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Spectroscopic Characterization of Palm Stearin Biodiesel Derived Through Base Catalysed Transesterification Process

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ABSTRACT

In this research work, the characterization of the palm stearin biodiesel was made using Nuclear Magnetic resonance (NMR), Fourier transform infrared spectroscopy (FITR) and GC/MS methods. Analysis of the composition of fatty acids was done using the GCMS apparatus based on the retention time. Fourier transform infrared spectrometer was used for the spectrum analysis of the various functional groups and bands located in it. The properties of the palm stearin biodiesel were predicted adopting the American Society for Testing and Materials (ASTM) standards. Measured values of the properties were the density at 18°C as 0.88 g/m^3, kinematic viscosity at 35°C as 3.4 mm^2/s, the calorific value of the palm stearin as 37121 kJ/kg and the flash and fire points of the biodiesel as 130°C and 160°C respectively. The rapid and correct characterization of the palm stearin biodiesel was made by the NMR.

INTRODUCTION

The upsurge in the increase in the use of fuel and environmental awareness of pollution has created the urge in many researchers to take up the perfect biodiesel from feedstocks (Sun et al. 1999). Biodiesel was extracted from the feedstocks of waxes, and classified into two namely, animal waxes and plant waxes. Palm stearin wax was classified as plant wax as it was extracted from palm trees. Naturally, waxes have long chain esters of fatty acids, which were converted from long chain esters to fatty acid methyl esters with the transesterification reaction (Hariram et al. 2017, Gelbard et al. 1995).

Gas chromatography coupled with mass spectrometry is a versatile tool for the separation, quantification and identification of unknown organic components and permanent gases. Analysis of complex mixtures can be done by combining sensitivity and high resolving power (Hariram & Vasanthaseelan 2015). The information obtained can be used for the detection of impurities, contamination control and improvement, for example, of semiconductor management. Various fatty acids concentrations were determined from the GCMS graph and ion concentration (Pereira et al. 2002). They depict many components that include ketones, hydrocarbons, ions, fatty acids and esters. FTIR was absorbed in bands that identified the various functional groups of molecules such as OH stretch, C-O and OH bending, C-C stretching, C-O stretch, C-H stretching and C-C and C-O bending which was located based on the wavelength. FTIR stands for Fourier Transform Infrared Spectroscopy, the preferred method for Infrared spectroscopy.

Nuclear magnetic resonance (NMR) spectroscopy is a powerful analytical technique used in the characterization of organic molecules through the identification of carbon-hydrogen frameworks within the molecules. It determines the physical and chemical properties of atoms or the molecules in which they are contained. NMR has two classifications of variations such as C-NMR and H-NMR used in the characterization of organic structures (Hariram et al. 2017). H-NMR is used in the determination of the number of hydrogen atoms present in the molecules and C-NMR represented in the determination of the number of carbon atoms present in the molecules. NMR is used in the identification of the blending levels in biodiesel blends. This paper gives the hydrogen and carbon atoms present in the palm stearin biodiesel using the NMR spectroscopy method. The focus of the paper is on optimization of various properties of palm stearin biodiesel such as flash point, fire point kinematic viscosity, calorific value, density and iodine value determined and compared with the properties of diesel. The various properties of palm stearin biodiesel are seen in ASTM standards of ASTM D6751-07b and European standards (EN 14214).

Bharathwaaj et al. (2018) have carried out extraction of biodiesel from the species of mellifera waxes. Long chain fatty alcohols are present in the bee wax. Biodiesel determines the properties that include: density of 880 kJ/m^3 and calorific value of 35.5 MJ/kg and the cetane number of 48. Arun Shankar et al. (2017) have indicated cottonseed biodiesel as produced from cottonseed oil using the transesterification process. Cottonseed biodiesel was characterized using the GC-MS and FTIR to find out the functional groups present in the biodiesel. The properties of biodiesel such as calorific value (36.18 MJ/kg, 33.78 MJ/kg), flash point (160^{o}C) and kinematic viscosity and density have been predicted. Ng & Yung (2019) have characterized palm oil biodiesel and its blends. NMR was employed for accurate characterization of the oil and its blends. Folayan & Anawe (2019) have done extraction of the argan oil from its kernel. Fatty acid content was employed using the GCMS results. The measured properties of the argan biodiesel were in accordance with the American Society for Testing Methods standards. Eman et al. (2015) have carried out extraction of green algae (*Spirogyra longata*) which was characterized using GC-MS. In GC-MS apparatus, helium gas was employed as a carrier gas with the employment of impact ionization mode. GC graphs show the presence of valuable components such as ketones, phenolic, hydrocarbons. Fatty acids were defined using FTIR spectrum. Naureen et al. (2015) have done biodiesel extraction from sunflower oil used in the prediction of the characteristics of biodiesel using the GC-MS, FTIR and NMR. GCMS results indicated, the presence of seven saturated, one polyunsaturated and three monounsaturated fatty acid methyl esters in the biodiesel.

Monteiro et al. (2009) saw the NMR results of soybean and castor oils biodiesel which had a complex chemical composition, NMR helped prediction of the chemical components in biodiesel mentioned above. Knothe (2001a) reported numerous studies on biodiesel formation and its characterization such as GC/MS, NMR and HPLC methodology which showcased a non-linear behaviour in their corresponding FTIR spectra. Knothe (2001b) have used NMR spectroscopy to predict the vegetable oils and miscible oil with convenient diesel fuel. H-NMR was employed for the determination of the biodiesel fuel quality. Knothe (1999) did the exploration of the chemical structures predicted by the dominant technique that employed the nuclear magnetic resonance spectrometry. Shimamoto et al. (2017) have done characterization of soybean and castor oil. Monterio et al. (2009) have used H-NMR spectroscopy for the characterization of the quality of the biodiesel-diesel blend. H-NMR was used in the making of a higher concentration not exceeding 2% of biodiesel concentration. Portela et al. (2016) evaluated biodiesel content in the diesel fluid using the H-NMR spectroscopy method. In this process, the multivariable regression model was generated by the use of partial least square (PLS).

In the current work, palm stearin biodiesel produced from palm stearin wax was characterized using NMR, FTIR and GC-MS. The molecular structure was predicted using GC-MS and compared with the NIST library. Characterization of the palm stearin biodiesel was done using FTIR for optimization of the functional groups present in biodiesel. NMR spectroscopy was used in the characterization of the biodiesel, it depicted the carbon and hydrogen groups presented in the biodiesel. In the present study, the properties of palm stearin biodiesel such as density, kinematic viscosity, flash point, fire point, calorific value were investigated through different experiments.

MATERIALS AND METHODS

Palm stearin biodiesel was produced in the laboratory using the palm stearin wax from the transesterification process. Techniques such as NMR, FTIR and GCMS were used for the characterization of palm stearin biodiesel.

Transesterification Process

Palm stearin bio-oil was produced by heating the palm stearin wax to 60^{o}C. In the single-stage transesterification process, the FFA content was below 2%. The FFA content of the bio-oil was 0.3% which was satisfactory, the single-stage transesterification process was employed in this process. The main factors were methanol to oil molar ratio, catalyst concentration and reaction duration which had a significant role in the process. A three-necked 100 mL flask containing palm stearin with reflux container was used in this process. The temperature and speed of the magnetic stirrer were set at 60^{o}C and 600rpm respectively. Initially, bio-oil was heated to 90^{o}C for removing the moisture content in the bio-oil. An appropriate amount of methanol and a catalyst were taken for 100 mL bio-oil. Sodium methoxide solution was produced by mixing NaOH and methanol at 60^{o}C at the speed of 600 rpm, it was mixed with the bio-oil to initiate the process. A magnetic stirrer was employed in this process for around 60 min for steering at a constant speed as shown in Fig. 1. The process took 60 minutes for the layer separation of glycerol and biodiesel. A clear separation was taken 12 hours. Biodiesel was cleaned using distilled water several times to remove the moisture content. After the transesterification process, the biodiesel was heated to 100^{o}C to remove the water content and the excess methanol was present in the biodiesel.

Gas Chromatography and Mass Spectrometry (GC-MS)

GC-MS was used for the characterization of the organic components. This technique was performed using the gas

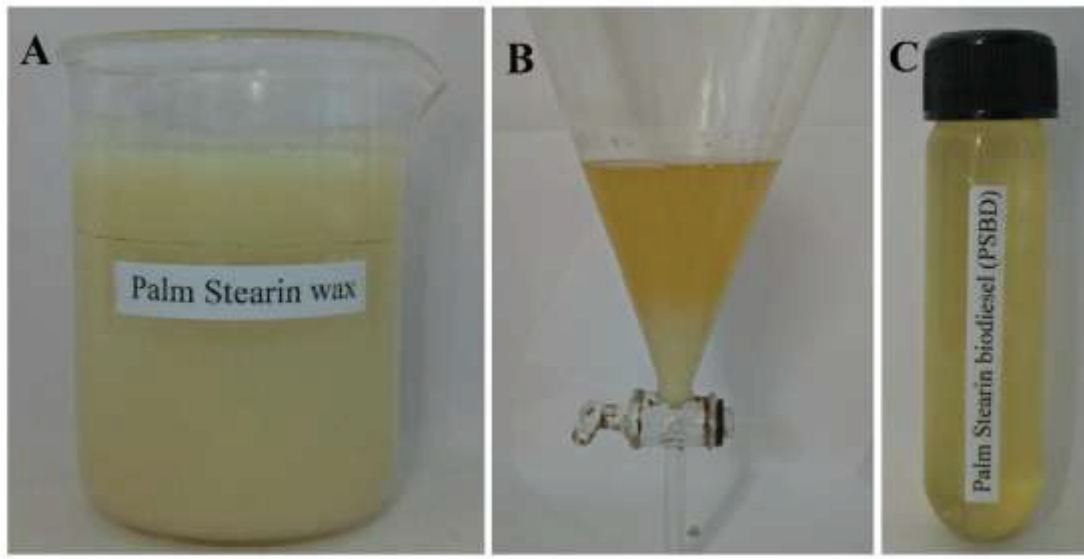

Fig.1: Transesterification process of palm stearin bio-wax.

chromatography and mass spectrometry to identify the samples. With the help of GC-MS, samples were distinguished into the individual compound by the temperature controlled capillary column. Associating and measuring the fragmentation of the unique mass spectrum (m/z) with NIST library, mass spectrum delivered the structural formula and molecular weight. GC-MS was used for the measurement of the molar mass and structural analysis of small biomolecules. In the present work, the JEOL GCMATE 2 GCMS with data system with the maximum resolution of 6000 Daltons with the sources of electron impact (EI), Chemical Ionization (CI) and fast atom bombardment (FAB) were employed. GCMS was employed for finding out the saturated and unsaturated fatty acids determined from retention time and ions.

Fourier Transform Infra-Red Spectrometry (FTIR)

With the help of the infra-red spectrometry, the fragmentation of samples in the infra-red region of the electromagnetic spectrum was absorbed. FTIR was used for the evaluation of the vibrations of organic components such as starching vibrations and bending vibrations of C-H, C-O, C=O component existing in the molecules. The type of the bond was determined by wavelengths of the radiation. FTIR spectrum was used in the estimation of some cases such as chemicals, pharmaceuticals, petroleum products, resins, etc. The Perkin Elmer system one FTIR/ATR instrument was used in this study. It has a scan range of 450-4000 cm^{-1} and resolution of 1.0 cm^{-1} and 50 mg sample was required to find out the results.

Nuclear Magnetic Resonance Spectroscopy (NMR)

Nuclear Magnetic Resonance spectroscopy is a very powerful non-invasive technique that provides detailed structural information relating to various molecular systems. Avance 3500 MHz NMR was employed in this study. NMR is a spectroscopy technique based on the absorption of electromagnetic radiation in the radio frequency regions 4 to 900 MHz by nuclei of the atom. But in the present work, the field strength of the NMR was 500 MHz. The $CDCl_3$ solvent dissolved in the samples in 1:1 ratio.

RESULTS AND DISCUSSION

Gas Chromatography and Mass Spectrometry

GCMS was used for carrying out the experiments to find out the various methyl esters present in the palm stearin biodiesel (Fig. 2). Methyl esters were predicted based on the retention time compound with the NIST library. There are ten various fragmentation patterns developed from the GCMS apparatus which helped determination of the methyl esters structure as tabulated in Table 1. The various retention times were predicted from RT 13.18 min to RT 26.68 min of gas chromatogram operation. Fig. 3A depicts the presence of dodecanoic acid, methyl esters with the highest peak mass ions 214 Daltons at a retention time of 13.18 min. Molecular structure was found to be methyl esters of lauric acid, with a concentration of 0.1%.

Fig. 3B presents the retention time of 15.75 min which had ester of methyl tetradecanoate. The highest mass to

change ratio was predicted at 242 Daltons, myristic acid was adopted as a fatty acid methyl ester with a concentration of 22.90%. Fig. 3C shows the pentadecanoic acid, 14, methyl esters fragmentation pattern at the reaction time of 18.43 with the highest value of peak mass ions of 270 Daltons which was present at the scan of 695 Daltons. Pentadecylic acid was shown as FAME which had a percentage of 25.8%. Fig. 3D portrays the retention time of 19.87 min with the fragmentation pattern of hexadecanoic acid, methyl esters. Palmitic acid was found to be a methyl ester. Methyl esters had a peak mass value of 270 Daltons located at scan 752. The presence of stearic acid is shown in Figs. 3E and 3F at

Table 1: Composition of FAME's in palm stearin biodiesel.

S. No	Retention time (min)	Name of the esters	Fatty acid methyl esters	% Conc.
1	13.18	Dodecanoic acid, methyl ester	Lauric acid	0.19
2	15.78	Methyl tetra deaconate	Myristic acid	22.09
3	18.43	Pentadecanoic acid, 14 -methyl, methyl ester	Penta-de-cyclic acid	23.71
4	19.87	Hexadecanoic acid, methyl ester	Palmitic acid	28.36
5	20.33	9 - Octadecanoic acid (2) - Methyl ester	Stearic acid	0.34
6	21.93	13,16 -Octadecadienoic acid, methyl ester	Stearic acid	0.15
7	22.53	11 - Eicosanoic acid, methyl ester	Arachidic acid	22.04
8	22.77	Eicosanoic acid, methyl ester	Arachidic acid	1.40
9	24.3	Decosanoic acid Methyl ester	Behenic acid	0.62
10	26.68	Octanoic acid, heptadecylic ester	Caprylic acid	1.10

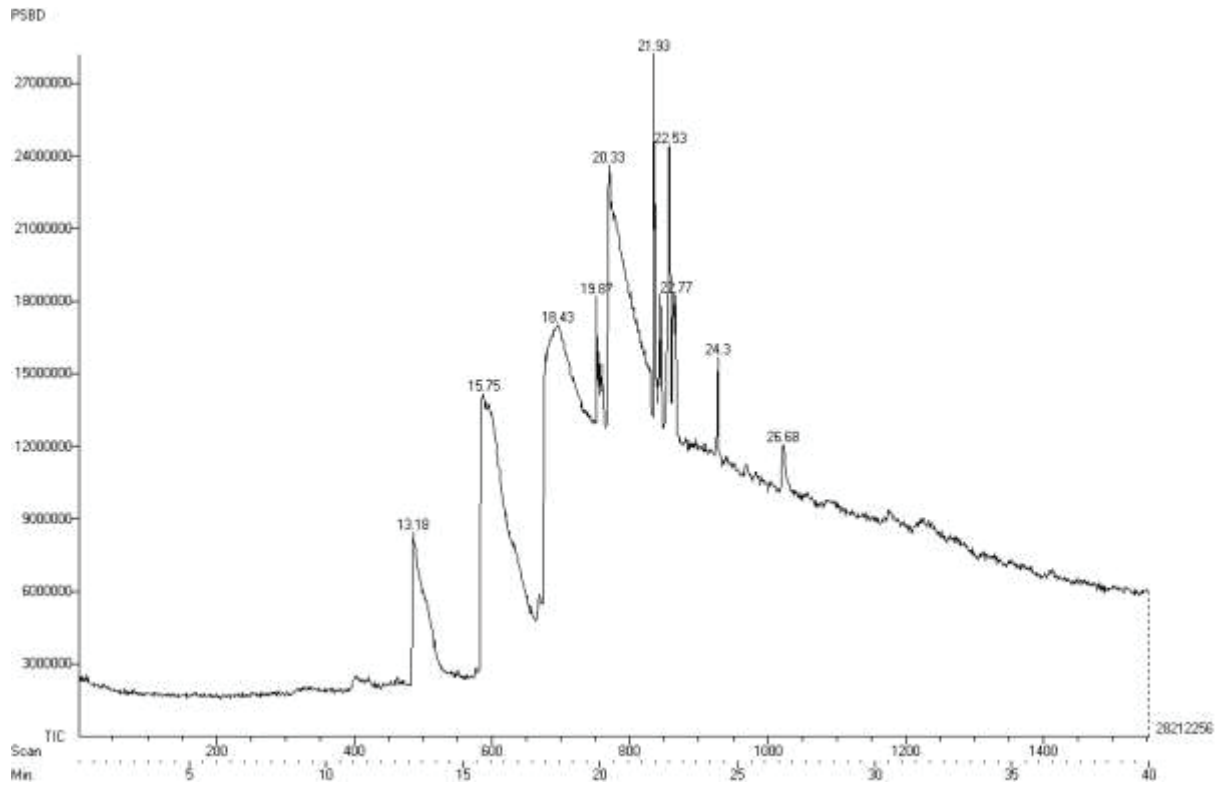

Fig. 2: GCMS chromatogram of palm stearin biodiesel.

the retention time of 20.33 min and 21.93 min which had a 9-octadecanoic acid and 13, 16 octadecadienoic acid, methyl ester. 300 Daltons of highest peak mass value was predicted from the fragmentation patterns of fatty acids. Fig. 3G depicts the 11, eicosanoic acid and eiosanoic acid esters at the retention times of 22.53 min and 22.77 min which had the highest peak value such as 324 Daltons and 326 Daltons respectively. Molecular structure of arachidic

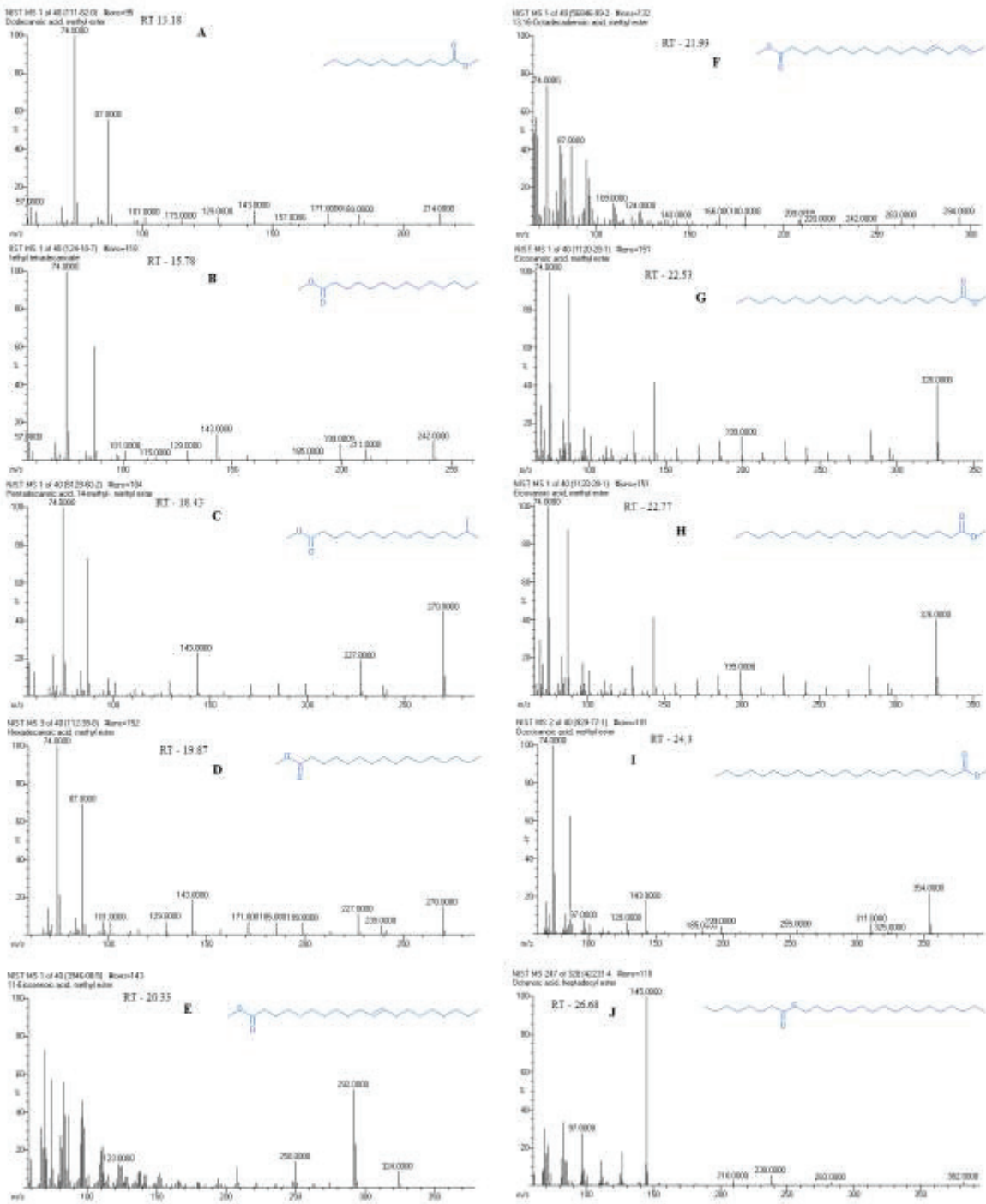

Fig. 3: Fragmentation pattern of palm stearin biodiesel.

acid was predicted from the fragmentation pattern (Hariram & Bharathwaaj 2015).

Fig. 3 presents the fragmentation pattern of the decosanoic acid, methyl esters at the retention time of 24.3 min with a structure of methyl esters to be behenic acid with the concentration of 0.6%, the highest peak value was predicted from the fragmentation pattern as 354 Daltons. Fig. 3J shows the octanoic acid, heptadecyl esters, which was present in the fragmentation pattern at the retention time of 26.68 min with the scan of 1023. Caprylic acid is a general fatty acid methyl ester of these esters which have a low value in concentration. 382 Daltons was predicted as the peak mass value of methyl esters. Finally, the palmitic acid and arachidic acid had a higher concentration in palm stearin biodiesel. Palmitic acid had a high value in the palm stearin biodiesel at the retention time of 19.87 min and 20.33 min.

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform Infrared spectroscopy (FTIR) was used for the prediction of the alkyl groups, ester ketones and alcohol group from the wavelengths. Fig. 4 shows the organic and inorganic compounds present in the palm stearin biodiesel. FTIR braced the bonds and steeling such as C-O stretch, OH stretch and C=0, C-H bonds. In the present work, FTIR was adopted for carrying out the experiments of palm stearin biodiesel. The Perkin ELMER system one FTIR spectroscopy was employed for finding the stretching and vibrations of various natural materials. FTIR was used in the determination of the organic and inorganic stretching vibrations and bonds present in palm stearin biodiesel. A graph has been generated by an instrument of FTIR which indicated the bonds according to wavelength in units of cm^{-1} and the transmittance as percentage placed in Y-axis.

Many vibrations have been created from the FTIR, practiced from 584.82 cm^{-1} to 2922.02 cm^{-1}. Fig. 4 depicts the presence of carboxylic acid and derivatives in O-H stretch present in the peak of 2922.02 cm^{-1} and another peak value of 1741.78 cm^{-1} which was present in the C=0 (saturated aldehyde) that was in aldehydes and ketone groups. The peak value of 1438 cm^{-1} revealed the presence of (α-CH_2 bending) aldehydes and ketones. α-CH_2 bending was present in the graph which shows in the peak value of 1260.66 cm^{-1} that is also in aldehydes and ketones. Some peak value was present in the same functional group which was in carboxylic acids and derivatives such as 1195.53 cm^{-1} which was indicated in the (0-C) (2 bands).

The peak value of 1242.33 cm^{-1} indicated the 0-C bands (sometimes 2 peaks) in carboxylic acids and derivatives. The

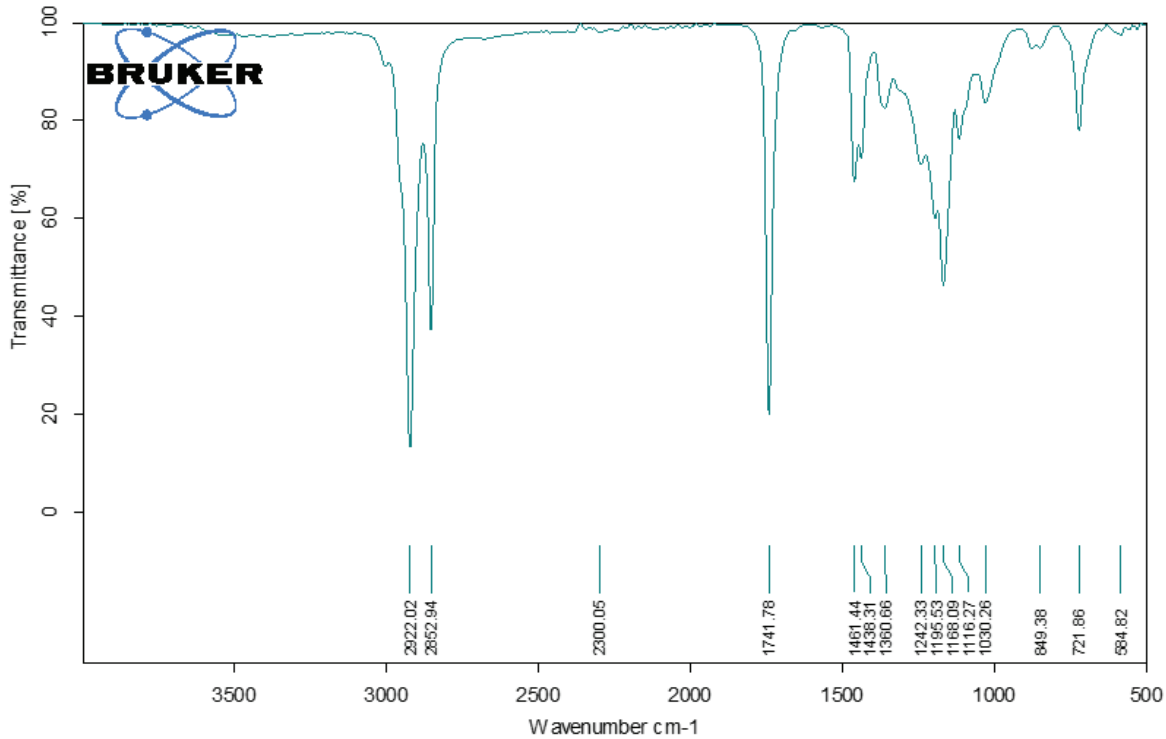

Fig. 4: FTIR spectrum of palm stearin biodiesel.

alkenes group was predicted from the peak value of 849.38 cm^{-1} and 721.86 cm^{-1} influenced in the process which had a functional group of alkenes indicating the CIS-RCH+CHR. The detailed groups of organic compounds and then stretchings and vibrations located in the FTIR spectrum as shown in Fig. 4.

Nuclear Magnetic Resonance (NMR)

Palm stearin wax is the feedstock for producing palm stearin biodiesel (PSBD). PABD mainly consists of stearin and oleins. The ^{13}C and ^{1}H NMR spectrum of PSBD have resemblance as shown in Fig. 5 and Fig. 6 respectively. The NMR spectrum (^{13}C and ^{1}H) of palm stearin wax and its biodiesel were compared. The prominent proton signal at 4.1-4.5 ppm with multiplets, multiplet peaks between 5.2 and 5.5 ppm and singlet peaks at 7.0 and 7.3 ppm of the palm stearin wax were no longer visible in the ^{1}H NMR. Similarly, singlet and multiplet peaks between 65 and 80 ppm of the palm stearin wax were no longer seen in the palm stearin biodiesel. These shifting of peaks indicate the efficiency of transesterification in the formation of fatty acid methyl esters. The palm stearin biodiesel which is the resultant product of transesterification, the palm stearin wax along with methanol reveals the presence of olefinic hydrogen atoms and methoxyl hydrogen atoms as singlet and multiplets peaks (Simpson 2012).

OCH_3, singlet peaks at 3.67 ppm and 3.49 ppm confirm the presence of methoxyl hydrogens, whereas multiplet peaks between 5.25 and 5.36 ppm confirm the presets of olefinic hydrogen. The singlet peak was seen at 7.25 ppm of H-NMR indicating the presence of multiple multiplet peaks between 0.8 and 2.4 ppm indicating the presence of aliphatic hydrogens. The absence of the peak between 4.5 and 5 ppm in the ^{1}H NMR along with the existence of olefinic hydrogens indicate the feedstock used in the transesterification process as having a vegetable based origin. The olefinic and methoxyl hydrogen signals of palm stearin wax and its biodiesel were integrated and calibrated. The existence of a linear relationship between the methoxyl and olefinic hydrogen atoms was observed. This relationship was also confirmed by the FTIR studies (Hariram 2019).

Intrinsic Characteristics of Properties of Palm Stearin Biodiesel

The properties of palm stearin biodiesel such as kinematic viscosity, flash point, fire point, calorific value and iodine

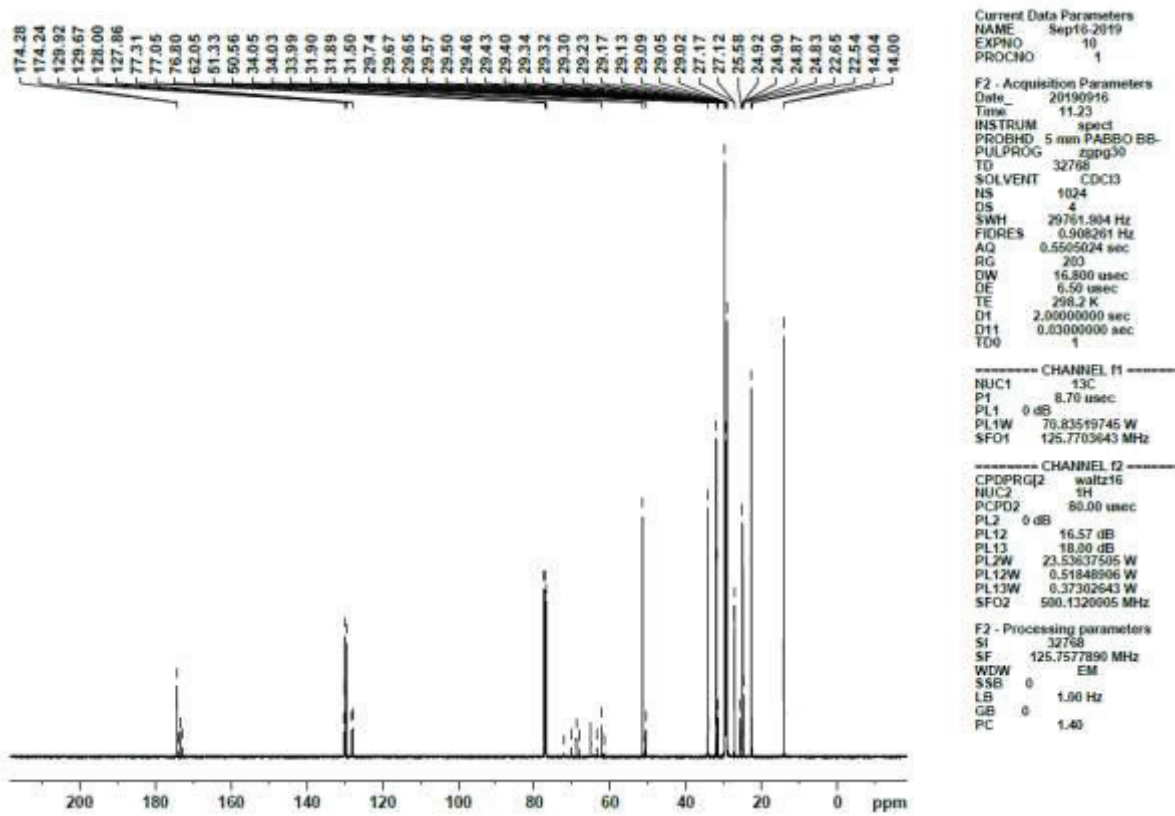

Fig. 5: ^{13}C NMR spectrum of palm stearin biodiesel.

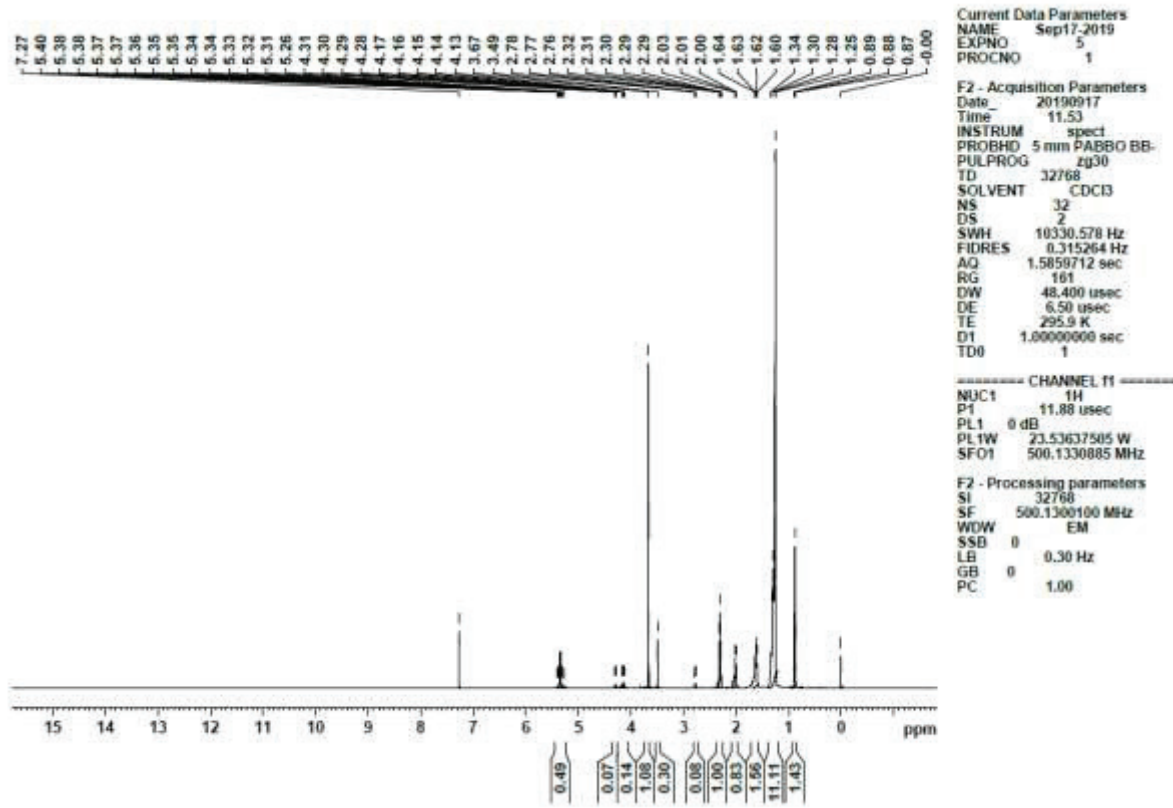

Fig. 6: ^{1}H NMR spectrum of palm stearin biodiesel.

value were characterized using ASTM standard methods as indicated in Table 2.

Kinematic viscosity is the main factor which influences the compression process. It has old and hot kinematic viscosities that affect the engine. Palm stearin bio-oil has a high kinematic viscosity ranges which do not fit the combustion process, that affect the entire engine setup. Palm stearin biodiesel, which had a significant kinematic range fitted for ASTM standards and European standards was produced following the transesterification process. Palm stearin biodiesel has a kinematic viscosity at 35°C of 2.4mm^2/s, which is in ASTM standards among 1.9-6.0 mm^2/s ASTM D6751 and EN14214 were placed in the fuel properties. Flashpoint is the lowest temperature at which the fuel is flashable in the presence of air. Generally, diesel has a flashpoint of 47°C. Palm stearin biodiesel has a flashpoint of 244°C which is in correct proportion compared with the ASTM standard limits. In comparison, the flashpoint of the diesel and palm stearin biodiesel, diesel has a low flashpoint temperature which fluids use in easy combustion. Palm stearin has a high flashpoint temperature and takes a long time to ignition leading to ignition delay. Minimum

Table 2: Physio-chemical properties of PSBD and mineral diesel.

Properties	PSBD	Diesel
Density @ 18°C (g/m^3)	0.88	0.8200
Kinetic viscosity @ 35°C (mm^2/s)	2.4	2.5
Calorific value (kJ/kg)	37121	42957
Flashpoint	244	47
Fire point	166	140

flashpoint regions are used for easy combustion and proper safety (Hariram 2016).

The fire point is one of the factors of characterization of the fuels. Fire points of the palm stearin biodiesel are predicted using the fire point apparatus which has a value of 166°C temperature compared to the value of 140°C related to diesel. They had similar values of fire point. Calorific value is defined by the amount of energy released from the fuels with the help of the combustion. Palm stearin biodiesel has a calorific value of 37121 kJ/kg related to the value of diesel as 42957 kJ/kg. The physio-chemical properties of the palm stearin biodiesel were found to be within ASTM standards. Ignition delay is identified when the calorific value is low. High calorific value improves engine efficiency. Calorific value is employed as the main factor in the process of combustion. Density was predicted from the experiments made in the laboratory. Palm stearin has a density of 0.88 g/m^3 at 18°C which is in line with ASTM standards. Diesel has a density value of 0.8200 g/m^3. Density is also a main factor in the combustion process which influences engine performance (Pimental et al. 2006, Demirbas 1997).

CONCLUSION

This article has discussed the characterization of the palm stearin biodiesel. GC-MS and FTIR spectrometer have proved the ten fatty acids in the palm stearin biodiesel and the C-H stretch, C-O stretch and organic components present in biodiesel. Gas chromatography and mass spectrometry results indicate the palmitic acid and myristic acid having the main role in palm stearin biodiesel. The GC-MS, FTIR and NMR results show the proof of the palm stearin biodiesel of the appropriate biodiesel. Kinematic viscosity of the palm stearin biodiesel to be 3.4 mm^2/s at 35°C and the density at 18°C be 0.88 g/m^3 were seen as the properties of biodiesel. The calorific value of the palm stearin biodiesel was 37,121 kJ/kg. The flash and fire points predicted using the apparatus were 130°C and 166°C. All the physio-chemical properties of palm stearin biodiesel satisfied and were in accordance with the ASTM standard limits. So, the palm stearin biodiesel is suitable for normal compression ignition engines as an alternative fuel without any major modifications.

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