



PRODUCTION, PROPERTIES AND FATTY ACIDS PROFILE OF SOME REFINED VEGETABLE OILS BASED BIODIESELS

ALIRU O. MUSTAPHA^{1*}, RASIDAT A. ADEPOJU¹, ROFIAT Y. AJIBOYE¹,
YEMISI T. AFOLABI¹, AKEEM A. JIMOH¹ AND ZULIAH A. ABDULSALAM¹

¹Department of Chemical, Geological & Physical Sciences, Faculty of Pure and Applied Sciences, Kwara State University Malete, PMB 1530, Ilorin, Kwara State, Nigeria.

AUTHORS' CONTRIBUTIONS

This work was carried out in collaboration among all authors. Author AOM helped in conceptualization, performed methodology, did software analysis and reviewed the manuscript. Author RAA performed data curation and wrote original draft of the manuscript. Author RYA performed visualization, investigation, software analysis. Author YTA did data validation, software analysis, helped in reviewing process. Author AAJ supervised the study, did software analysis and reviewed the manuscript. Author ZAA wrote, reviewed and edited the manuscript. All authors have read and agreed to the published version of the manuscript.

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ABSTRACT

Utilizing varieties of non-edible vegetable oils such as Jatropha (*Jatropha curcas*; JSO), Neem (*Azadirachta indica*; NSO) and Castor (*Ricinus communis*; CSO) are attractive options to produce biodiesel economically, but, high free fatty acids (FFA) in the vegetable oils are serious issues for the transesterification. In this work, the physicochemical qualities of the three crude and refined jatropha oil, RJO; neem oil, RNO and castor oil, RCO were investigated to determine the influence of refining processes of degumming; alkaline and bleaching on their physicochemical qualities, respectively. Using the Association of Analytical Chemist (AOAC) and American Standard for Testing Materials (ASTM) standard techniques, while the trans-esterification reactions were monitored by Fourier Transform Infra-Red (FT-IR), the fatty acid methyl esters (FAME) quality assessment were obtained from the Gas Chromatography–Mass Spectroscopy (GC-MS). The results of the physicochemical properties of refined feedstock (RJO, RNO, RCO) compared with the crude (JSO, NSO, CSO) indicated higher percentage of improved quality over the feedstock in the parameters measured such as yield (56.0, 55.7, 58.3 %); acid value (1.122 mg/KOH); saponification value (179.6, 302.9, 261.0 mg/KOH); iodine value (26.7, 20.3, 37.9 I₂/100g); peroxide value (2.4, 18.4, 8.8 Meq O₂/kg); density (1.5, 1.3, 1.4 g/cm³); viscosity @ 40 °C (32.0, 58.9, 20.0 %); refractive index @ 30 °C (1.5, 1.5, 1.5 %), respectively. The physicochemical properties of the biodiesel showed similar trend in the quality that compared with the ASTM standard for biodiesels. The fatty acid profile assessment of refined feedstocks shows different 14 compounds with varying percentage composition (1.61 - 20.78 %) with oleic acid as the most abundant in RNO followed by ricinoleic acid (19.59 %) in RCO. The biodiesel produce here indicates better physicochemical improvements that may serve as better alternative energy source along with environmental pollution and production cost reduction.

Keywords: Biodiesel; physicochemical; vegetable oil; fatty acid; refinement; non-edible; fuel.

*Corresponding author: Email: aliru.mustapha@kwasu.edu.ng;

1. INTRODUCTION

Biodiesel is typically made by chemically reacting lipids (e.g., vegetable oil, soybean oil, animal fat, tallow), and can be classified as vegetable oil or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, ethyl, propyl) esters with an alcohol producing fatty acid esters. These esters can be obtained from vegetable oils by trans-esterification with methanol/ethanol. Biodiesel can be synthesized from edible and non-edible oils, non-edible oils have good potential as an alternative diesel fuel, and their production is friendly to the environment. Recently, the interest in biodiesel fuel production has increased tremendously due to its environmental benignancy. A large variation of plants that produce non-edible oils can be considered for biodiesel production. The non-edible oils, such as jatropha, neem, castor, pumpkin, etc., are easily available in developing countries and are very economical comparable to edible oils [1-5].

Biodiesel is composed of acid with long-chain monoalkyl fatty esters gotten from renewable vegetable oils or animal fats in compliance to the requirements of ASTM D6751 [6].

Presently, renewable resources such as biofuels, wind, water, and hydrothermal energy are being widely considered as potential alternative sources of energy [7]. The creativity behind the alternative renewable biodiesel development is that there are some main issues like greenhouse effect, global warming effect, fossil fuel diminishing reserves, and higher petroleum prices are driving worldwide interest on the development of biodegradable and sustainable biofuels [8]. Fossil fuel combustion leads to about 98% of carbon emissions [9]. A life cycle analysis of biodiesel fuel showed that overall CO₂ emission is reduced by 78% in comparison with petro-diesel fuel, hence climate-friendly [10]. Thus, the potential of lowering the net gas emissions from the transportation sector, generating sector, that causes global effect warming could significantly reduce the mass and carcinogenicity of particulate matter emissions.

Biofuels such as biodiesel are investigated to be a possible candidate to substitute for petro-diesel fuel. In addition it is ranked among the fastest developing alternative to petro-diesel fuel in many developed and developing countries worldwide [7]. This is because the net level of carbon dioxide in the atmosphere is not increased by burning bio-fuel, and this minimizes the intensity of greenhouse effect [11] besides, it decreases particulate emissions, unburned hydrocarbons, and sulfur dioxide generated through its combustion process [12].

Trans-esterification is the reaction in which triglycerides react with an alcohol in the presence of catalyst to produce biodiesel and by-product, glycerol. This reaction is mostly affected by various factors among others which include: alcohol to oil molar ratio, reaction time, nature and amount of catalyst, reaction temperature, and the nature of feedstock composition [13]. Nonetheless, alcohol to oil molar ratio is believed to be the most critical in the dynamics of biodiesel production. Conventionally, biodiesel is trans-esterified using refined vegetable oils, catalyzed by an alkali [14, 15].

Therefore, the use of refined non-edible oils for biodiesel production may be good alternative in our search for cheaper fuel sources in the future and therefore require investigation. This work hopes to evaluate the fuel properties including the free fatty acid composition of low-cost non-edible seeds to get varieties of biodiesel of higher grades.

2. MATERIALS AND METHODS

2.1 Materials and Chemicals

Chemicals and equipment were obtained from Cerrilliant (Round Rock, TX, USA LGC), Aldrich Sigma (Gillingham Dorset, UK). Biodiesel analyzed using reflux set, micro pipette, separating funnel, digital hot plate with magnetic stirrer, pycrometer (ASTM D941), pH scale meter (Hanna HI 4212 Model), Cannon- Fenske Viscometer Ostwald (Cannon instrument Co. State College, PA, USA). Feedstocks (neem, castor, jatropha seeds) were obtained from Mandate Market, Ilorin, Kwara State, Nigeria.

2.2 Refining Processes

Non-edible seeds oils was subjected to the refining processes of degumming, alkaline, bleaching treatment are concern with removing all impurity before economical transesterification to supply refined seeds oils (RSOs). The raw seed oils was measured and mixed with aqueous 300µg/mL NaCl (aq) solution. The mixture was agitated at 60°C for 1hour on a hot plate with constant stirring. The insoluble phosphatides obtained were separated using C5 bench top centrifuge at 1000rpm for about 30minutes. The colloidal mixture formed was removed from the oil by decanting. The degummed seed oil (DSO) was measured into a beaker and agitated on a hot plate for about 5minutes, 2.5M NaOH (aq) solution was measured and added to the beaker with constant stirring for 30minutes. A thick mixture was formed at the bottom of the beaker which

was believed to contain a mixture of soaps and the remaining phospholipids was separated by decanting. The degummed-alkaline seed oils (DASO) was measured into a beaker. The oil was heated to about 100°C to completely remove all water molecules present. Activated charcoal was added and the mixture on hotplate with constant stirring for 45 minutes. The obtained mixture was centrifuged for 15 minutes at 2500 rpm using C5 bench top centrifuge. The acid esterification required the free fatty acid (FFA) value to be brought below 0.5, for the trans-esterification to proceed [16, 17]

2.3 Trans-esterification Technique

The refined seed oil methyl esters (RSOME) were obtained from the reaction of refined seed oils (RSOs) and methyl alcohol, heated and stirred for 1 hour @ 65°C, at oil to methyl alcohol molar relation of 1:6, in the presence of NaOH as catalyst [18, 19]

2.4 Physicochemical Analysis

The three different refined methyl esters (RSOME) were evaluated by the Association of officials of Analytical Chemists and American Society for Test for quality determination of acid value (mgKOH/g), iodine value (I_2 mg/100g), peroxide value (MeqO₂/Kg), Saponification value (mgKOH/g) and free fatty acid. The specific density, higher heating value, kinematic viscosity, ester value and cetane number (ASTM D6751-09) Determination of free fatty acid profile of RSOME: Gas Chromatography Mass Spectrometry (GC-MS) analysis was conducted with Varian 3800 gas-chromatography together with Agilent MS capillary column (30m* 0.25mm i.d). FTIR analysis was done with Shimadzu instrument. KBr (Potassium bromide) was grounded into (powdery form) pelletized (with hydraulic press) and scanned at a frequency vary of 4000-400cm⁻¹ [9, 20-21].

3. RESULT AND DISCUSSION

3.1 Physicochemical Properties of CSO, RSO and RSOME

Table 1 presents the physicochemical properties obtained for CSO, RSO, and RSOME. For jatropha, neem and castor seeds oils. The yield of RJO, CNO and RCO from CJO, CNO, and CCO was 56%, 55.66% and 58.25% respectively. The value of 88.88 mm² at 40°C was given by [16] for viscosity characteristic determination. Kinematic viscosity property ought to be between the 1.9-6.0 mm²/s. the viscosity value of CJO, CNO, CCO and RJO, RNO,

RCO was 32.0, 30.2, 60.0 and 58.85, 21.05 and 20.0 (mm²/s) respectively were observed.

The level of acid value of 39.89, 46.60 and 2.230 mg/KOH for (CJO, CNO and CCO) respectively was found before refinement. The acid value was drastically reduced after refinement to 1.122 mgNaOH/g for RJO; RNO and RCO. The huge reduction of 97.18, 97.59, 49.73 % FFA in RJO; RNO and RCO Respectively, they encourages the recycling prospect of the used oils, and it has gained better domestic and industrialized use with improved resistance to oxidative damage. However, the acid value in this work was considerably lower than the 6.1721 mg/KOH from the biodiesel produced from waste cooking oil reported by Rahadanti et al. [16]. The peroxide value of RJO after refinement increases from 1.6 to 2.4 Meq O₂/kg, representing about 50 % reduction while for RNO and RCO after refinement decreases from 22 to 18.4 Meq O₂/kg, and 10.4 to 8.8 Meq O₂/kg, representing about 16.36% and 15.39% reduction respectively. The differences in peroxide values of 65.76 and 59.64 Meq O₂/kg were observed from refinement of *Citrullus. colocynthis* and *Pongamia pinnata* oils as reported by Duhan et al. [22]. The result was an indication that the refined oil was of better-quality after refining treatments. The iodine values obtained for CJO, CNO, and CCO was (32.89, 18.57 and 41.52 I₂ mg/100g), and RJO, RNO and RCO was (26.65, 20.25 and 37.96 I₂/100g) shows remarkable improvement respectively with the reduction of 18.97, 8.96 and 8.57% after refinement.

This result shows that the refined oil possesses better oxidative stability and can be employed for industrial application purposes. Duhan et al. [22] reported a reduction rate of 25.59% and 27.37% respectively for iodine value after refinement. Saponification value result shows that RJO, RNO and RCO comprises of fatty acids of moderate long chain lengths (C₁₆-C₂₀) due to their relatively low Saponification values of all oils have values below ASTM specified standard (≥ 312). The Saponification ranges of 188.2 - 207 mg/KOH have been reported by Mohammed and Bhargavi [23] indicating the suitability of oil for further application as shown in Fig. 1-3 (GCMS chromatograms for RJME, RNME and RCME). The results also compared well with data obtained for *Ximenia americana* seed oil (XASO) and Linseed oil [24 – 26].

3.2 Fatty acid Profile of Biodiesels

Table 2 provide the fatty acids profile of the RJO, RNO and RCO relative to their retention time and total percentage composition of 52%; 59.76% and 66.48%, respectively. The oleic acids composition of

(18.09 %); (20.78%); and (19.59%) in RJO, RNO and RCO were the most abundant followed by n-hexadecanoic acids (12.03 %); (10.65%); and (17.30%), for the same RJO, RNO and RCO respectively. The RJO, RNO and RCO samples have saturated acids in smaller quantities which confirm their suitability as feedstock. The fatty acids profile of RJO are acetic acid, octanoic acid, oleic acid, n-hexadecanoic acid, octadecanoic acid, dodecanoic acid, methyl ester, eicosanoic acid, 6-octadecenoic acid, methyl ester, (z)-and 9- octadecenoic acid, 12-

hydroxy-,methyl ester, [r-(z)], while the RNO have nonanoic acid, n-hexadecanoic acid, oleic acid, 9,12,15-octadecatrienoic acid, (z, z, z)-; 9, octadecadienoic acid; eicosanoic acid and RCO have octanoic acid, l-valine, ethyl ester , 9,12-octadecadienoic acid (z,z)-carbonic acid, prop-1-en-2-yl tridecyl ester, n- hexadecanoic acid, oleic acid , ricinoleic acid and 9-octadecenoic acid, 12-hydroxy-, methyl ester, [r-(z)]-. The results also compared well with data obtained for ASTM D6751-09 standards [6].

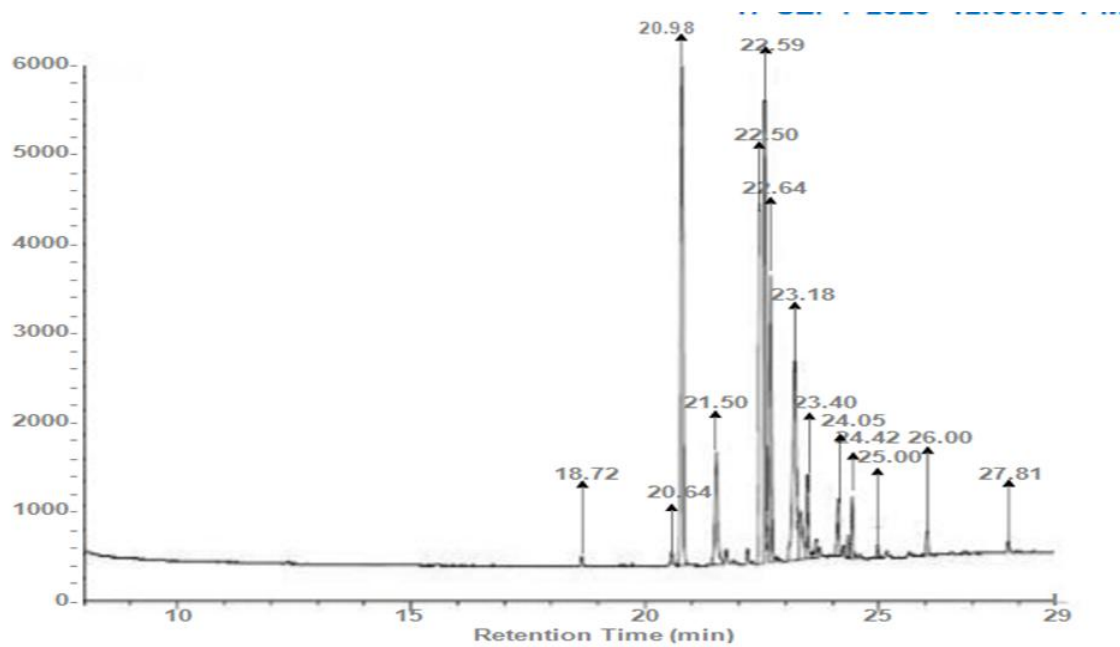


Fig. 1. GCMS chromatograms for RJME

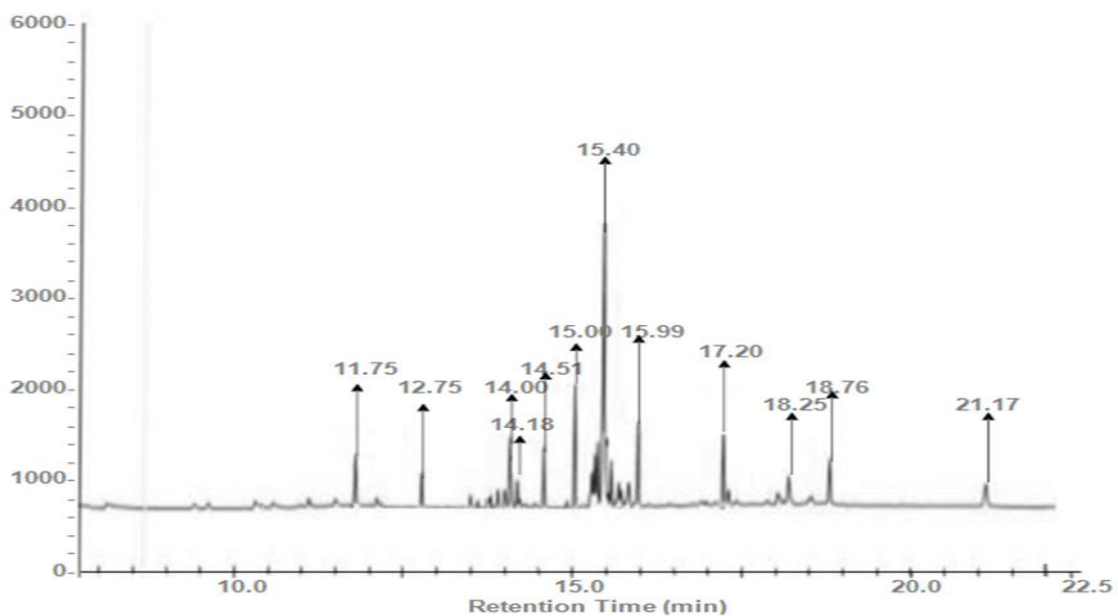


Fig. 2. GCMS chromatograms for RNME

Table 1. Physicochemical properties of plants' seed oils

Physicochemical Properties	Jatropha oil		Neem oil		Castor oil		Ximenea americana seed oil* (XASO)	Linseed oil**	ASTM Standard
	Crude Jatropha oil (CJO)	Refined Jatropha oil (RJO)	Crude Neem oil (CNO)	Refined Neem oil (RNO)	Crude Castor oil (CCO)	Refined Castor oil (RCO)			
Yield (%)		56%		55.66%		58.25%	45.7	-	46-55
Colour	Golden Yellow	Golden Yellow	Light Brown	Brown	Brown	Light Brown	Dark	Yellow	Amber
Refractive index (30 °C)	1.454 ± 0.00	1.462 ± 0.00	1.459 ± 0.00	1.468 ± 0.01	1.472 ± 0.01	1.479 ± 0.00	1.467	1.479	1.476-1.479
Specific gravity(30 °C)	0.902	0.83	0.857	0.921	0.90	0.934	0.912	0.98	0.957–0.968
Density at 40°C (g/cm ³)	1.441	1.450	1.324	1.331	1.353	1.390			0.88
Viscosity (cp, 30 °C)	32.0± 0.00	30.2± 0.07	60.0 ± 0.14	58.85 ± 0.07	21.05 ± 0.07	20.00 ± 0.00		-	1.9-6.0
Acid value, (mg/KOH)	39.89 ± 0.01	1.122 ± 0.00	46.60 ± 0.01	1.122 ± 0.00	2.230 ± 0.00	1.122 ± 0.00	16.13	1.0	0.4 – 4.0
Saponification,(mg/KOH)	72.94 ± 0.00	179.55 ± 0.00	117.83 ± 0.01	302.95 ± 0.00	123.44 ± 0.00	261.00 ± 0.00	178.5	185.0	175 – 187
Iodine Value, (I ₂ mg/100g)	11.065 ± 0.01	32.89 ± 0.01	18.57 ± 0.01	20.25 ± 0.01	41.52 ± 0.00	37.96 ± 0.00	152.28	188.0	82 – 88
Peroxide Value, (Meq O ₂ /kg)	1.6	2.4	22	18.4	10.4	8.8	31.25	-	10
Ester value, (mg/KOH)	33.10 ± 0.01	178.43 ± 0.01	71.24 ± 0.00	301.83 ± 0.00	258.75 ± 0.02	122.31 ± 0.01			
Calorific value(J/Kg)	43343.53	-10559.63	40694.58	-130106.8	40271.369	32265.11			
% Glycerine	1.80937	9.7536	3.8942	16.4992	14.1443	6.6859			
%FFA	20.03	0.564	23.43	0.564	1.122	0.564	8.07	0.5	0.3 – 1.0
Av. Molecular weight(gmol ⁻¹)		943.227 ± 0.00		557.58 ± 0.00		647.611 ± 0.00	314.34	-	

*Oladipo etal (2013)*Majumder (1990) ***

Table 2. Comparison of fatty acid profiles of refined seeds oils

Ret. Time	RJO			Ret. Time	RNO			Ret. Time	RCO		
	Composition	Saturation	Composition (%)		Composition	Saturation	Composition (%)		Composition	Saturation	Composition (%)
18.72	Acetic acid	C ₂ H ₄ O ₂	1.92	12.75	Nonanoic acid	C ₉ H ₁₈ O ₂	6.30	14.50	Octanoic acid	C ₈ H ₁₆ O ₂	2.94
22.59	Octanoic acid	C ₆ H ₁₆ O ₂	4.22	15.00	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	10.65	14.98	L-valine, ethyl ester	C ₇ H ₁₅ NO ₂	2.69
22.59	Oleic acid	C ₉ H ₁₂	18.09	15.40	Oleic acid	C ₁₈ H ₃₄ O ₂	20.78	19.98	9,12-Octadecadienoic acid (Z,z)-	C ₁₈ H ₃₂ O ₂	10.45
22.64	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	12.02	15.99	9,12,15-Octadecatrienoic acid, (Z, z, z)-	C ₁₈ H ₃₀ O ₂	7.99	20.72	Carbonic acid, prop-1-en-2-yl tridecyl ester	C ₁₇ H ₃₂ O ₃	1.37
23.18	Octadecanoic acid	C ₁₈ H ₃₆ O ₂	8.76	17.20	9, Octadecadienoic acid	C ₁₈ H ₃₂ O ₂	8.47	21.50	n- Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	17.30
24.05	Dodecanoic acid, methyl ester	C ₁₃ H ₂₆ O ₂	2.23	18.25	Eicosanoic acid	C ₂₀ H ₄₀ O ₂	5.76	23.35	Oleic acid	C ₉ H ₁₂	3.26
25.00	Eicosanoic acid	C ₂₀ H ₄₀ O ₂	1.62					23.42	Ricinoleic acid	C ₁₈ H ₃₄ O ₃	19.59
26.00	6-Octadecenoic acid, methyl ester, (Z)-	C ₁₉ H ₃₆ O ₂	2.00					25.13	9-Octadecenoic acid, 12-hydroxy-, Methyl ester, [R-(z)]-	C ₁₉ H ₃₆ O ₃	8.88
27.81	9-Octadecenoic acid, 12-hydroxy-,methyl ester, [R-(z)] -	C ₁₉ H ₃₆ O ₂	1.61								
	Total composition of fatty acids		52.47				59.76				66.48

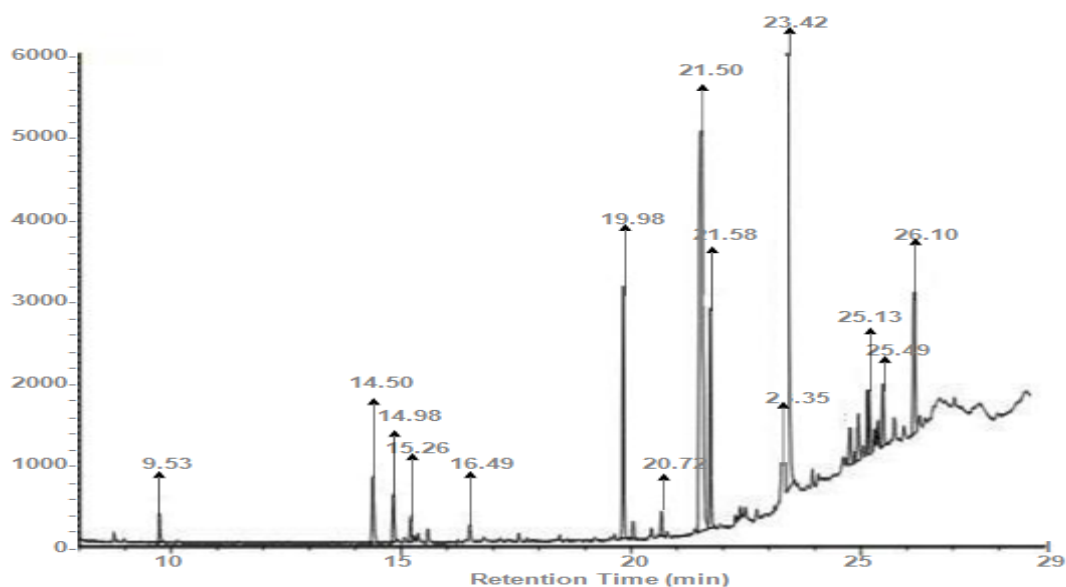


Fig. 3. GCMS chromatograms for RCME

3.4 The FTIR Analysis

In Table 3, Fourier Transform Infra-red (FTIR) foremost IR peak region that indicated chemical

changes were found at 1600-3600 cm^{-1} . The Fourier Transform Infra-red (FTIR) spectroscopic analysis results for biodiesel showed the foremost IR peak region that indicated chemical

Table 3. The foremost IR peak region indicated chemical changes

Frequency (cm-1)	Functional groups	Observed vibration bands/peaks (cm ⁻¹)		
		RJME	RNME	RCME
3600 – 3200	O–H stretching vibration (alcohol)	3456.55	3450.77	3444.98
29500 - 2840	C-H aliphatic stretching vibration.	2924.18	2824.18	2928.04
1750 – 1720	C=O stretching (ester)	1745.64	1745.64	1745.64
1465 – 1440	- CH ₃ bend	1464.02	1460.16	1464.02
1390 – 1365	O-CH ₂ bend	1377.22	1377.22	1377.22
1200 – 1020	C-O stretching vibration	1166.97	1165.04	1166.97

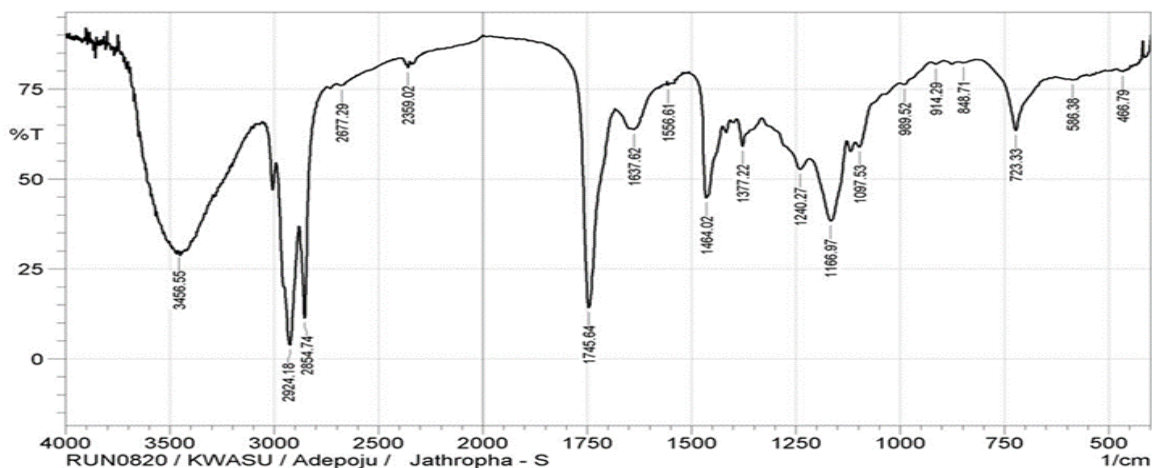


Fig. 4. The FTIR spectrum of RJME

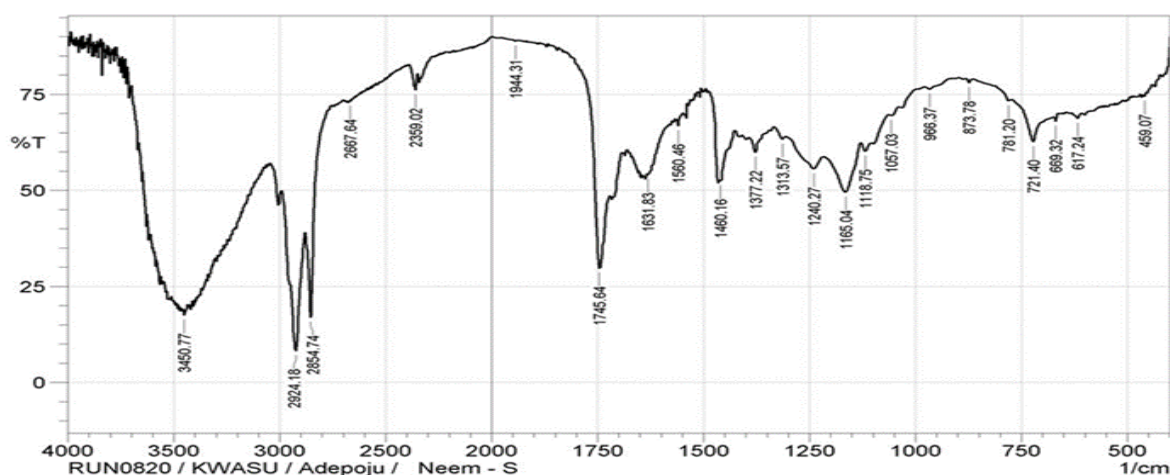


Fig. 5. The FTIR spectrum of RNME

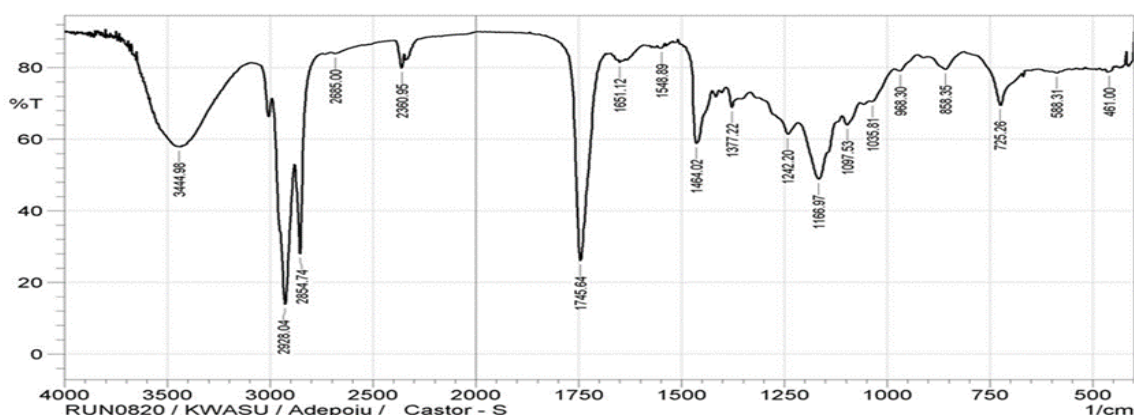


Fig. 6. The FTIR spectrum of RCME

In Figs. 4 - 6, the spectral bands for RJME, RNME AND RCME with 1377.22 cm^{-1} showed the corresponding presence of glycerol group O-CH_2 (mono, di and triglycerides). The IR spectral 1745.64cm^{-1} corresponds to the presence of ester carbonyl group. The IR band $1460.16 - 1464.02\text{ cm}^{-1}$ was as a result of its uneven bending and likewise matches with $-\text{CH}_3$

4. CONCLUSIONS

The work investigated the production of neat biodiesel with quality evaluations after refining processes of degumming, alkaline and bleaching treatments. The refining processes effectively decreased the levels of free fatty acid and associated impurities in the vegetable oils which significantly improved biodiesel properties. Comparisons of quality parameters of refined oil samples (RJO, RNO and RCO) showed quality enhancement of samples with saturated acids in smaller quantities which confirm

their suitability as feedstock. The utilization of RJO; RNO and RCO can therefore reduce the high cost of biodiesel which could possibly reduce wastes.

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COMPETING INTERESTS

There is no competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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