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Analyzing the Fatty Acid Methyl Esters Profile of Palm Kernel Biodiesel using GC/MS, NMR and FTIR Techniques

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ABSTRACT

Rapid depletion of fossil fuel and increased demand for petroleum products in energy and transportation sector motivated the researchers to find a substitution for petroleum diesel. Biodiesel is one among the alternate to this crisis. In this present study, extraction and characterization of Palm kernel biodiesel is studied. The presence of Fatty Acid Methyl Esters is identified using GC/MS technique. The study revealed the presence of 12 prominent methyl esters especially between C11:0 and C24:0 having short and long chain hydrocarbons which includes Undecylic acid, Tridecyclic acid, Pentadecyclic acid, Ginkgolic acid, Myristic acid, Oleic acid, Margaric acid, Gondoic acid, Arachidic acid, Behenic acid, Tricosanic acid and Lignoceric acid. It is noticed that at Retention time 19.07, C14:0 Myristic acid showed a higher level of its presence. Further confirmation is carried out using NMR (^{13}C and ^{1}H) and FT IRspectral studies which supported the findings of GC/MS spectral data.

KEY WORDS: Biodiesel, Transesterification, Palm Kernel oil, Spectral studies.

1. INTRODUCTION

Presently, the world is confronted with dual crisis. One in the form of demand for fossil fuels for the transportation and energy sector and the other, rapid depletion of fossil fuel along with environmental degradation. Due to growth of economy at 8% to 12% GDP for developing nations like India, China, Brazil and South Africa, the demand for energy and fossil fuel is ever increasing. This led to the need of exploring alternative fuels to meet the ever growing energy demands. Interestingly, vegetable oils as an alternate diesel engine fuels dates back several decades. Rudolf diesel was first to use peanut oil in compression ignition (CI) engine (Ghobadian, 2009). During Second World War, attempts were made to use vegetable oil as diesel engine fuel. However, due to their viscosity, poor volatility and high cost, the vegetable oils were not preferred as a substitute to diesel as a fuel in CI engine.

Vegetable oils hold a special position as it can be produced from the plant grown in rural areas. Vegetable oils extracted from Soyabean, Peanut, Neem, Cottonseed, Sunflower, Rapeseed, Coconut, Linseed, Karanja, Jatropha, Mustard, Pongamia, Castor and others have Cetane number and calorific values comparable to diesel and also, they are compatible with the distribution and vehicle fuel system with minimal modification. However, its main disadvantages are its high viscosity which gives difficulties in fuel injection system along with cold starting issues.Solutions to difficulties with physical properties of neat vegetable oil can be overcome by proven process like transesterification. Transesterification process produces esters of respective oils commonly known as biodiesel. (Agarawaland Das, 2001).

Biodiesel is a non-polluting fuel obtained from organic oils of vegetable origin chemically known as free fatty acid methyl ester (FAME). The esters of fatty acids derived from transesterification of vegetable oils have properties closer to diesel fuels. These fuels tend to burn cleaner, perform comparably to conventional diesel fuel (Agarawaland Das, 2001; Sharma and Bhaskar Singh 2010). Irrespective of the difficulties mentioned above, esters of vegetable oils prove to be one of the alternate options to substitute the diesel fuel in future. Biodiesel is a clean, renewable, non-toxic, bio-degradable, free from Sulphur with higher Cetane value and enhanced oxidizing property leading to better combustion which can be used in any CI engine without major modification (Xu and Wu 2003).India with a large non-arable and primarily agricultural based nation, cultivating crops for producing vegetable oils for energy sector will not be a great burden.

Samios, (2009) analysed a double step transesterification process of Sunflower and Linseed oil for production of their respective biodiesel. They followed consecutive base acid catalysis approach which resulted in easy separation, higher conversion, phase definition and enhanced reaction. The quantity and purity of biodiesel was analysed using standard characterization techniques along with NMR studies. Monterio (2009), experimentally investigated the different types of biodiesel from castor oil and soybean oil from various sources and studied it characteristics using NMR techniques. The blending of fuel was made with three different fatty acid composition and three different diesel fuels were analysed using ^{1}H NMR techniques with calibration curves. Jerekais Gandure (2014), compared the fuel properties of native African plant kernel oil namely Sclerocaryabirrea, Tylosemaesculentum, Schiziophytonrautanenii and Jatropha curcus with diesel fuel. The authors compared the physio-chemical properties of the above mentioned oil under ASTM and EN standards. Oil from Schiziophytonrautanenii proved to be possessing better cold flow property among the other oils.The physical

characteristics of biodiesel produced from waste cooking oil were studied by Sary Awad (2010) and it was compared to European standard EN 14214. The study was performed to identify the optimum conversion ratio by combining the variables namely, catalyst amount, reaction time and temperature, alcohol to oil molar ratio, effect of free fatty acid content in oil and type of alcohol used in the reaction. Jens Wäring and Krister Svanberg (2012). They studied the characterisation of chemical decomposition of rapeseed biodiesel with a focus on B10, B30 and B100 blends.

Characterization of alkali transesterified biodiesel obtained from crude Jatropha was compared to ASTM and EN diesel and biodiesel standards by Traoré and Thiam (2014). Calorific values of diesel decreased from 42.7 to 42.13, 42.330, 42.47 and 41.48 MJ/kg with a blend of 10% of Sandbox seed, Dika nut, Physic nut and Castor bean respectively. The calorific values of the produced biodiesel fromCastor bean, Sandbox seed, Physic nut and Dika nut oils are 30.50, 40.4, 39.005, 37.00 MJ/kg respectively. Results from the tests carried out showed that biodiesel produced conform to ASTM and EN standards. A comprehensive characterization was performed by Camelia Ciubota (2013) on using Camelinasativabiodiesel (CSB) as a viable biofuel alternative based on the European (EN 14214) and American (ASTM D6751) standards. Results from the study showed that CSB contained approximately 90% unsaturated fatty acids. This unusual fatty acid pattern was due to presence of abundant C18:1 (12.8–14.7%), C18:2 (16.3–17.2%), C18:3 (36.2–39.4%) and C20:1 (14.0–15.5%) fatty acids. High C18:3 content was incompatible with EN 14214 standards and it affects negatively the biodiesel properties like Cetane number, iodine value, oxidation stability and linolenic acid methyl ester content.

In this present study, biodiesel is produced from kernels of Palm through two stage transesterification process. The characterization of Palm kernel biodiesel is carried out to analyze it utility as a biodiesel to be used in compression ignition engine using Gas Chromatography Mass Spectrometry. Nuclear Magnetic Resonance (^{13}C and ^{1}H) and Fourier Transform Infra-Red Spectrometry analysis are also conducted for further confirmation of the presence of FAMEs.

2. MATERIALS AND METHODS

Production and Transesterification of Palm Kernel Oil: The palm seed is procured from local vendor in Chennai. The outer layer of the palm is removed for the extraction of edible palm oil which is used for cooking purpose. The palm kernels were collected for the production of Palm kernel oil. It is crushed in a mortar-pestle type crusher for the extraction of oil. In the process 445 grams of palm kernel oil is extracted from 1000 grams of kernel seed and it is heated between 100ºC and 105ºC to remove moisture content. The extracted oil is subjected to two stage transesterification process for converting it into biodiesel since the free fatty acid content is found to be around 8.5%.

The first stage constitutes acid catalysed transesterification in which concentrated hydrochloric acid and methanol are used and followed by base catalysed esterification with sodium hydroxide and methanol at a molar ratio of 1:6. 100 ml of crude palm kernel oil is poured in a round bottomed flask and heated up to 80ºC. 5 ml and 220 ml of concentrated hydrochloric acid and methanol respectively are mixed thoroughly and poured in the flask containing crude Palm kernel oil. Continuous stirring at 200 rpm for 90 min with temperature at 80ºC is carried out and later, it is allowed to cool for 2 hrs in an inverted separating funnel. Phase separation in the mixture enables the removal of sludge and Palm kernel oil with less than 2% of free fatty acids is recovered. By the above process, 82 ml of processed Palm kernel oil is obtained. Base catalysed transesterification is initiated by blending 2.6 grams of sodium hydroxide and methanol at molar ratio of 1:6 to form sodium meth-oxide solution. The remains of first stage (82 ml of palm kernel oil) is thoroughly mixed with sodium meth-oxide solution in a stirrer fitted three necked round bottomed flask and heated up to 80ºC to 85ºC at 250 rpm for 90 min. Then, the mixture is allowed to cool for 60 min. It is noticed that, the transesterification reaction is initiated at 65ºC with the formation of glycerol as the bottom layer. As temperature is increased to 85ºC, the transesterification reaction showed enhanced yield of biodiesel. Beyond 85ºC, sludge formation (negative effect) is observed. By this process 76 ml of Palm kernel biodiesel is obtained at a transesterification efficiency of 92.68%. Even though potassium hydroxide dissolves much easily in methanol compared to sodium hydroxide, NaOH is preferred in this transesterification process as it is cost effective and readily available. The comparison physio-chemical properties of Palm kernel biodiesel with other oils is shown in Table 1. (Knothe and Steidleys 2005; Ahmad, 2010).

Biodiesel Characterization: Chemical analysis is carried out to ascertain that the profiles of Fatty Acid Methyl Ester of Palm kernel oil are of suitable quality for use as a feedstock for biodiesel production. The method involves analyzing standard reference samples, generating calibration curves for fatty acids identified in the standard samples, quantifying and identifying fatty acids present in the FAME palm kernel oil.

The determination of FAME's is carried out by Gas Chromatography Mass Spectrometry equipped with high energy collision induced dissociation linked scan unit, allowing structural analysis. Gas Chromatography is carried out in Agilent 7673 automatic liquid sampler and Agilent 6890 with electronic pressure control. NIST mass spectral database library is bridged with MS-Data Processing Software to identify the compounds. Spectra obtained are measured at a resolution of 6000 and mass range of m/z 1 to m/z 1000 with maximum calibrated mass of 1500 Daltons. Lipid fraction is re-suspended in n-hexane and applied to silica gel column chromatography. Aliphatic

hydrocarbon fraction passes through the column fatty acid and carotenoid fractions are trapped. The one passing through fraction is defined as hydrocarbon fraction and the lipid components in hydrocarbon fraction are identified by GC/MS. The sample measuring one micro litre (1µl) is auto-injected into the system by a split-less injector with the set temperature of 300°C.

Table.1.Comparison of physio-chemical properties – Palm kernel, Soyabean and Jatropha

Properties	Test	Limits	Palm Kernel Oil	Soybean oil	Jatropha
Acid value (mg/ KOH/g)	ASTM D664	0.50 Max	3.25	0.18	0.21
Cetane number	ASTM D613	47 Min	48	63	59
Cloud point(^{0}C)	ASTM	-3 to - 12	-2	-6	-1
Density(Kg/m^3)	ASTM	880	880	784	753
Flash point(^{0}C)	ASTM D93	130 Min	165	157	162
Kinematic	ASTM D445	1.9 to 6	3.68	4.23	3.98
Pour point(^{0}C)	ASTM D97	-15 to -	2	0	-3.5

FTIR Spectral data for palm kernel oil biodiesel is obtained using Perkin Elmer Spectrum One- I Pc FTIR Spectrometer. It is equipped with Kbr pellet Technique and Mylar beam splitter. Spectra data obtained are at a resolution of 1.0 cm^{-1} in the range of 4000 cm^{-1} - 450 cm^{-1}. Lipid fraction is evaporated on the Thalium bromide and FR-IR spectra with the resolution of 4 cm-1, Scan Number: 3.One Dimensional ^{1}H and ^{13}C NMR Spectrum of Palm Kernel oil Biodiesel is measured using Bruker Avance III 500 MHz NMR Spectrometer. Instrument is capable of generating magnetic field strength of 11.7 Tesla with phase resolution better than 0.1° and frequency resolution 0.1Hz. Additionally, it can produce the proton frequency of 500 Mhz. Phase and baseline correction, chemical shift calibrations for resulting spectra is carried out using Bruker Topspin 2.0 Software.

3. RESULTS AND DISCUSSION

Gas Chromatography-Mass Spectroscopy Analysis: In this present study, quantification of Fatty Acid Methyl Esters present in Palm kernel oil biodiesel is categorized using Gas Chromatography Mass Spectroscopy. The major FAME's present in biodiesel sample are tabulated in Table 2.

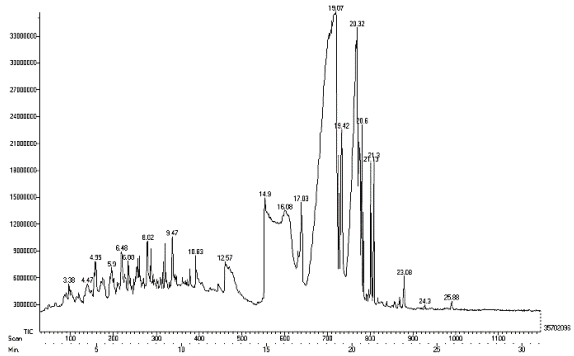

Figure.1. GC-MS spectrum of Palm kernel oil biodiesel

Hydrocarbon grouping is differentiated as <C15, C15 – C20, C20 – C30 and >C30 with respect to the retention time of standard hydrocarbons like Octadecane, Heptadecane, Eicosane, Tricosane and molecular carbon chain length. The analysis revealed that presence of 12 major fatty acids between C11:0 and C24:0 such as Undecylic acid, Tridecyclic acid, Pentadecyclic acid, Ginkgolic acid, Myristic acid, Oleic acid, Margaric acid, Gondoic acid, Arachidic acid, Behenic acid, Tricosanic acid and Lignoceric acid. Among the resulting fatty acids, short chain hydro carbons between C11:0 and C15:0 holds the major mass fraction composition. Typical GC-MS Spectrum of Palm kernel oil biodiesel is shown in Figure 1. Test results also revealed the presence of branched hydrocarbons in FAMEswhich includes Ginkgolic acid, Oleic acid and Gondoic acid which is also termed as Poly Unsaturated Fatty Acid (Lijuan Zhao, 2016).

Fourier Transform Infra-red Spectroscopy: Palm kernel oil biodiesel is subjected to Fourier Transform Infrared Spectroscopy to characterize and emulate the presence of various carbon isomers. Typical FTIR spectrum of Palm kernel oil biodiesel shown in Figure 2 confirms the presence of volatile compounds at varying blending vibrations such as alkanes, aldehydes, alcohols, esters and derivatives of carboxylic acid. Complete inferences of FTIR spectrum is illustrated in Table 3.

Table.2. Fatty acid methyl esters in Palm kernel oil biodiesel

No	Retention Time	Name of the Ester	Name of the Fatty acid	Corresponding Acid	Molecular Structure of Fatty acid	Number of Ions	Scan
1	12.57	Undecanoic acid,10-methyl-,methyl ester	Undecylic acid	C11:0		1445	461
2	14.90	Tridecanoic acid,12-methyl-,methyl ester	Tridecyclic acid	C13:0		1299	554
3	16.08	Pentadecanoic acid, methyl ester	Pentadecyclic acid	C15:0		1237	601
4	17.03	Pentadecanoic acid,14-methyl-,methyl ester	Ginkgolic acid	C15:1		1200	639
5	19.07	Tetradecanoic acid,5,9,13-trimethyl-,methyl ester	Myristic acid	C14:0		978	720
6	19.42	16-Octadecenoic acid,methyl ester	Oleic acid	C18:1		1075	734
7	20.35	Heptadecanoic acid, 9-methyl-, methyl ester	Margaric acid	C17:0		1109	771
8	21.13	11-Eisosenoic acid,methyl ester	Gondoic acid	C20:1		1141	802
9	21.30	Eicosanoic acid, methyl ester	Arachidic acid	C20:0		1117	809
10	23.08	Docosanoic acid, methyl ester	Behenic acid	C22:0		1456	880
11	24.30	Tricosanoic acid, methyl ester	Tricosanic acid	C23:0		1686	928
12	25.88	Tetracosanoic, methyl ester	Lignoceric acid	C24:0		1630	991

Weak-Medium peak signals at 722 cm^{-1} and 880 cm^{-1} showed the presence of aromatic alkyne group and weak benzene derivatives (branched along with Br and Cl). Esters known to be a phenomenal component for the biodiesel, it contributes the major stretching areas between 1016 cm^{-1} to 1246 cm^{-1} and the signal at 1743 cm^{-1} with continuous, broad, strong-medium signal. Presence of carboxylic derivative is identified by the inconsistent strong signal obtained at areas of 2854 cm^{-1} and 2925 cm^{-1}. The medium signals found at 1363 cm^{-1} to 1465 cm^{-1} and 3468 cm^{-1} showedthe presence of phenols, aldehydes and alkene groups at very minimal level.

Table.3. Palm kernel biodiesel spectrum of FTIR analysis

No	Wave Length (cm^{-1})	Inferences of FTIR Spectrum	Signal Characteristic
1	722	Aromatic C-H Bending, Benz. Monosubst, Benz.1,3-disub, Benz.1, 2, 3-trisub, Bromides $(C,H)_3$=-C-Br, Chlorides $(C,H)_3$=-C-Cl.	Medium
2	880	Benz.1, 2, 4-trisub, Benz.1, 3, 5-trisub.	Weak, Broad
3	1016, 1117, 1170,1196,1246*	Alcohol/Phenol O-H & C-O Stretch, Esters uconj. C=O & C-O Stretch,*Acetate C (=O)-O-.	Medium, Broad
4	1363	CH_3-C(=O)-O (CH_3), CH_3-O-C(=O)R (CH_3), CH_3-X (X=halogen) (CH_3).	Medium
5	1436	Ammonium ion, CH_3-C (=O)-O (CH_3), CH_3-N= (R1, R2) (CH_3), CH_3-O-C (=O) R (CH_3), CH_3-X (X=halogen) (CH_3), CH_3-S- (CH_3).	Medium
6	1465	CH_3-O-R (CH_3), CH_3-NR-C (=O) - amides (CH_3), CH_3-X (X=halogen) (CH_3).	Medium

7	1743	Esters uconj. C=O & C-O Stretch.	Strong
8	2854	Alkyl C-H Stretch, Carboxylic Acid C=O & O-H Stretch unconj, Methylene (-CH_2-) C-H stretch.	Medium
9	2925	Alkyl C-H Stretch, Ammonium ion, Carboxylic Acid C=O & O-H Stretch unconj, Methylene (-CH_2-) C-H stretch.	Strong
10	3468	Alcohol/Phenol O-H & C-O Stretch, Amine (prim, ali) N-H Stretch.	Weak

Nuclear Magnetic Resonance

^{1}H-NMR: Proton NMR classifies the presence of hydrogen functional group at various places of carbon chain. ^{1}H NMR spectrum of Palm kernel oil biodiesel is shown in Figure 3. The presence of alkyl, alkynes, ethers, alcohols with α-proton shift and esters are observed and it is detailed in Table 4. Varying triplet and doublets found between 1.32 ppm and 2.82 ppm showed the presence of alkyl and alkine group with α-proton shift. Peaks with medium signals between 3.52ppm and 3.67 ppm indicates the presence of ethers. Phenomenal component ester is found between 3.81 ppm to 4.18 ppm, along with trace amounts of ethers. However, a strong signals between 4.58 ppm to 5.37 ppm ensures the presence of esters in major composition. Weak decilets signal are observed at frequent basis also confirms the presence of ester and ethers (Alan Aitken, 2016; Elenilson, 2016).

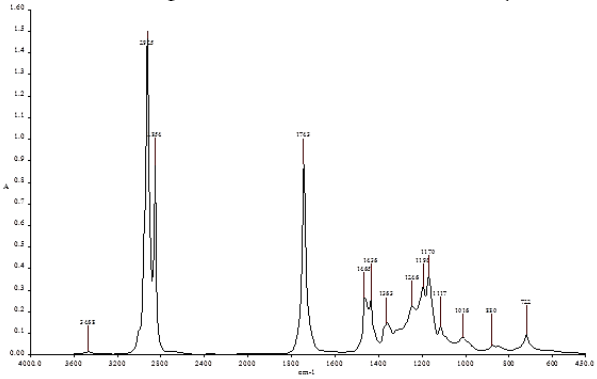

Figure.2. FT-IR spectrum of Palm kernel oil biodiesel

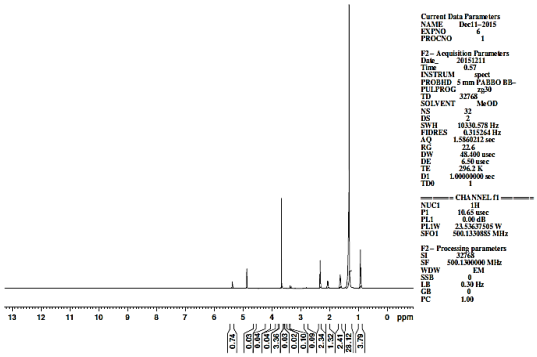

Figure.3. ^{1}H-NMR spectrum of Palm kernel oil biodiesel

Table.4.Palm kernel biodiesel spectrum of ^{1}H-NMR spectrum

PPM	Inferences of ^{1}H-NMR Spectrum	Signal Characteristic
0.92,0.93,0.94	CH=CH-CH_2-CH_3 Methyl H-shift, R-(C=O)-NH-CH_2-CH_3 Methyl H-shift, R_2N-CH_2-CH_3 Methyl H-shift, C-CH_3 Saturated alkyl.	Triplet
1.32	Ph-CH_2-CH_3 Methyl H-shift, RS-CH_2-CH_3 Methyl H-shift, Ph-O-CH_2-CH_3 Methyl H-shift, C-CH_2-C, C_2=CH-C (Methine).	Singlet
1.61,1.63,1.64	C=C-CH_3 Methyl H-shift, C_2=CH-C (Methine).	Triplet
2.06, 2.07	-C=C-C(R) =C-CH_3 Methyl H-shift, R-N=C-CH_3 Methyl H-shift, R-COO-CH_3 Methyl H-shift, C=_C-H.	Doublet
2.31,2.32,2.34	Ph-CH_3 Methyl H-shift, C=_C-H.	Triplet
2.79,2.80,2.82	R-(C=O)-NH-CH_3 Methyl H-shift, C=_C-H.	Triplet
3.33	R-O-CH_3 Methyl H-shift, R-O-CH (ethers).	Singlet
3.37	R-O-CH_3 Methyl H-shift, C(H)-OH (alcohols), R-O-CH (ethers).	Singlet
3.52	C(H)-OH (alcohols), R-O-CH (ethers).	Singlet
3.56,3.57,3.58	C(H)-OH (alcohols), R-O-CH (ethers).	Triplet
3.67	Ph-O-CH_3 Methyl H-shift, C(H)-OH (alcohols), R-O-CH (ethers).	Broad Singlet
3.81, 3.83, 3.84, 3.85	Ph-CO-O-CH_3 Methyl H-shift, C(H)-OH (alcohols), C(=O)-O-CH (esters), R-O-CH (ethers).	Quadruplet
Between 4.07 and 4.18	C(H)-OH (alcohols), C(=O)-O-CH (esters), R-O-CH (ethers).	Decilets
4.58	C(H)-OH (alcohols), C(=O)-O-CH (esters).	Singlet
4.87	C(=O)-O-CH (esters)	Strong Singlet
5.36,5.37, 5.37	R-CH=C-R (alkene)	Triplet

^{3}C-NMR: Typical ^{13}C-NMR technique spectrum for Palm kernel oil biodiesel is shown in Figure 4 and Table 5 shows the complete inferences of the spectral data. On analyzing the spectral data, signals found between 62.73ppm and 174.24 ppm indicates the presence of esters, which also inferenced with other spectral results of Proton NMR, GC-MS, FT-IR and based on this it can be concluded that the sample is trans-esterified biodiesel. In the resulting spectral data obtained signals for presence of methyl-amine group is found between 26.87ppm and 33.50ppm. A strong peak signal at 55.6ppm confirms the carboxylic group in the composition. Traces of alcohols

are found with respect to signals at 24.73ppm to 25.28ppm(Abraham Casas, 2012; Jahanshahi, 2016; Werstler 1986; Perry, 2000).

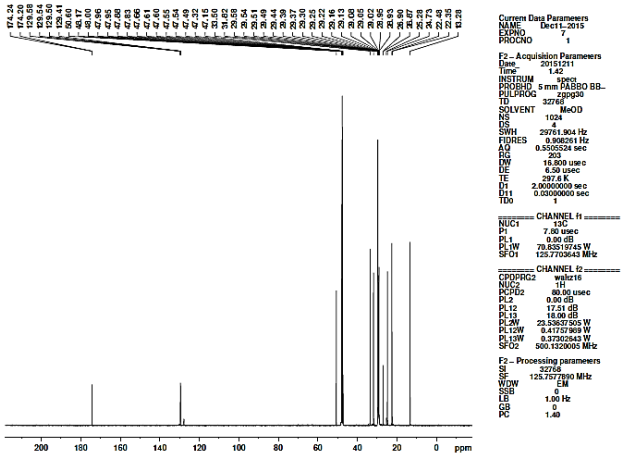

Figure.4.^{13}C-NMR spectrum of Palm kernel oil biodiesel

Table.5. Inferences of ^{13}C-NMR spectrum

PPM	Inferences of ^{13}C-NMR Spectrum	Signal Characteristic
13.28	C (Quarternary)	Singlet
22.35, 22.48	(H,R)-CH_3, (H,R)-CH_2-(H,R), C (Quarternary), CH_3-N.	Singlet
24.73, 25.28	(H,R)-CH_3, (H,R)-CH_2-(H,R), C (Quarternary), CH_3-N, Alcohol $(CH_3)_2COH$.	Singlet
Between 26.87 and 29.58	(H,R)-CH_3, (H,R)-CH_2-(H,R), C (Quarternary), CH_3-N.	Doublet, Triplet
31.82,33.50	(H,R)-CH_2-(H,R), (H,R)-CH-(H,R), C (Quarternary), CH_3-N.	Singlet
50.6	(H,R)-CH-(H,R), CH_3-O-.	Singlet
62.73	R-OH (alcohols), (=O)-O-CH_2-R (esters), Alcohol $(CH_3)_2COH$.	Singlet
65.09	R-OH (alcohols), (=O)-O-CH_2-R (esters), R-NO_2 (nitro).	Singlet
69.77	R-OH (alcohols), (=O)-O-CH_2-R (esters), R-O-R (ethers), R-NO_2 (nitro).	Singlet
Between 127.71 and 129.58	C=C, C-Aromatic, C-Heteroatomic.	Singlet, Triplet
174.20,174.24	C-Aromatic, C=O (acids), C=O (amides), C=O (esters).	Doublet

4. CONCLUSION

The present study is focused on extraction and characterization of biodiesel derived from Palm kernels and its suitability as a substitute for petroleum diesel. Based on the study, the following conclusions are drawn.

- Palm kernel oil extracted by mortar and pestle method yielded 445 grams of oil from 1kg of kernels. As the free fatty acid content is more than 8.5%, two stage transesterification is used with primary stage involving acid catalyzed transesterification using concentrated hydrochloric acid and methanol followed by base catalyzed transesterification with NaOH and methanol at a molar ratio of 1:6 which yielded 92.68% of biodiesel.
- Biodiesel is subjected to GC/MS spectral study which established the presence of 12 major methyl esters namely Undecylic acid, Tridecyclic acid, Pentadecyclic acid, Ginkgolic acid, Myristic acid, Oleic acid, Margaric acid, Gondoic acid, Arachidic acid, Behenic acid, Tricosanic acid and Lignoceric acid. Myristic acid (C14:0, RT 19.07) showed prominent presence.
- Further, the biodiesel is subjected to FT IR analysis which showed the presence of esters in major stretching areas between 1016 cm^{-1} to 1246 $cm^{-1}$and 1743 cm^{-1} with continuous, broad, strong-medium signal. At 2854 cm^{-1} and 2925 cm^{-1}, the presence of carboxylic derivative is also identified.
- ^{1}H NMR spectral analysis also supported the GC/MS data with the presence of esters by a strong signal between 4.58 ppm to 5.37 ppm. Weak decilets signal observed at frequent basis also confirms the presence of ester and ethers.^{13}C NMR spectrum study revealed astrong peak signal at 55.6ppm confirming the presence carboxylic group in the composition.

5. ACKNOWLEDGEMENTS

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