; Odjobo & Umar, 2019). More than 350 edible and non- edible crops capable of producing oil have been identified suitable for biodiesel production (Odjobo & Umar, 2019). In Nigeria, Black date (Canarium Schweinfurthii) is one of the oil-bearing crops, from subclass *rosidae* and sub-kingdom *tracheophyta*. The tropical tree is found in the northern and south-eastern parts of Nigeria. The tree is a perennial plant grown purposely for its edible fruits, seed kernel and oil. At maturity, the tree's height is between 12-57m, with feathery like shiny leaves. The tree produces it fruits in the rainy season, and it is commonly known as "Atile" in Hausa, "Ubengba" in Igbo and "Origbo" in Yoruba (Duru & Maduka, 2020; Odjobo & Umar, 2019).

Examples of researches in biodiesel production are: Lin and Lei, (2021) who produced biodiesel from palm oil with various initial contents. The optimum initial water content was found to be between 0.02wt% and 0.05wt %. Alphonso Mango seed oil was used to produced biodiesel by Momoh et al., (2014), the optimum yield was obtained at 60°C, 60mins and 1:4 oil to methanol ratio. Ibrahim et al., (2016) used calabash seed oil to produced biodiesel at 0.6, 0.8, 1.0, 1.2 and 1.4% w/w of oil catalyst loading. The biodiesel yield was 61.8, 62.3, 69.7, 69.5 and 78.1% respectively. Mustapha et al.,(2020) used alkaline refinement to produced biodiesel from waste cooking oil. It was shown that alkaline treated cooking oil had improved physicochemical and fuel properties over waste cooking oil. CaO/MgO catalyst was used to produced biodiesel from Jatropha Curcas seed oil by Ibrahim et al., (2013) for 50, 60, 70, 80, and 90 minutes. The biodiesel yield was 98.4, 99.0, 98.3, 98.1 and 100.0%



respectively. Sani et al., (2018) produced blends of diesel and biodiesel from lanatus seeds oil denoted as B5, B10, B15, B20, and B25 respectively. It was observed that most of the physico-chemical properties of the blends falls within the American Society for Testing and Materials (ASTM) standards for biodiesel. Kaisan et al., (2017) produced binary and multi-blends of diesel with Cotton, Jatropha and Neem biodiesel denoted as B5, B10, B15, B20, B25 and B30 respectively. It was established that most fuel samples had heating values above ASTM standards and all samples were consistent with ASTM standards for flash point and cetane number. The GCMS technique used by (Kaisan et al., 2018) to compare Jatropha and Neem seed biodiesel, showed that Jatropha biodiesel contained 91.9% methyl ester and Neem biodiesel contained 70.2% methyl ester. Samuel et al., (2019) produced biodiesel from rubber seed oil. The highest yield of 92.5% rubber seed oil ethyl ester was obtained at ethanol/oil molar ratio of 6/1. KOH of 4.5wt% and 40 min reaction time.

In the present study, Black date (*Canarium Schweinfurthii*) seed kernel was used to produce biodiesel. Reason because of its high content of unsaturated fatty acid that favours low viscosity of fuel. The aim of this study was to analyze the chemical transformations from crude oil to biodiesel via FTIR and GCMS technique. The contribution of this study is to add more alternatives to diesel fuel.

Materials and Methods Materials

Seed Collection and Preparation

Canarium schweinfurthii seeds were collected from the plants grown at Kagoro, Kaura local government in Kaduna State, Nigeria.

The collected seeds were separated and cleaned of fruits pulp/fibres and other dirts. The seeds kernels were dried under the sun for 6 hours daily- from 10:00 hour to 16:00 hour for fifteen (15) days to reduce their moisture content. The dried kernels were roasted and decorticated. The kernels (Mesocarp – the soft fleshy part) as stored were further dried by heating at 50°C using a dryer for one hour. The dried kernels was crushed to grains / powder using a grinder. The ground kernels were then sieved to get less than or equal to 1mm particle size powder for more surface areas pores exposure, which is crucial for the oil extraction process. The dried and ground powders were weighed, and values were recorded.

Methods

Oil extraction procedure

The engine driven mechanical expeller press method outline by Kaisan *et al.*, (2014) was adopted for this study. The machine was operated at 120 bars to extract oils from *Canarium schweinfurthii* kernels powder, preheated at 50°C. The block diagram illustrating the oil extraction process is shown in Figure 1.

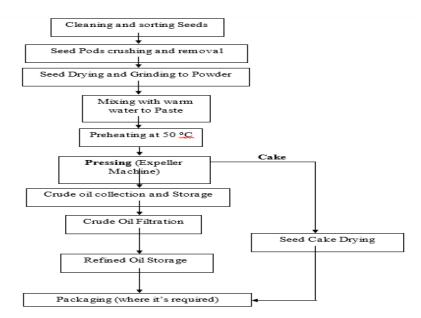


Figure 1: The oil extraction process



Biodiesel Production

Free fatty acid (FFA) determination

The acid value of the oil was determined by dissolving 1.0 g of oil sample in 25 ml of propan-2-ol, two drops of phenolphthalein indicator was added in a conical flask and the solution formed was titrated against 0.1M Potassium hydroxide (KOH) poured up to the 25ml level of a burette clamped on a resort stand, until the titrate turned red in colour. The titre value was recorded. Two more trials of titration were conducted and the average value from the three titre values was calculated as the accepted titre value. A blank titration was also performed by titrating 25 ml of propan-2-ol against 0.1 M potassium hydroxide to red colour appearance. The difference of the two titre values, v was used for determining the Free Fatty Acid (FFA) of the feed stock as expressed by (Ibrahim et al. 2014). The percentage FFA and acid values were calculated using equation 1 and 2 respectively.

Free Fatty Acid $(FFA) =$							
KOH Titre Value x Molarity of KOH x Molar Mass of KOH							
Mass of oil							
Acid Value $(A.V.) = \frac{\% \text{ FFA}}{2}$	2						

Esterification reaction

For %FFA greater than 0.5 mg KOH/g, then the esterification process was carried out and repeated on multiple runs until the %FFA of the subsequent esterified oil is ≤ 0.5 mg KOH/g. An appropriate mix of methanol (CH₃OH) as the reagent and sulphuric acid (H₂SO₄) as the catalyst was added into the heated (heated to 60°C) oil in the conical flask placed on a regulated hotplate. A magnetic Stirrer was inserted into the mix and stirred at 150 rpm at the controlled temperatures of 60°C for 60 minutes. The conical flask was sealed with aluminium foil paper with a graduated laboratory thermometer inserted. The thermometer is to aid the manual regulation of the reaction temperature of 60°C to minimize the evaporation of methanol as its boiling temperature is 64.7°C and to support stirring manually, while the foil paper is meant to retain the evaporated and escaping methanol molecules at 60°C by reflecting them back into the mixture. This is to help maintain a consistent methanol to oil molar ratios and to ensure the reaction temperature stays at 60°C throughout the reaction time of 60 minutes. Also, the magnetic stirrer is to maintain continuous stirring at 150 rpm for homogeneity and consistency of the binding sites. The mass of each reagent KOH, H2SO4, the mass of alcohol and mass of the catalyst were determined using Equations 3, 4 and 5 respectively as given by (Ibrahim et al., 2016)

Mass of sbustance = % FFA of the oil X Molarity of substance X Mass of oil being processed 3

$$Mass of Alcohol (g) = \frac{Mass of Oil (40)(g)x [Alcohol Molar ratio x Molar mass of Alcohol (32.04)(g)]}{Molar mass of Trioleum (885.46) (g)}$$

$$Mass of Catalyst = Mass of oil (g) x \frac{Percent of Catalyst Concentration as selected (\%)}{100}$$
5

Transesterification reaction

The base or alkaline-catalysed transesterification process was used to convert other triacylglyceride to the desired biodiesel or fatty acid alkyl ester (FAAE) because it is a faster and less corrosive process compared to the acidcatalysed type. The transeterification process is similar to the esterification process which has same reaction conditions of 60° C, 60 minutes and rate of agitation, but may be at different alcohol (CH₃OH) to oil molar ratios and different catalyst (NaOH) concentrations. For this production, the methanol to oil molar ratio is ratio six to one (6:1) and the catalyst concentration is 0.25% NaOH.

Purification of biodiesel

The ester (biodiesel) at the top layer of the funnel was separated by draining the glycerol. The biodiesel in the funnel was then purified by washing impurities from the finished biodiesel many times with distilled water to reach neutral pH of \sim 7.0, and to remove all the residual by-products like excess alcohol, excess catalysts, and glycerol

remainder. The volume of distilled water added each time was 30% of the biodiesel by volume. The funnel was shaken gently for 1 minute and left to stand for 3 hours on the table to allow separation of the biodiesel layer at the top and water layers at the bottom. After separation, the water was drained, and the biodiesel was transferred to a clean conical flask. The washing process was repeated several times until the washing water became clear. The clean biodiesel was dried using sodium sulphate in an incubator for 48 hours to further remove the small amount of water that is made during the chemical reaction of transesterification and wash-clear process. Biodiesel was dried to avoid build-up of scale and microbial growth in storage containers. These dirt can cause blockages in fuel delivery pipes resulting to some loss of mechanical power. A quick test which is also known as Ester test was performed on each of the biodiesel samples produced by dissolving 10 ml of biodiesel in 40 ml of methanol. The mixture was stirred thoroughly and left for 10 minutes. Complete miscibility and dissolution confirm complete transesterification reaction. After 10 minutes, it



was observed that there was a homogenous mixture without traces of oil.

Determination of functional groups and compounds

Agilent technologies FTIR Spectrophotometer was used to record the infrared spectrum of the samples. The scanning range was 650 to 4000 cm⁻¹ and the IR spectra of samples was obtained using potassium bromide disc method. In the present study, FTIR was used to analyze the functional groups in the crude oil and Biodiesel samples.

A GCMS method outlined by (Odjobo & Umar, 2019) was adopted. 1 μ L of sample oil was injected into a gas chromatography (GCMS-QO2010 Shimadzu Japan) fitted with a Mass Spectrometer (MS) detector and a fused silica column, packed with Elite-5MS (5% biphenyl 95% dimethylpolysiloxane, 30m × 0.25mm ID × 250 μ m df). The initial temperature was 60°C and maintained for 0min, then

increased by 10°C per min till it reaches 180°C, held for 2min and then finally increased by 15°C per min to reach 280°C. The final temperature was held for 4 min in the column. The oven, injector and interface temperatures were fixed at 60°C, 250°C and 250°C respectively. Helium was used as carrier gas at a constant flow of 1mL/min at a 50:1 split ratio. GC-MS was used to analyze the compounds in the crude oil and Biodiesel, and their respective percentages. The unknown spectrum from the GCMS result was compared with the National Institute Standard and Technology library (NIST05s) as reference. Compounds were identified according to molecular mass, structure, retention times, mass spectra and fragments calculated. The most probable compound was the one with the closest match.

Results and Discussion *FTIR Analysis*

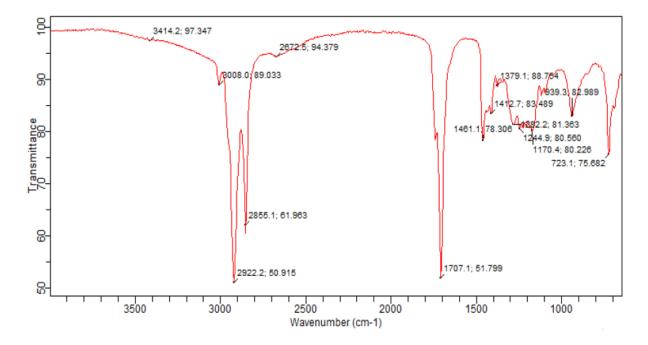


Fig 1: FTIR spectrum of crude Black date oil

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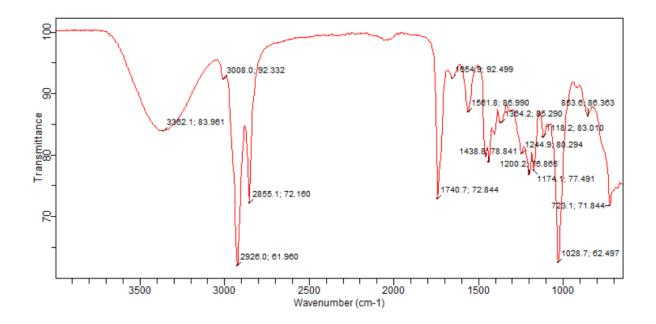


Fig 2: FTIR spectrum of black date biodiesel

S/N	Wavelength (cm ⁻¹)	Crude oil	Biodiesel	Functional group	
1	3414	O-H weak band		Carboxylic acid	
	3362		O-H broad band	Alcohol	
2	3004	=C-H weak stretch	=C-H weak stretch	Alkene	
3	2922, 2855	C-H Asymmetrical and symmetrical strong stretch	C-H Assym and sym strongh stretch	Alkane	
4	2672	O=C-H weak stretch	Aldehyde		
5	1707	C=O strongh stretch		Carboxylic acid	
	1740	C C	C=O strong stretch	Ester	
6	1654		C=C weak stretch	Alkene	
	1561		C=C medium stretch	Alkene	
	1461	C-H medium stretch (methyl bending)		Alkane	
	1438		C-H medium stretch (methyl bending)	Alkane	
7	1379	C-H methyl rock		Carboxylic	
	1364		C-H methyl rock	Ester	
8	1282-1170	C-O medium stretch		Ester	
	1244-1174		C-O medium stretch	Ester	
9	1028		=C-H strong stretch	Alkene bending	
	939	O-H medium stretch	-	Carboxylic bending	
10	853		C-H bend	Alkane bending	
11	723	C-H long chain methyl rock	C-H long chain methyl rock	Alkane	

Table 1: Functional groups in Black date crude oil and Biodiesel

From Table 1; the weak O-H band at 3414cm⁻¹ in crude oil was due to carboxylic group in free fatty acid (FFA). This band is very weak, indicating very few FFA and majorly because the structures of plant oils is triglyceride. As

observed from fig. 2, the presence of a medium broad O-H band at 3362cm⁻¹ in the biodiesel. Reason because unsaturated carbon atoms in the long chain reacted with methanol and can also be due to traces of alcohol that was



used in the synthesis of the biodiesel, as also observed by (Oil et al., 2019). No transformation occurred to the =C-H bond at 3004 from crude oil to biodiesel. This is because transesterification reaction affects only the carboxylic group and not the double bond. The C-H asymmetrical and symmetrical stretch at 2922 and 2855cm⁻¹ remains unaffected in both oils. Reason is that the long fatty acid chain that contains the C-H bond does not undergo any reaction during transesterification. A weak stretch of aldehyde at 2672cm⁻¹ in the crude oil has disappeared in the biodiesel. This can be due to substitution of hydrogen attached to the C=O in the aldehyde. A strong stretch at 1707cm⁻¹ in the crude oil due to C=O carboxylic group was transformed to an ester C=O with a strong stretch at 1740cm⁻ ¹ due to transesterification reaction. The C-H methyl bend in the crude oil at 1461cm⁻¹ was transformed to a cyclic C-H in-ring stretch at 1438cm⁻¹ in the biodiesel, due to transesterification reaction. The C-H methyl rock at 1379cm⁻¹ in the crude oil was transformed to an ester C-H methyl rock at 1364cm⁻¹ due to the transesterification process. The carboxylic C-O stretches from 1282-1174cm⁻¹ in the crude oil, was transformed to ester C-O stretches from 1244-1174 in the biodiesel because of the transesterification reaction. Strong stretch of =C-H bending in the biodiesel occurred at 1028cm⁻¹, weak carboxylic O-H bending in the crude oil stretched at 939cm⁻¹, and both oils showed C-H methyl rock at 723cm⁻¹ due long chain alkanes.

This finding suggests that: alkanes, alkenes, and long chains alkanes are present in both the crude oil and biodiesel. A marked transformation has occurred from carboxylic group to ester group due to the transesterification reaction.

GCMS Analysis

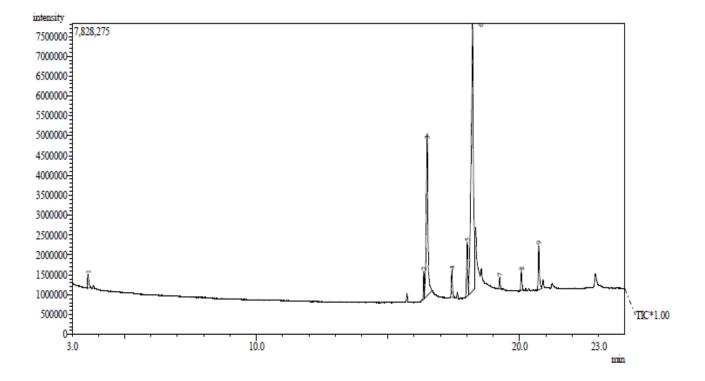


Fig 3: GCMS spectrum of crude Black date oil



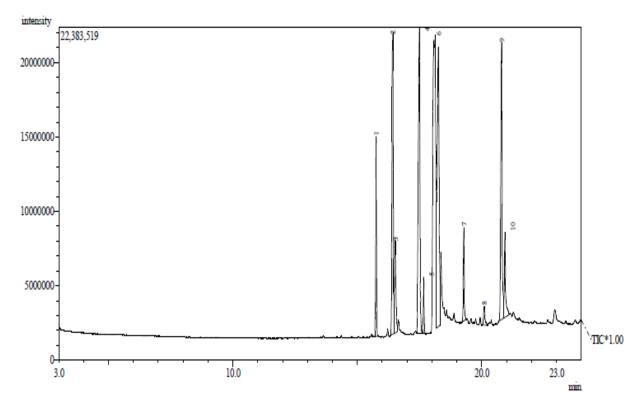


Fig 4: GCMS spectrum of Black date biodiesel

	Crude oil			Biodiesel		
S/N	Library/ID	Area %	Formula	Library/ID	Area %	Formula
1	Acetic acid, 2-propenyl ester	1.57	C5H8O2	Palmitic acid, methyl ester	7.34	C17H34O2
2	Palmitic acid, ethyl ester	1.95	$C_{18}H_{36}O_2$	Palmitic acid ethyl ester	16.32	$C_{18}H_{36}O_2$
3	Palmitic acid	24.54	$C_{16}H_{32}O_2$	Palmitic acid	5.80	$C_{16}H_{32}O_2$
4	16-Octadecenoic acid, methyl ester	2.74	C19H36O2	16-Octadecenoic acid, methyl ester	20.56	C19H36O2
5	E-11-hexadecenoic acid, ethyl ester	5.36	C18H34O2	Octadecanoic acid, methyl ester	2.11	C19H38O2
6	9-Octadecenoic acid (Z)-	56.71	$C_{18}H_{34}O_2$	9-Octadecenoic acid ethyl ester	22.23	$C_{20}H_{38}O_2$
7	Octadecane, 1- (ethenyloxy)-	0.96	$C_{20}H_{40}O$	Hexadecanoic acid, 2,3- dihydroxypropyl ester	3.77	C ₁₉ H ₃₈ O ₄
8	2-Heptanol,2-methyl-	1.79	$C_8H_{18}O$	3-Tetradecanol	1.16	$C_{14}H_{30}O$
9	9,12-Octadecadienoic acid (Z,Z)-	4.39	$C_{18}H_{32}O_2$	18-Nonadecec-1-ol	16.29	C ₁₉ H ₃₈ O
10				Octadecane, 1- (ethenyloxy)-	4.42	C20H40O

From Table 2, 9-Octadecenoic acid (Z)- has the highest percentage area of 56.71% in crude oil. After the transesterification process, 9-Octadecenoic acid ethyl ester highest area of 22.23% and 16-Octadecenoic acid methyl ester with 20.55% area were formed in the biodiesel. Palmitic acid with 24.54% in the crude oil was reduced to 5.80% in the biodiesel indicating incomplete reaction. New product of palmitic acid methyl ester with 7.34% area was

formed in the biodiesel. Palmitic acid ethyl ester occupying 1.98% area in the crude oil increased to 16.32% area in the biodiesel, because some of the palmitic acid in the crude oil were converted due to transesterification reaction. 9,12-Octadecadienioc acid was transformed to Octadecanoic acid methyl ester occupying 2.11% in the biodiesel. Alcohols like 3-Tetradecanol and 18-Nonadecen-1-ol were also detected in the biodiesel. This is due to the addition reaction between



the unsaturated carbon atoms in the long chain and the methanol during the transesterification reaction.

The FTIR and GCMS analysis of the chemical transformation of Canarium Schweinfurthii from crude oil to biodiesel, showed agreement. The FTIR in Table 1 identified alkanes, alkenes, long carbon chains, esters, and carboxylic groups in crude oil of black date palm. These functional groups were also confirmed in the crude oil by the GCMS analysis in Table 2. Similarly, The FTIR in Table 1 identified alkanes, alkenes, long carbon chains, esters, and carboxylic groups in biodiesel of black date palm. These functional groups were also confirmed in the biodiesel by the GCMS analysis in Table 2. The difference between the crude oil and biodiesel was the conversion of carboxylic groups to esters due to transesterification reaction. The changes in percentage area of fatty acids and esters in the crude oil and the biodiesel was a main difference.

Conclusion

In the present study, FT-IR and GC-MS compound transformation as analyzed from crude oil to biodiesel of Black date (*Canarium Schweinfurthii*) was investigated. The following conclusions can be drawn from the results:

The FTIR analyses of crude oil and biodiesel showed that; the =C-H functional group in the crude oil was not affected by the transesterification reaction; it also appeared in the biodiesel. This is because, transesterification reaction occurs between carboxylic group and alcoholic group. The FTIR analyses showed that the strong stretch at 1707cm⁻¹ in the crude oil due to C=O carboxylic group shifted to 1740cm⁻¹ in the biodiesel. This was due to the formation of ester C=O group due to transesterification reaction. The FTIR analyses showed presence of alcohols in the biodiesel due to the addition reaction of unsaturated carbon atoms with methanol.

Th GCMS analyses of crude oil showed 9-Octadecenoic acid (Z)- with 56.71% area, while 9-Octadecenoic acid ethyl ester with 22.23% area and 16-Octadecenoic acid methyl ester with 20.55% area were found in the biodiesel. This indicates successful transesterification. The presence of palmitic acid in the biodiesel showed incomplete transesterification reaction. The detection of alcohols; 3-Tetradecanol and 18-Nonadecen-1-ol in the biodiesel confirmed the presence of a medium broad O-H band at 3362cm⁻¹ in the FTIR analyses of the biodiesel.

Acknowledgements.

The authors acknowledge the support of the following Laboratories for the provision of the equipment used in conducting the experiments; 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. All Authors were involved in the laboratory work, writing of the manuscript and in the interpretation of the results.

Declaration of Conflict of Interest

The authors have no conflict of interest to declare.

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