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In-situ Hybridization of Waste Palm Oil: A Physicochemical, Thermal, and Spectroscopic analysis

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Abstract

Hybridization is one of the techniques for unearthing novel feedstock and diversifying the existing waste cooking oil feedstock stream. In the present research, in-situ hybridization was carried out on waste palm oil (WPO) samples obtained from different sources. The aim of this current study is to investigate the effect of hybridization on the physicochemical properties, thermal degradation, and spectroscopic on both the WPO and hybridized samples. Two WPO samples were mixed in different ratio and subjected to property determination and characterization. Hybridization was found to increase the iodine value, and reduce the density, kinematic viscosity, and saponification values but does not affect the acid value, cetane index and higher heating values of the samples. All the samples witnessed one stage of thermal decomposition; samples A, B, C, D, and E experienced 13 %, 11 %, 10 %, 8 %, and 3 % weight loss respectively between 320 °C and 470 °C. The peak of derivative weight percentage of -0.06 $\%m^{-1}$ was observed at 433 °C, -0.05 $\%m^{-1}$ at 430 °C, -0.11 $\%m^{-1}$ at 432 °C, -0.09 $\%m^{-1}$ at 422 °C, and -0.06 $\%m^{-1}$ at 430 °C for samples A, B, C, D, and E respectively. The infrared spectrum curves revealed that the peculiar peaks at 1226 cm^{-1}, 1363 cm^{-1}, and 1378 cm^{-1} found in the parent samples A and B disappeared in the spectrum curves of hybridized samples C, D, and E. The outcome of this investigation shows that hybridization is a viable technique for improving the quality of existing feedstock as well as creating novel high-quality feedstock for biodiesel generation.

Keywords: Characterization, feedstock, in-situ hybridization, waste palm oil;

1. Introduction

Renewability, biodegradability, environmental sustainability, and affordability are some of the factors that have popularized the application of biodiesel as a sustainable replacement for fossil-based diesel (FBD) fuel to run compression ignition (CI) engines. The International Energy Agency has projected that the global oil demand will escalate to 105.4 MMbpd in 2030 from the 96.9 MMbpd recorded in 2018 [1]. This increased energy demand has made the use of alternative energy a priority in order to meet the soaring global energy demand. Similarly, the damaging effect of the exploitation and utilization of FBD fuel, depletion of oil reserves, deteriorating oil production capacities, and the increasing price of FBD fuel in the global market has brought about the necessity to move to low-carbon emitting fuels a global priority. The depletion of fossil fuel reserves has made the search for alternative and sustainable fuel inevitable. Such alternative fuel must be affordable, environmentally benign, and carbon neutral [2-4].

Consequently, researchers have continued to commit considerable time and resources to the production and utilization of biodiesel [5, 6]. Biodiesel is biodegradable, more environmentally friendly, more lubricating, and it emits less carbon monoxide, soot, and unburnt hydrocarbon emissions, and generates less engine noise and vibration as well when compared with FBD as CI engine fuel. Biodiesel has also been found to exhibit a higher cetane number and flash point, low sulphur content, and is non-carcinogenic, less toxic, and safer to handle when compared with FBD fuel [7-11].

The high cost of feedstock, the conflict between some food-based feedstock and the food chain, and scarcity of feedstock have continued to negatively impact the commercialization of biodiesel. Economically, the price of feedstock has been found to account for between 60 % to 80 % of the overall production expenditure of biodiesel [10, 12-14]. The use of inedible oil, waste cooking oil (WCO), and recovered animal fats have been discovered to considerably lower the production cost of biodiesel. The use of these feedstocks does not conflict with the food chain and serve as a sustainable waste disposal mechanism. For example, biodiesel generated from WCO was much cheaper than that generated from neat palm oil [15-17]. Lee et al. [18] reported a production cost of 0.7 US$/L of biodiesel when using waste canola oil compared to 1 US$/L for fresh canola oil. At 1.65 US $/L, WCO [19] is the cheapest feedstock when compared with neat vegetable oil at 4.2 US $/L [16], and neat soybean oil at 6.234 US $/L [20].

Waste palm oil (WPO) is commonly used as biodiesel feedstock due mainly to its availability and reasonably low cost when compared with other forms of inedible vegetable oil [21, 22]. WPO is obtained when palm oil is used domestically for frying. Palm oil is obtained from palm

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fruit, which is predominantly grown in tropical Africa, South America, and Southeast Asia. About 90 % of palm oil is believed to be consumed domestically as food, while the remaining 10 % is used for industrial, cosmetics, lubricants, fuels, and as a bio-asphalt binder, among other uses [23, 24]. The domestic consumption of palm oil as food has continued to increase over the last five years (Figure 1). This is because of urbanization, increased population, and change of lifestyles. Exposure of palm oil to temperatures above 200 ºC in the presence of moisture and salt during frying predisposes the oil to physio-chemical and thermal decomposition. Domestic consumption of palm oil that has been used for frying is harmful to health, causing cancer, diabetes, and other diseases [25, 26].

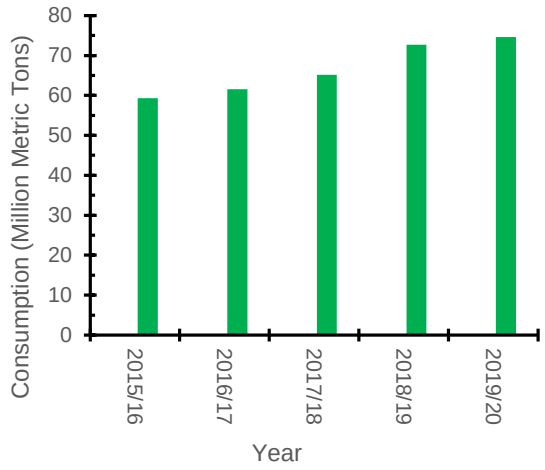

Figure 1. Consumption of palm oil (Million Metric Tons) from 2015/16 to 2019/20

Hybridization is the mixing of two or more distinct feedstocks in varying fractions to create a novel feedstock. The hybridized feedstock has always different properties compared with the parent stocks. Feedstocks can be hybridized in-situ, ex-situ, bi or poly, with the main target being the generation new feedstocks with enhanced physicochemical properties, improved conversion efficiency, and thermal properties [27, 28]. In in-situ blending, which is the method for this research, two different WPO are blended to generate another feedstock with a distinct fingerprint from the parent feedstock.

Not many studies on hybridization of used vegetable oil as feedstock are published in literature. In separate researches, Eloka-Eboka and Inambao [27, 28] carried out in-situ and ex-situ mixing of oils and biodiesels from *Moringa oleifeara* and *Jatropha curcas* in varying proportions. The new products possess distinct properties from their parent feedstock and methyl esters with the potential to open a new vista in the biofuel industry. The relevant question to ask, therefore, is whether the possibility of hybridization of feedstock in creating new and improved feedstocks has been well interrogated. The objective of this research is to investigate the effects of hybridization of two samples of WPO on the properties, thermal degradation and spectroscopic transformation of the outcome of the hybridization.

Specifically, in-situ hybridization was carried out between two WPO samples that have been utilized to fry different food. The samples were mixed in different proportions and the resulting mixtures were tested and analyzed. The motivation was to create different sets of feedstocks, which are expected to have different properties and thermal behavior from the parent WPO samples. The scope of the current research was limited to in-situ hybridization of two WPO samples in varying proportions which were then subjected to density, kinematic viscosity, acid value, iodine value, and saponification value testing as well as characterization by thermogravimetric analysis (TGA), derivative thermogravimetric analysis (DTG) and Fourier Transform Infrared Spectroscopy (FTIR).

2. Materials and methods

2.1. Material collection and samples preparation

Two WPO samples were collected from two restaurants in Durban, KwaZulu-Natal Province, South Africa at the point of disposal. One of the WPO samples had been used to fry fish and chips (WPO_{FC}) for 14 days while the second WPO sample was used to fry sausages and chips (WPO_{SC}) for 14 days. The samples were pretreated by subjecting them to heating on an electric stove / magnetic stirrer maintained at 110 °C and stirring speed of 50 rpm to remove the moisture trapped in the oil. The samples were later subjected to vacuum filtration to eliminate food particles, debris, and other foreign bodies in the oil [25]. Figure 2 shows the picture of the parent samples.

The oils were heated to 60 ºC and poured into a clean beaker and weighed on an electric weigh balance and poured into a bigger beaker where the oils were mixed in a specified ratio. In the present investigation, simple mixing ratios were adopted in order to prevent undue influence of the one parent sample over the other. The oils in the mixing beaker were stirred with the aid of a magnetic stirrer maintained at a speed of 50 rpm for 20 min to allow for a homogeneous mixture of the oils.

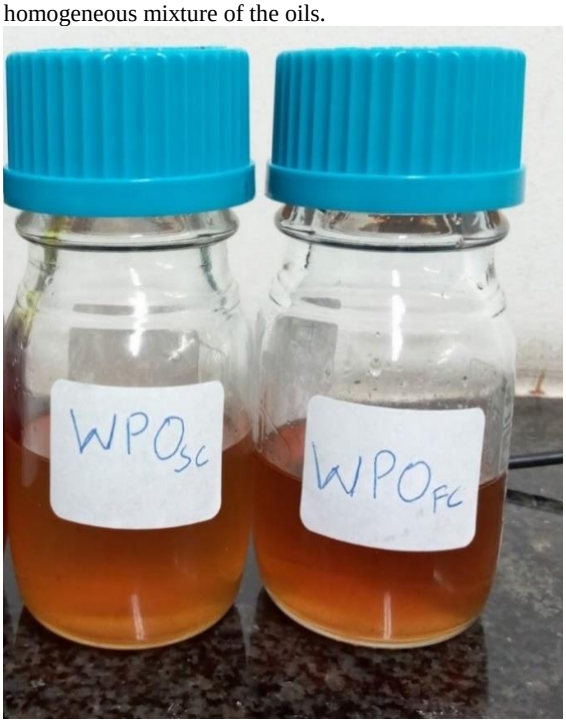

Figure 2: Picture of the parent samples

The hybridized samples are labeled accordingly and stored in airtight glass bottles for property determination

and characterization procedures. The details of the in-situ hybridization are shown in Table 1 while Figure 3 illustrates the flowchart of the methodology. The hybridization protocols are chosen to have different scenarios of mixing the two parent samples WPO_{FC} and WPO_{SC}.

Table 1. Details of the samples and hybridization protocol

Sample notation	Hybridization protocol ratio	
	WPO_{FC}	WPO_{SC}
A	0	1
B	1	0
C	1	1
D	2	1
E	1	2

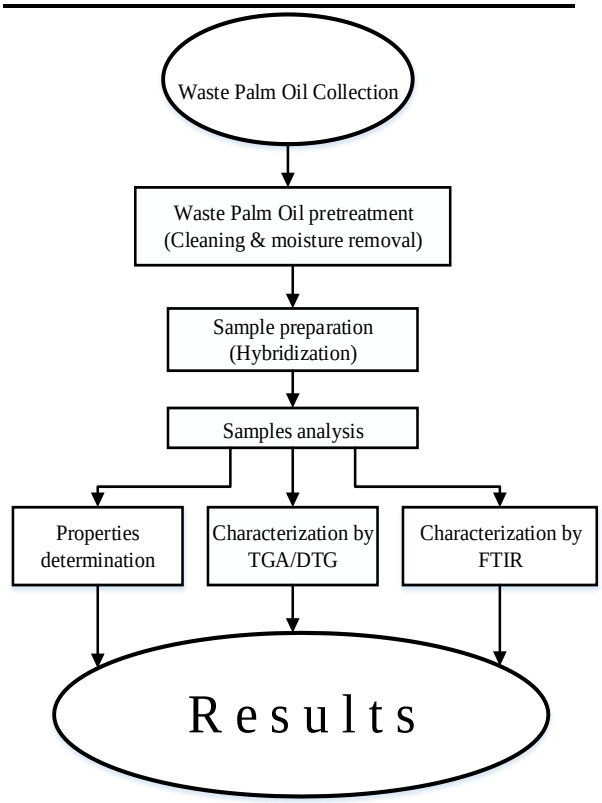

Figure 3. Flowchart of the methodology

2.2. Property determination of samples

The density, kinematic viscosity, acid value, iodine value, and saponification value of the samples were determined by using the appropriate method as shown in Table 2, and the procedures were highlighted in our previous works [18].

Table 2. Methods for properties determination

Property	Unit	Method
Density at 20 ºC	Kg/m^3	ASTM D1298
Kinematic viscosity at 40 ºC	mm^2/s	ASTM D445
Acid value	mgKOH/g	AOCS Ca 5a-40
Iodine value (IV)	cg/g	AOCS Cd 1b-97
Saponification value (SV)	mg KOH/g	AOCS Cd 3-25
Cetane index (CI)	N/A	By calculation
Higher heating value (HHV)	MJ/kg	By calculation

The CI and the HHV were calculated using the mathematical relations shown in equations 1 and 2.

$$CI = 46.3 + \frac{5458}{SV} - \frac{0.225}{IV} \quad [29] \quad (1)$$

$$HHV = 49.43 - 0.041(SV) - 0.015(IV) \quad [30] \quad (2)$$

2.3. Spectroscopic characterization of samples

In order to obtain a recognizable absorption spectrum, the dilution and homogenization of the samples were measured and recorded from 300 cm^{-1} to 4000 cm^{-1} on a spectrometer (model system 1000 FTIR, Perkin Elmer Co., USA) with a resolution of 2.0 cm^1.

2.4. Thermal characterization of samples

The TGA/DTG analyses were performed using a DTG-60AH simultaneous DTA-TG apparatus coupled with a TA-60WS thermal analyzer (Shimadzu). The sample weight was about 10 mg, a temperature range of 30 °C to 500 °C, a heating rate of 20 °C/min and a nitrogen flow rate of 50 cm^3/min. The data were analyzed by using the TA-60 ch 1 DTG-60AH workstation.

3. Results and Discussions

3.1. Effects on physicochemical properties

The density, kinematic viscosity, acid value, IV, SV, CI, and HHV of the samples are presented in Table 3. The density of the individual parent samples and the hybridized samples are within the same range, though the hybridized samples C, D and E presented slightly lower density than the samples A and B. The kinematic viscosity of the hybridized samples C, D, and E are marginally lower than those of samples A and B. The slight reduction recorded in the values of density and kinematic viscosity can be attributed to the effects of the physical mixing of the parent samples. This conforms with the outcomes of similar work reported in the literature [27, 28]. Rahiman and Santhoshkumar [31] reported that the density of liquid does are not only affected by mixing or blending but also by the temperature. They attributed the variation to the effect of the intermolecular interactions between mixing liquids. The acid value of the samples is not affected by hybridization while the saponification value of the hybridized samples C, D, and E were lower than that of the parent samples A and B. Hybridization lowers the iodine value of the samples when compared with the parent samples. CI and HHV are not affected by hybridization since mixing did not affect the heating capacity of the samples. Since hybridization of the samples took place at room temperature and no chemical reaction was witnessed, the variations in the physicochemical properties of the hybridized samples can only be traced to the effect of the mixing compared with the parent samples.

3.2. Effects on TGA

The outcomes of the TGA examination of the five samples are presented in Figure 3. The TGA plot compares the percentage weight loss of the two-parent WPO samples

with the three hybridized samples concerning the change in temperature. A temperature range of 30 °C to 500 °C was adopted for the five samples. The samples experienced single-stage thermal degradation which starts at around 330 °C for all the samples. With the test temperature differential, sample A, B, C, D, and E witnessed 13 %, 11 %, 10 %, 8 %, and 3 % weight loss, respectively. The thermal degradation commenced at between 320 °C to 330 °C for all the samples. The thermal degradation stopped at 450 °C for samples A, B, C, and D while that of sample E ended at 470 °C. Only the degradation curve for sample E dovetailed into the negative region of the curve. The commencement of the degradation temperature agrees with the outcome of our earlier work [26]. The high degradation temperature was due to the presence of several complex chemical compounds in the samples [32]. Waste cooking oil is susceptible to thermal decomposition at high temperatures as a result of the existence of unsaturated fatty acids which require low thermal energy to break [33-35].

3.3. Effects on DTG

The samples exhibited a similar trend of percentage derivative weight during the thermal degradation process. As shown in Figure 4, the thermal decomposition occurred between 350 °C and 500 °C with each curve presenting a single noticeable peak. The peak in the curves were observed as -0.06 $\%m^{-1}$ at 433 °C for sample A, -0.05 $\%m^{-1}$ at 430 °C for sample B, -0.11 $\%m^{-1}$ at 432 °C for sample C, -0.09 $\%m^{-1}$ at 422 °C for sample D, and -0.06 $\%m^{-1}$ at 430 °C for sample E. During the degradation process, sample C showed the greatest percentage derivative weight, followed by sample D and sample E in that other. This indicates that hybridization slightly influenced the derivative weight percentage and showed that hybridization can produce new sets of feedstocks different from the parent feedstock with the capability of positively influencing biodiesel conversion efficiency [36-38].

Table 3. Properties of the oil samples

samples	Density @ 20°C (Kg/m^3)	Kinematic viscosity @ 40 °C (mm^2/s)	Acid value (mgKOH/g)	Iodine value (cg/g)	Saponification value (mg KOH/g)	Cetane index	Higher heating value (MJ/kg)
A	9188.3	35	1.38	54.4	197.6	73.92	40.51
B	9171.2	34.2	0.97	74.4	199.9	74.30	40.32
C	9150.4	31.7	1.18	89.7	195.9	73.88	39.97
D	9161	30	1.09	92	193	74.58	40.14
E	9169.3	30.8	1.16	86	192	74.72	40.27

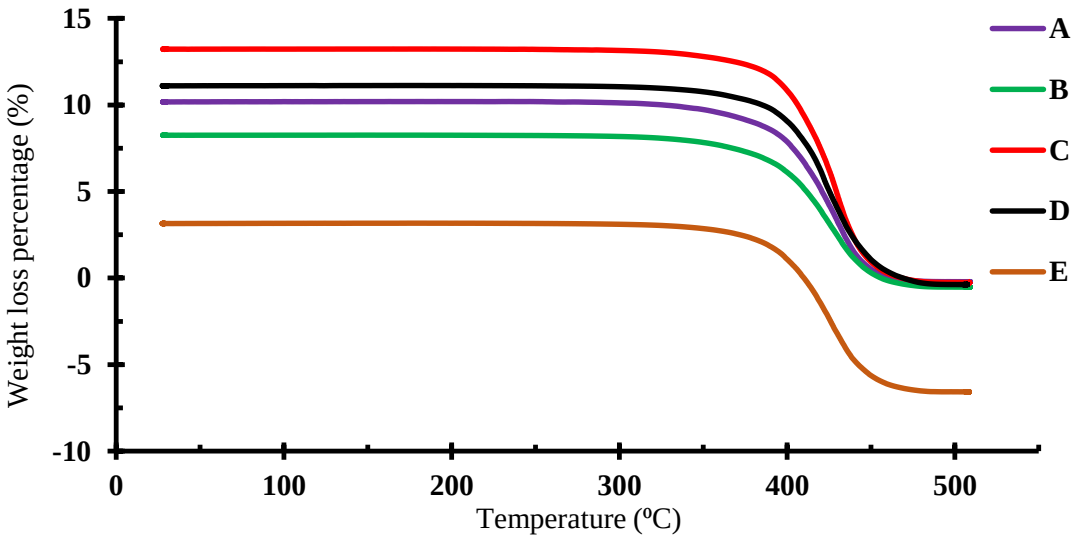

Figure 3. TGA curves for the samples

3.4. Effects on FTIR

The FTIR spectra depicting the functional groups of the five samples are shown in Figure 5. Four regions are identifiable with characteristics peaks in the IR spectrum. The four peculiar regions are distinguishable with characteristic peaks in the FTIR spectrum namely 4000 cm^{-1} to 2500 cm^{-1}, 2500 cm^{-1} to 2000 cm^{-1}, 2000 cm^{-1} to 1500 cm^{-1}, and 1500 cm^{-1} to 400 cm^{-1} are present in the curves. These peaks could be assigned to (C-H) symmetrical, asymmetrical stretching of the saturated carbon-carbon bond, C=O group of triglycerides, and stretching vibrations of the (C-O) esters group. However, the FTIR spectrum in the second region represented by 2500 cm^{-1} to 2000 cm^{-1} is absent in the curves. This is in agreement with the outcome of similar research as reported in the literature [39, 40]. The five samples have common significant and recognizable peaks at 723 cm^{-1}, 1165 cm^{-1}, 1747 cm^{-1}, 2855 cm^{-1}, and 2924 cm^{-1} indicating the existence of similar chemical groups in the compositions and similar fingerprints [41-43]. However, the parent samples A and B have peculiar peaks at 1226 cm^{-1}, 1363 cm^{-1}, and 1378 cm^{-1} that were found to either disappear or be minimized in the hybridized samples C, D, and E spectrum. The band 1165 cm^{-1} shows the manifestation of methyl esters close to carbonyl groups. The vibration band noticed at 723 cm^{-1} accounts for ν(=C-H) and ν(–(CH_2)n) functionals. The type of frequencies, functional group, and absorption intensity of the wave numbers noticed in the spectrum are presented in Table 4.

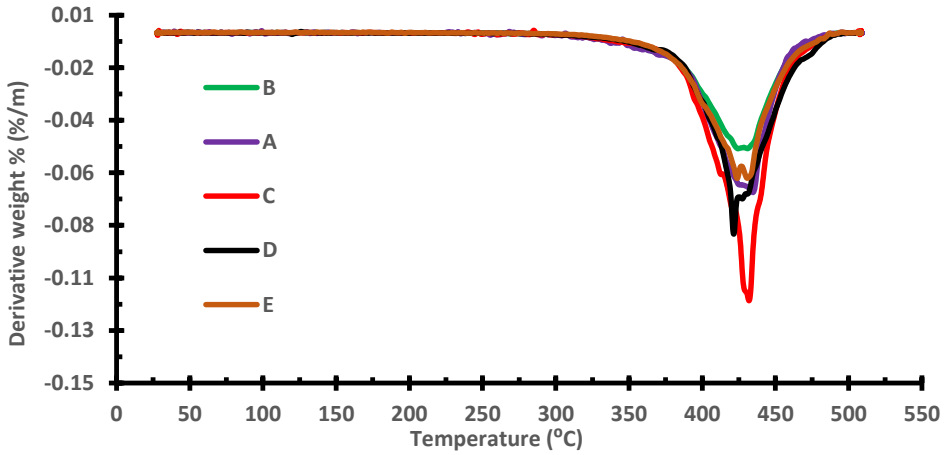

Figure 4. DTG curves for the samples

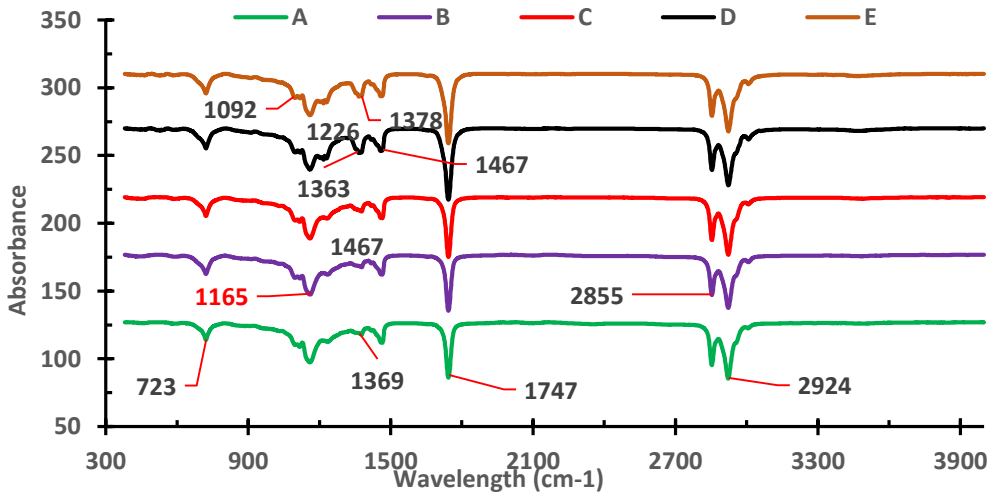

Figure 5. FTIR spectra of the samples

Table 4. Description of the peaks of the spectrum of the samples

Wave number (cm^{-1})	Types of vibration	Functional group	Absorption intensity	Ref
2924	Asymmetrical stretching	C-H of alkanes	Middling	[44]
2855	Asymmetrical stretching	C-H of methylene	Strong	[44]
1747	Stretching	C=O ester band	Strong	[45]
1378	Bending in plane	-C-H(CH_3)	Weak	[46]
1363	Bending	CH_2	Weak	[47]
1165	Stretching, Bending	-C-O, -CH_2-	Strong	[46]
723	Bending	=C-H and -(CH_2)n	Weak	[48]

4. Results in Comparison with previous findings

Availability, ease of conversion to biodiesel, and the desire to find an affordable feedstock for biofuel production have triggered interest of researchers in WCO. Among the properties investigated are density, viscosity, saponification value, acid value, fatty acid composition, and iodine value. Table 5 showed outcomes of some of the investigations as published by various authors. When compared with the outcome of this research as shown in Table 3, it can be shown that the properties of WCO are largely dependent on factors such as source of the oil, degree of usage, frying temperature, food used to fry, and hybridization [27, 49].

The Muppaneni et al.[53] , Almazrouei et al.[54], Ullah et al.[52], and Çaylı and Küsefoğlu [55] reported that WCO witnessed one stage thermal degradation. The TGA thermographs showed that thermal decomposition commenced at between 350 °C and 400 °C and were completely decomposed at temperature between 450 °C and 500 °C. This agrees with the outcome of this research. The shapes of the TGA curves were similar to the one shown in Figure 3. The authors attributed the slow thermal degradation of WCO to the high viscosity and molecular tension produced by bulky triglyceride molecule in the oil samples.

The outcome of the DTG characterization as reported by researchers showed that WCO witnessed derivative weight lost at between 350 °C and 500 °C with the peak weight loss of 38.76 % at 414 °C [55]. The thermal and spectroscopic properties of WCO are affected by source of the neat oil, degree of usage, frequency of usage, food items the oil was used to fry.

The outcome of the FTIR characterization by Ullah et al. showed peak vibrations at 2920.30 cm^{-1}, 2851.9 cm^{-1}, and 1743.1 cm^{-1} which are assigned to (C-H) symmetrical, asymmetrical stretching of the saturated carbon-carbon bond, and C=O group of triglycerides respectively. A small band at 1656.69 cm^{-1} resulting from cis C=C bond. The Bands at 1463.79 cm^{-1} resulting from the bending vibrations of CH_2 and CH_3 aliphatic groups were also noticed. Other bands noticed at 1157.19 cm^{-1} and 1116.87 cm^{-1} could be attributed to the stretching vibrations of the (C-O) esters group. These and other similar peaks exhibited by the samples at 2924 cm^{-1}, 2855 cm^{-1}, 1747 cm^{-1}, 1467 cm^{-1}, 1363 cm^{-1}, 1167 cm^{-1}, and 723 cm^{-1} as shown in Figure 5 and described in Table 5. However, to the best of authors knowledge, the effects of hybridization of WCO on TGA, DTG, and FTIR have not been reported.

5. Conclusion

The outcome of the effects of the hybridization of feedstock on the physicochemical properties, thermal characterization, and infrared spectroscopy has been presented. Hybridization is one of the novel ways of improving the quality of feedstocks. Hybridization of feedstock creates an entirely new feedstock from the existing feedstock with better properties and behavior regarding biodiesel generation. A hybridized feedstock is expected to combine the properties and peculiarities of the parent feedstocks by mixing the parent feedstocks in each ratio. In this research, two samples of WPO were mixed in different ratios and a total of five samples were analyzed by property determination and characterization by TGA/DTG and FTIR. The results of the parent samples were compared with that of the hybridized samples. From the outcome of this research, it is possible to conclude as follows:

1. Apart from the effects of oil source, degree of usage, the food the oil was used to fry, contamination, the frying frequency, the frying temperature, etc on the properties, thermal and spectroscpic properties of WCO, hybridization has a major influence on the physicochemical properties and the characterization behaviour of WCO.
2. Hybridization has no significant effect on the acid value, cetane index, and HHV of the feedstock. However, the density, kinematic viscosity, and saponification value was found to reduce with hybridization while the iodine value of the hybridized feedstock was found to be higher than the iodine value of the individual parent feedstock.
3. TGA and DTG of feedstock are affected by the hybridization of feedstock. The rate of thermal decomposition increases with the mixing of feedstocks, though all the samples witnessed single-stage thermal degradation. The TGA and DTG of both the parent and the hybridized samples occurred within the same temperature range.
4. The FTIR of the parent WPO samples were slightly different from the FTIR of the hybridized samples. Though the five samples A, B, C, D, and E presented similar peaks, the parent samples A and B showed peculiar peaks at 1226 cm^{-1}, 1363 cm^{-1}, and 1378 cm^{-1} which were not noticeable in the spectrum of the hybridized samples.
5. Hybridization of feedstock provides an easy, cheap, and novel way of improving the properties, thermal decomposition, and infrared spectroscopy of feedstocks thereby improving their conversion efficiency to biodiesel.

Going forward, more investigations are needed in in-situ hybridization particularly in mixing more than two different feedstocks at different mixing ratio of feedstocks, more property determination, and characterization techniques. The effects of hybridization of feedstocks on the conversion efficiency, biodiesel fingerprints, combustion, performance and emission characteristics of biodiesel needs to be quantitatively ascertained.

Table 5. Some properties of WCO mined from literature.

Properties	Unit	[49]	[49]	[50]	[51]	[52]
Density	Kg/m^3	904.3	913.4	870	910 – 924	901.3
Kinematic viscosity@ 40 °C	mm^2/s	44.25	38.41	5.03	36.4 – 42	44.956
Saponification value	mgKOH/g	NA	NA	NA	188.2 – 207	177.97
Acid value	mgKOH/g	0.66	1.13	0.29	1.32 – 3.6	4.03
Iodine number	cg/g	81.7	54.2	NA	83 – 141.5	NA

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