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Thermal Degradation and Spectroscopic study of Neat Palm Oil, Waste Palm Oil, and Waste Palm Oil Methyl Ester

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Abstract. Research into finding an affordable, renewable, biodegradable and environmentally friendly alternative fuel to power compression ignition engine has been intensified in the past few decades. The application of waste palm oil (WPO) as feedstock for biodiesel has gained traction and has led to a decline in the production cost of biodiesel, and prevents the contamination of aquatic and terrestrial habitats. The present effort is geared towards characterization of neat palm oil (NPO), WPO and waste palm oil methyl ester (WPOME) through Thermogravimetric analysis (TGA), Derivative thermogravimetric (DTG), and Fourier Transform Infrared Spectroscopy (FTIR). Samples of NPO and WPO were collected from takeaway outlets. The WPO was converted into WPOME by transesterification. The outcome of DTG analysis showed that the vaporization of NPO, WPO, and WPOME occurred between 320 ^{o}C and 480 ^{o}C; 320 ^{o}C and 470 ^{o}C, and 27 ^{o}C and 103 ^{o}C respectively. The TGA analysis revealed that NPO, WPO, and WPOME exhibited similar thermal degradation trend and lost 98 %, 29 %, and 63 % of their weight respectively, during the one stage of thermal degradation. The spectra curve for NPO and WPO followed the same trend and achieve peaks at nearly the same wavelength of 1743 cm^{-1} while the peculiar peak of the spectra curve for WPOME occurred at 1022 cm^{-1}. In conclusion, this work reinforces that usage and transesterification alters the thermal degradation and spectroscopic pattern of palm oil.

1. Introduction

The use of petroleum-based diesel fuel (PBDF) to power compression ignition (CI) engines has come with an attendant cost, engine performance, and environmental challenges. This scary scenario has necessitated the need for more interest and research in biodiesel, an important member of the biofuel family, as an alternative to PBDF. Despite some shortcomings, including the high cost of feedstock, low energy conversion, degradation during transportation and storage, biodiesel offers obvious advantages in the world's quest to meet its energy needs [1, 2]. Biodiesel, commonly referred to as fatty acid methyl ester (FAME), is generated by transesterification of feedstocks, mainly vegetable oils (edible or inedible), animal fats, etc. using alcohol (methanol or ethanol) with or without a catalyst [3]. Despite its renewability, low sulfur content, safer handling, higher cetane number, etc., large-scale production and application of FAME has been hampered by the high cost of feedstock, food versus fuel debate and the long time required to cultivate inedible vegetable feedstocks e.g 3- 4 years for palm tree to bear fruit [4], and 2 - 3 years for moringa tree to bear fruit [5]. When compared to PBDF, application, and combustion of FAME in CI engines are more environmental friendly as it generates less carbon monoxide (CO), between 75 % and 90 % reduction in unburnt hydrocarbon (UHC) and fewer polycyclic aromatic hydrocarbons (PAHs) [6]. Economically, it costs about US$0.35 to produce a liter of PBDF from fossil fuel compared to about US$0.5 to produce a liter of FAME, with raw materials accounting for most of the cost [7]. The feedstock is believed to account for between 70 % and 95 % of the cost of FAME production [8, 9]. One of the strategies to make the commercial production of FAME attainable and affordable is the adoption of waste cooking oil (WCO) as feedstock. The initial hurdles in collection logistics and infrastructure pointed out by Janauna and Ellis [10] and Atadashi et al. [11] are being overcome by partnering with operators of fast food, takeaway, and restaurants.

Palm oil is gotten from an oil palm tree (*Elaeis guineensis*). It is generally regarded as the world's most versatile and most widely used vegetable oil. Global production of palm oil rose from about 15.2 million tonnes in 1995 to an excess of 62.6 million tonnes in 2015 and has been projected to attain 240 million tonnes by 2050. Malaysia, Indonesia, and Nigeria top the 43 palm oil growing countries globally. Production and domestic use of palm oil by some African countries between 2013 and 2018 are shown in figure 1 [12]. Application of palm oil includes excellent frying properties at a cheaper cost compared with cottonseed or sunflower oils, a good material for packaging baked foods, raw materials for shampoo, liquid soap, detergent, paints, cosmetics, inks, lacquers, as preservatives in processed foods and an adhesive for binding fibreboards [13].

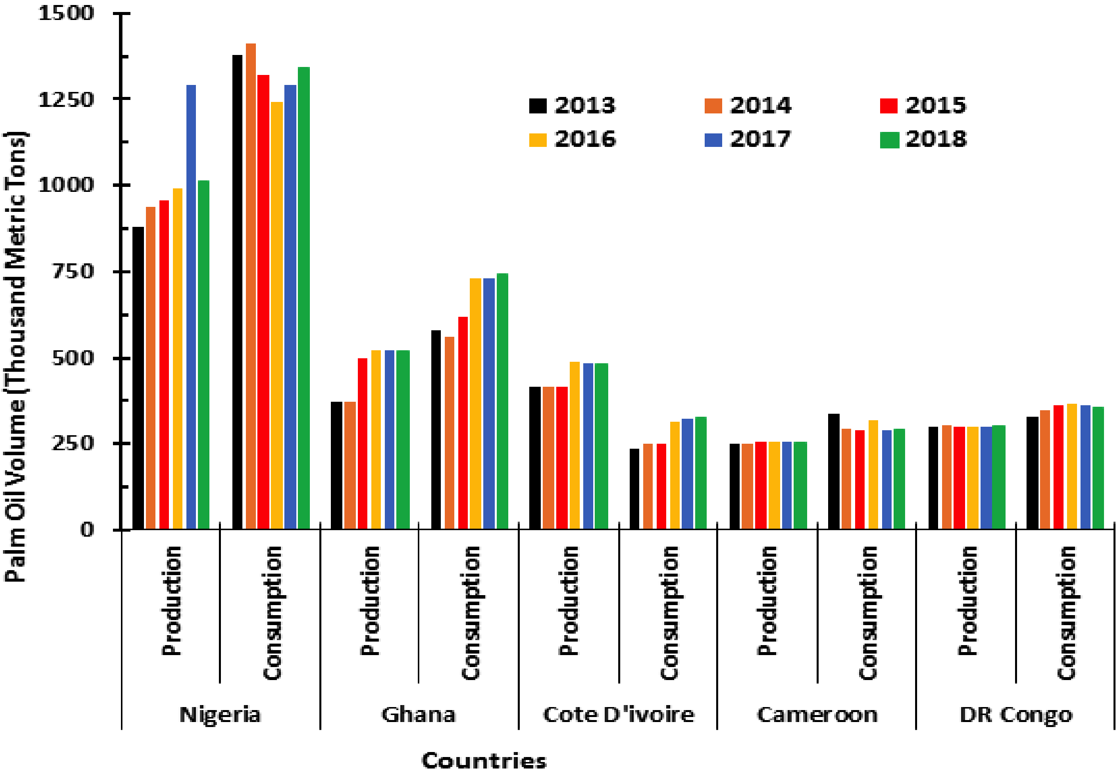

Figure 1. Production and domestic consumption of palm oil in some African countries (Thousand metric tonnes). Adapted from [12]

Due to population explosion, urbanization, and change in lifestyles, the consumption of palm oil, particularly in households, restaurants and fast food outlets for cooking and frying, has continued to rise in recent years with palm oil accounting for the greatest percentage of 34 % and 33 % in production and consumption of all the edible oils respectively. Application of palm oil is, however, not limited to cooking and frying but include lubrication, grease, cosmetics, animal feed, glycerol, polyurethane and alkyl resins which are raw materials for production of paints, inks, lacquers, varnishes and enamels as well as other socio-cultural purposes thereby providing income and employment in the entire value chain. Sadly, growing oil palm is credited to contribute to deforestation with damaging effects on wildlife and forests. Inappropriate disposal of the waste palm oil (WPO) has created problems by blocking the sewer pipes, corrode metals and structural problems by weakening concrete structures [14, 15].

During frying, the oil is heated repeatedly to between 170 ^{O}C and 220 ^{O}C in the presence of oxygen, and sometimes moisture, which causes the palm oil to be exposed to physical, thermal and chemical degradations. Thermal degradation also predisposes the palm oil as a suitable candidate for FAME production by various techniques including pyrolysis, dilution, microemulsion, and transesterification [16, 17]. In an attempt to gain more insight into this feedstock, a lot of investigation are being conducted for its characterization using various non-destructive techniques.

Thermogravimetric analysis (TGA) is a technique for comparing the mass of a sample against temperature or time when the sample is exposed to thermal decomposition through a change in temperature or heated. The analytical technique measures the change in mass of samples during oxidation, reduction, sublimation, decomposition, or vaporization. TGA is usually conducted in a controlled environment. However, outcomes of TGA can be affected by heating rate, buoyancy, and thermal expansion, sample-furnace-sensor arrangement, condensation and reaction, the thermal conductivity of the sample, atmospheric turbulence, etc. [18]. Fourier Transform Infrared (FTIR) spectroscopy is a non-destructive analytical method that uses ultraviolet light to identify the chemical properties of the sample. FTIR has been widely used for the detection, classification, authentication, and categorization of contaminations, additives, fats and oils, fibers, powders, solids, films, polymers, etc. The major advantage of using FTIR include its wavelength precision and competency to quantify spectra with signal-to-noise ratio [19, 20]. The combination of TGA)/DTG and FTIR has been found useful in the evaluation of thermal degradation and oxidative stability characteristics of the physicochemical properties of the samples. TGA/DTG shows weight loss and derivative weight change against temperature.

Over the last decade, a few researchers have characterized either unused vegetable oil or used vegetable oil with a view at comparing the effect of usage on their structure, properties, and degree of saturation. Ullah et al. [21] carried out the characterization of used and un-used palm cooking oil using TGA/DTG, and FTIR to unravel the effect of usage on saturation and properties of the oil. They reported no significant spectroscopic and structural changes in the sample, though there was a remarkable difference in the TGA spectra. Li et al [22] investigated thermal degradation of peanut oil and peanut biodiesel using TGA and FTIR. They observed similarities in their spectra which imply the existence of matching chemical groups in the chemical composition, though the discovery of CH_3 axial deformation in $O{-}CH_3$ at 1435.6 cm^{-1} in peanut biodiesel was not noticed in peanut oil. The TGA revealed similar thermal degradation narratives throughout the process though, the two samples were found to be thermally stable at different temperature and varying rate.

Similarly, Elkady et al. [23] researched the characterization of waste vegetable oil and its biodiesel using TGA and FTIR techniques. The differences in the FTIR spectra were attributed to the alteration, during transesterification, of the ester groups in the used vegetable oil into methyl esters in the biodiesel [24]. The TGA showed different commencement and end of temperatures for the thermal degradation, different degradation temperature ranges and percentage weight loss for the samples. Rabelo et al. [25] employed FTIR for the characterization of neat soybean oil and FAME produced from soybean oil. The slight divergences in the position of the peaks in their profile were attributed to the effect of the transesterification process. The difference in their spectrum was noticed in the fingerprint section between 1500 cm^{-1} and 900 cm^{-1}. Also, Matwijczuk et al. [26] engaged the use of FTIR to characterize sunflower oil used to fry French fries, sunflower oil used to fry fish and commercial unblended FAME. The FTIR spectrum revealed a clear difference in the band shapes of the profile of the waste sunflower oil within range of 1770-1670 cm^{-1} due to the effect of the items fried with the oil. In the case of the spectrum for commercial B100 FAME, it was found to be least intensive as a result of the effect of the carbonyl C=C vibration.

With the advancement in research in the production and use of FAME in the last few decades, there is a need to investigate thermal degradation trajectories along the value chain of biodiesel from neat vegetable oil, used vegetable oil to biodiesel produced from the used vegetable oil with a view to engendering better understanding on the quality, behavior, and storage of FAME and its various feedstocks. This current effort aims at the characterization of the neat palm oil (NPO), waste palm oil (WPO) samples that were used to fry different food items and the biodiesel generated from the waste palm oil samples to deepen the information available in the literature. Relevant questions to be answered through this investigation include; what are the effects of thermal degradation on neat palm oil (NPO), waste palm oil (WPO), and waste palm oil methyl ester (WPOME). This research is limited to the comparison of TGA, DTG and FTIR analysis of NPO, WPO and WPOME with a view for better understanding of this aspect of FAME and its feedstock.

2. Materials and Experimental work

The materials used for this research include neat palm oil, waste palm oil samples, biodiesel generated from the waste palm oil samples, chemicals, reagents, and glassware.

2.1. Materials collection and treatment

Neat palm oil and waste palm oil samples were collected from the local owner-operated restaurant at Durban central, KwaZulu Natal province, Republic of South Africa. The waste palm oil has been used for repeatedly for 14 days to fry various food items including fish, and chips at the point of disposal. The cycle of usage and the frying temperature could, however, not be ascertained. Waste chicken eggshells were obtained from the University of KwaZulu Natal, Durban cafeteria. The waste chicken eggshells were converted to CaO catalyst through high-temperature calcination at 900 ^{o}C as earlier reported [27].

The WPO was heated to 110 ^{o}C to remove moisture, allowed to cool to about 70 ^{o}C, where the sample is still in liquid form, and filtered by using laboratory sieve to remove any food debris and other particles trapped in the samples. The acid value of the clean WPO sample was determined to know its suitability for the transesterification process while the remaining sample was kept in glass bottles to prevent contamination.

Methanol (analytical grade, 99.5 %; Merck, South Africa, Univar) was used as alcohol. Magnesol® (analytical grade, 60-100 mesh, the molar weight of 100.39 g/mol, Sigma-Aldrich, Germany) was used as an adsorbent. Magnesol® becomes is hygroscopic once the package is opened. It was therefore carefully re-sealed as tight as possible and contact with eyes was prevented.

2.2. Experimental work

The experimental work consists of the conversion of the WPO to biodiesel via transesterification and the characterization of the samples, namely NPO, WPO, and WPOME, by TGA, DTG, and FTIR.

2.2.1. Transesterification of waste palm oil. The acid value of WPO was determined to ascertain their suitability for conversion to WPOME through one stage transesterification process. The clean feedstock was separately mixed with methanol and calcined CaO catalyst into a 500 ml flat-bottom flask in a required quantity and heated on an electric cooker with a magnetic stirrer. A reaction temperature of 60 ^{o}C was selected because it is close but lower than the boiling point of methanol [28]. Other process parameters for the transesterification reaction include methanol to oil ratio of 6:1, the catalyst particle size of 75 μm and 1 %w/w catalyst: oil ratio. The homogeneous mixture was maintained by a magnetic stirrer at 1200 rpm while a digital thermocouple was employed to verify the reaction temperature throughout the process.

The resulting mixture was thereafter filtered in a Buchner funnel filtration system set up to recover the powder catalyst. The filtered solution was conveyed to a separating funnel and exposed to 12 h sedimentation process for the glycerol to coagulate and settle at the base of the separating funnel. The glycerol is tapped out and the remaining crude biodiesel is decanted ready for the purification process.

1 %w/w Magnesol: crude biodiesel was added to the crude biodiesel and heated to 60 °C for 30 mins and mixed at 60 rpm by a magnetic stirrer. The resulting solution was filtered using vacuum filtration, heated to 110 °C to vaporize excess moisture and methanol trapped in the biodiesel and further polished by using 0.45 µm PTFE membrane syringe filter. The purified WPOME was stored in an airtight glass vial and labeled accordingly for characterization.

2.2.2. Characterization of samples. Three samples, namely NPO, WP, and WPOME were subjected to TGA, DTG and FT-IR analyses. The FTIR was analyzed using a Perkin-Elmer Spectrum 100 spectrometer from 380 cm^{-1} to 4000 cm^{-1}. The TGA-DTG were determined using a Perkin-Elmer Simultaneous Thermal Analyzer STA 6000 at a temperature from 30 °C to 600 °C, heated at 10 °C/min with nitrogen as the carrier gas at a gas flow rate of 20 ml/min for the analysis.

3. Results and Discussion

3.1. DTG analysis of NPO, WPO, and WPOME

The outcomes of the DTG analysis of NPO, WPO, and WPOME is shown in figure 2. The DTG curves of NPO and WPO samples showed a similar trend during the entire thermal degradation process which leads to the vaporization of the samples. The evaporation starts at 320 °C for NPO and WPO and ends at 480 °C for and 470 °C respectively until the samples are fully vaporized. On the other, the evaporation of WPOME commences and ends at 27.63 °C and 103 °C, and derivative weight of 3.42 %/m and 0.48 %/m respectively, after which the curve becomes flat, signifying the sample has been fully vaporized. The curves show a single peak for all the samples but at different temperature ranges showing one weight loss stage of the evaporation. The peak in the curves are found as 18.63 $\%m^{-1}$ at 418.37 °C for NPO, 18.5 $\%m^{-1}$ at 418.37 °C for WPO, and 15.25 $\%m^{-1}$ at 63.19 °C for WPOME. This shows that WPOME is thermally degraded in an endothermic reaction at a lower temperature compared with NPO and WPO samples under nitrogen atmosphere. This accounts for why FAME is a better CI fuel than neat vegetable oils because it easily combusted at a lower temperature. These results agree with outcomes of similar researches by Li et al. [22], Niu et al. [29], and Li et al. [30].

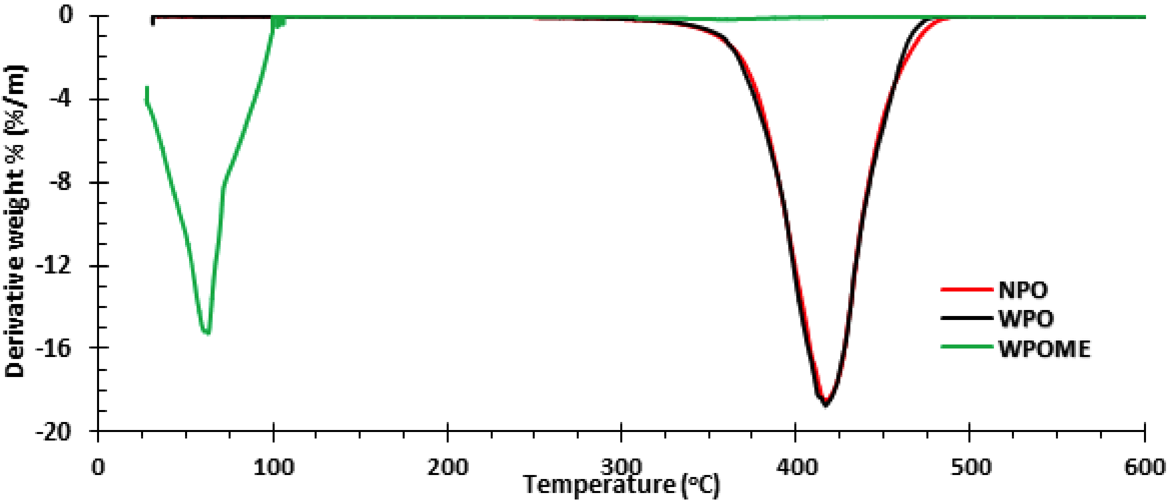

Figure 2. DTG curves for NPO, WPO, and WPOME

3.2. TGA analysis of NPO, WPO, and WPOME

The outcomes of the TGA analysis of NPO, WPO, and WPOME is shown in figure 3. NPO, WPO, and WPOME present similar trends during the entire thermal decomposition process with a single stage weight loss visibly identified. The TGA curves show a distinct step from 30 °C to 600 °C. The thermal degradation range of NPO varies from 330 °C to 480 °C with overall percentage weight loss of 98% while the overall percentage weight change within degradation scope of 350 °C to 470 °C for WPO is 29 %. For the WPOME sample, thermal degradation starts at 350 °C and ends at 470 °C and approximately lose 63 % of its weight. The NPO is degraded at a higher temperature than WPOME due to the fact that NPO is more viscous than WPOME and therefore requires a higher temperature to be degraded. The Lower degradation temperature of WPO compared to WPOME may be attributed to the effect of repeated heating and contamination during usage which has lowered its viscosity and make the bonds to be degraded at a lower temperature. The outcomes of the TGA characterization in this study are in consonance with those in the published and well-referred journals [22, 23, 31, 32].

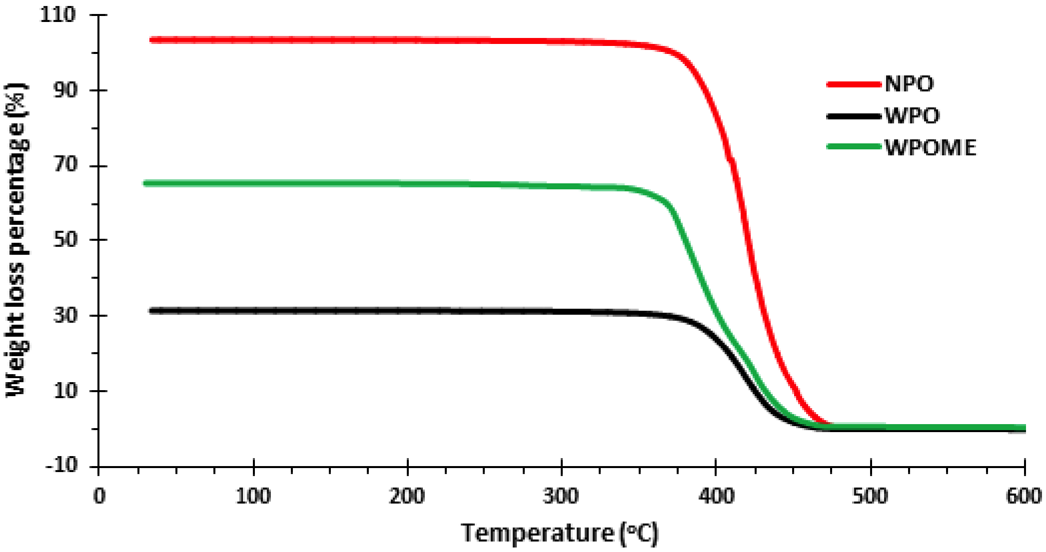

Figure 3. TGA curves of NPO, WPO, and WPOME

3.3. FTIR analysis of NPO, WPO, and WPOME

Four identifiable regions are noticed with characteristics peaks in the FTIR spectrum. The functional groups in the first region with the peaks from 4,000 cm^{-1} to 2,500 cm^{-1} represents N-H, C-H, and O-H single bonds. Peaks from 2,500 cm^{-1} to 2,000 cm^{-1}, showing triple bonds represent the second region while the third region which ranges between 2,000 cm^{-1} and 1,500 cm^{-1}, and the peak corresponds to absorption instigated by double bonds like C=O, C=N, and C=C. From 1,500 cm^{-1} to 400 cm^{-1}, the fingerprint region has many single bonds peaks represents the fourth region [33, 34]. The IR spectrum of the present samples does not contain any peak in the second region as shown in figure 4.

The FTIR spectra showing the functional groups of NPO, WPO, and WPOME are illustrated in figure 4. The resemblances in the FTIR spectra of NPO and WPO 3012 cm^{-1}, 2922 cm^{-1}, 2855 cm^{-1}, 1744 cm^{-1}, 1457 cm^{-1}, 1379 cm^{-1}, 1233 cm^{-1}, 1159 cm^{-1}, and 722 cm^{-1} is an indication of the presence of similar chemical groups in their compositions [35, 36]. This is ascribed to =C-H asymmetrical stretching in alkenes, C-H asymmetrical stretching in alkanes, C-H asymmetrical stretching in methylene, C=O bending in ester carbonyl functional group in FAME, =C-H and –CH_2 bending in methylene groups of cis distributed alkenes and aromatic, respectively [22, 29], as shown in table 1. The peaks for the spectra of WPOME are located at 627 cm^{-1}, 1023 cm^{-1}, 1450 cm^{-1}, 2832 cm^{-1}, 2941 cm^{-1} and 3327 cm^{-1}. These peaks correspond to C-Cl axial deformation in alkyl halide, C-O axial deformation in alcohol, C=C axial deformation in aromatic, O-H axial deformation in axis, C-H axial deformation in alkane, and O-H free axial deformation in alcohol, respectively. The detection of spectra peak at unique wavelengths in WPOME but not in NPO and WPO can be attributed to the distinctive variation between FAME and triacylglycerides [22, 37, 38], and a confirmation of conversion of WPO into WPOME [39].

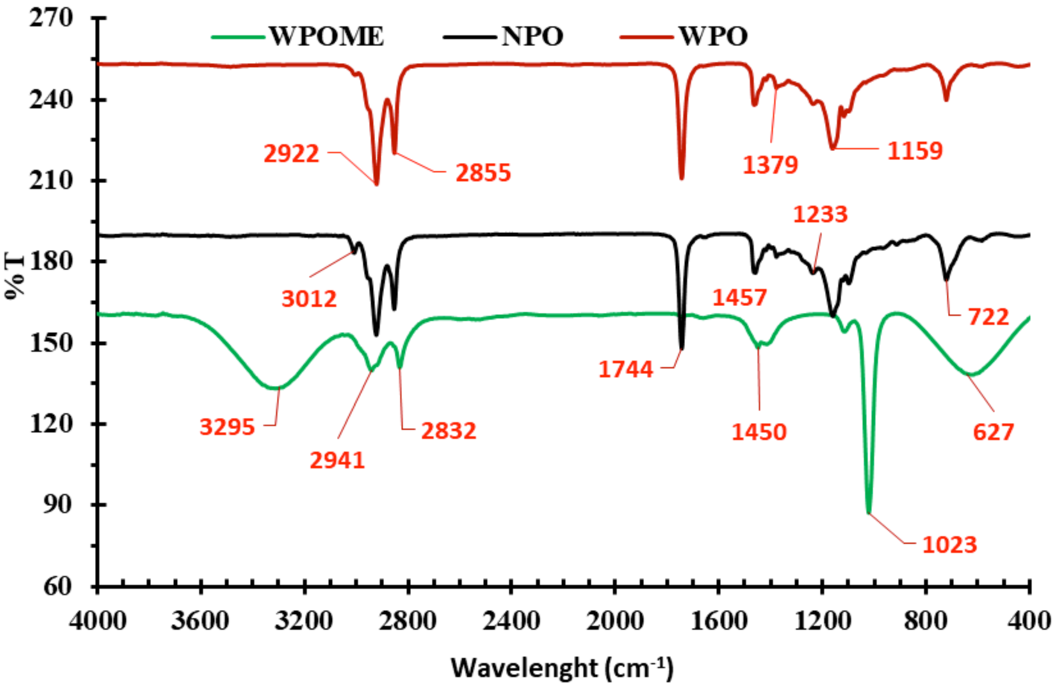

Figure 4. FTIR spectra of NPO, WPO, and WPOME samples

Table 1. Functional group frequencies of NPO, WPO, and WPOME samples [40-44]

Wave number (cm^{-1})	Types of vibration	Functional group	Intensity
3012	Stretching	=C-H, C-H	Strong, broad
3008	Asymmetrical stretching	=C-H of alkenes	
2922	Asymmetrical stretching	C-H of alkanes	
2853	Asymmetrical stretching	C-H of methylene	Medium
1745	Stretching	C=O ester band	
1462	Bending	=C-H (cis)	Variable
1381	Bending in plane	-C-H(CH_3)	Variable
1159	Stretching, Bending	-C-O, -CH_2-	
1024	Stretching	C-O	
722	Bending	=C-H and –(CH_2)n	
627	Out of plane bending	=C-H cis in alkene and aromatics	Strong

4. Conclusions

The DTG, TGA, and FTIR of NPO, WPO, and WPOME are presented. The WPO was taken from the NPO while the WPOME was from the catalytic transesterification of WPO. Neat palm oil was exposed to repeated heating and contamination when used to fry food items. These activities alter the form, and degree of saturation of the palm oil, leading to a change in properties and behavior. The thermal and spectroscopic state of the palm oil is altered during frying, and conversion to biodiesel through transesterification process. The DTG and TGA of NPO, WPO, and WPOME followed similar trend through the thermal degradation and weight loss occurred at different temperatures, owing to the influence of usage and transesterification process. The DTG of the three samples occurred between 330 °C and 480 °C in a one stage weight loss. The NPO exhibited the lowest commencement of thermal degradation temperature followed by WPOME and NPO in that order. The spectra curve of NPO and WPO followed a similar pattern and have their peaks at nearly the same wavelength while WPOME presented a peculiar peak wavelength.

Arising from the outcome of this research, the authors are investigating WPO samples used in frying varieties of food at varying temperature and cycle of frying with a view to establishing the effects of temperature and contamination from food items on its thermal decomposition. Since the outcome of GCMS of fuels has been used extensively to predict the properties, engine performance, combustion, and emission characteristic of internal combustion engines, the use of FTIR should be employed to predict or estimate the fatty acid composition and properties of fuels.

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