Original Research Article

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> Performance evaluation of ecofriendly biolubricant obtained from waste cooking palm oil

6 ABSTRACT (ARIAL, BOLD, 11 FONT, LEFT ALIGNED, CAPS)

The use of vegetable oils as renewable sources for the production of ecofriendly biolubricant is gaining support of the renewable energy researchers and petrochemical engineering. These studies were carried out in order to evaluate the quality assessment parameters of ecofriendly biolubricant obtained from waste cooking palm oil (WCPO). The crude sample was filtered, and dried over Na₂SO₄ crystals for 72 hours. The quality assessment parameters of the crude sample were determined in order to authenticate its potentials for the production of multigrade lubricating oils. Viscosity index of 208 was obtained from the crude extract, while kinematic viscosities at 100 °C and 40 °C were obtained as 8.26 cSt and 36.98 cSt respectively. A mixture of 68.75 % (crude extract), 23.75 % (SN 500), and 7.50 % (additives), was used for the production of the biolubricant. The biolubricant produced has kinematic viscosities at 100 and 40 °C of 10.72 cSt and 59.32 cSt respectively; viscosity index of 173; flash point of 234 °C ; pour point of -31 °C ; acid value of 21.04 mgKOH/g; and iodine value of 127.85 l₂g/100g. The produced biolubricant was found useful as renewable and ecofriendly lubricating oil.

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Keywords: Ecofriendly; biolubricant; palm oil; production.

1. INTRODUCTION

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Current developments in human lifestyle and significant population growth have gradually lead to increased in the consumption of fossil fuels. Excessive consumption of non-renewable fuels also means depletion of fossil oil reserves. Depletion of the world's energy reserves; increase in petroleum price; increase in environmental awareness; growing regulations over environmental pollution and contaminations resulting from emission of greenhouse gases (GHG) (such as carbon monoxide, carbon dioxide, heavy metals, volatile organic matters and polyaromatic hydrocarbons) have accelerated the development of renewable and biodegradable energy sources [1-6]

Accidental and deliberate lubricant losses to the environment by leakages, spills and evaporation, are major concerns regarding environmental health and pollution. Anjana and Preeti [7] reported that about 10 millions tonnes of petroleum products enter the environment annually inform of urban runoff, refinery processes, spills, industrial and municipal wastes, and condensation from marine engine exhaust. It is therefore mandatory on the producers of lubricants, for certain applications, to enforce strict specifications on toxicity, occupational health and safety, biodegradibility, and emissions.

Fats and oil have recently been found useful in biolubricating processes to produce tailor-made products [8-10]. According to Erhan *et al.* [11], 85-90 % of the total global lubricant production comes from non-renewable petroleum sources.

Biolubricants act as antifriction which ease smoother working while reducing the risks associated with
 machine failures and maitaining optimum machine operations. They are essential for heat transfer,
 power transmission, lubrication, and corrosion inhibition in machinery [12].

The main purposes of lubrication are to protect the surfaces from corrosion; reduce oxidation; reduce wear due to contact; prevent heat loss from the surfaces in contact; to act as insulator in transformer applications; act as sealing agents against dust, dirt and water and improve efficiency of machines [13,14].

According to Amit and Amit [12], the main characteristic of any lubricant is viscosity, which is responsible for preventing friction between two surfaces in contact. Other important qualities used for

selecting lubricants include temperature stability, prices, availability, environmental friendliness,

39 toxicity, chemical stability, corrosiveness, flammability, and compatibility.

40 Lubricating oil is composed of base stocks and additives formulated to enhance the performance of

41 the oil. A lubricant is primarily base oil (75-90 %) and additive formulated to improve its performance

42 properties such as pour point, viscosity index and oxidative stability [7].

The remains of palm oil which has been used for food processing is called waste cooking palm oil (WCPO). It contains free fatty acids (FFAs) with water and other impurities.

45 Waste vegetable oils are less expensive than fresh vegetable oils and, therefore, a promising 46 feedstock for the synthesis of biolubricants [6,15-19]. Waste vegetable oils are generated from 47 eateries, food industries and restaurants around the world [6,19, 20] and their main use is in the 48 production of animal feeds, manufacture of biolubricants, and manufacture of soaps [16].

Waste cooking palm oil essentially consists of triglycerides, unsaturated fatty acids, with glycerol
 [6,18, 20]. Details of some fatty acids in vegetable oil and quality assessment parameters are
 presented in Table 1.1 and Table 1.2 respectively:

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53 Table 1.1: Fatty acids compositions of some vegetable oils

Vegetable oils	Palmitic acid (16:0)	Stearic Acid (18:0)	Oleic Acid (18:1)	Linoleic Acid (18:2)	Linolenic (18:3)	Reference
WCO	28.91	0.93	26.51	27.44	4.60	[20]
Palm oil	41.50	2.70	40.60	11.90	0.30	[21]
Palm oil	39.32	4.36	42.52	11.35	-	[22]

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Keys: (-) = Not Applicable

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58 Table 1.2: Quality assessment parameters of some vegetable oils

Oils	KV @100°C (cSt)	KV @40°C (cSt)	VI	Density @15°C (g/cm³)	FP (°C)	PP (°C)	AV (mgKOH/ g)	IV (I₂g/ 100g)	Refe renc e
Palm oil	-	52.13	-	0.91	-	-	6.35	-	[6]
Palm oil ester	10.96	50.33	214	-	253	5	-	-	[6]
WCO lub	8.50	36.70	220	-	-	-2	1.56	-	[22]
Palm kernel	7.80	34.90	210	0.92	322	-15	0.05	89.60	[23]

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Keys: (-) = Not Applicable; AC = Acid Value; FP = Flash Point; KV = Kinematic

Viscosity; **IV** = Iodine Value; **PP** = Pour Point; and **VI** = Viscosity Index

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64 Depletion of the world's energy reserves and growing regulations over environmental pollution and

- 65 contaminations resulting from emission of greenhouse gases have accelerated the development of
- 66 renewable and biodegradable energy sources

67 This research focused on the production of ecofriendly biolubricant using waste cooking palm oil as

68 renewable base stock.

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70 2. MATERIAL AND METHODS

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72 The materials used for this research include analytical balance; thermometer; viscometer; FTIR 73 spectroscopy machine; and pensky martens flash point analyser. The reagents used were analytical 74 grade methanol; potassium hydroxide; potassium iodide; sodium thiosulphate; and others.

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77 2.1 Sample Collection and Treatment

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The waste cooking palm oil was obtained from Owuna Catering & Restaurant Services, IkorOchekwu, Apa Local Government Area, Benue State, North Central, Nigeria.

Solid particles, salt, pepper and spices in the crude sample were removed by filtration. The crude sample containing water and other impurities was centrifuged at 15000 rpm. Water content of crude sample was dried over sodium sulphate, Na₂SO₄, crystals for 72 hours. Na₂SO₄ forms clumps when they absorb water. After standing for a short period of time, the crystals are removed by decantation. Then the WCPO mixed with n-hexane (1:3 oil/hexane, volume ratio) to remove the remaining impurities [20].

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88 2.2 Production of biolubricant

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Waste Cooking Palm Oil (68.75 % wt), SN 500 (23.75 % wt), and additives (7.50 % wt) were mixed
together in a conical flask [24,25]. The mixture was done at the temperature of 40 °C, agitation speed
of 600 rpm for 15 minutes. The additives contain anti-oxidants, anti-foam, anti-wear, detergent,
dispersant, viscosity index improver, and pour point depressant.

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95 2.3 Quality assessment parameters of the crude oil and biolubricant

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97 The quality assessment parameters of the crude waste cooking palm oil and the produced 98 biolubricants were determined as detailed below:

99 The sample of oil was poured into a clean measuring cylinder and accompanying air bubbles were 100 allowed to settle. The oil sample was stirred continuously with a thermometer and the temperature 101 was recorded to the nearest 0.25 °C immediately the sample stabilised. Once the thermometer was 102 removed, the hydrometer was depressed about two scale division into the oil and it was released. 103 Sufficient time of about 10 minutes was allowed for the hydrometer to remain stationery in the oil and 104 at this point, the hydrometer reading was taken. The actual value of the density was read at 15 °C 105 from the petroleum measurement table using the value of the specific gravity observed from the 106 experiment at the observed temperature [24].

107 The oil was poured into a viscometer and placed in water bath and allowed to stand until bath 108 equilibrium temperature was attained. The water bath was maintained at 40 and 100 °C respectively 109 for kinematic viscosities measurement. When the equilibrium temperature was attained, the head 110 level of the test sample was adjusted, using a suction pump, to the capillary of the viscometer tube 111 above the first timing mark. The stop watch was started when the meniscus of the sample was at the 112 upper timing mark, and stopped when the meniscus got to the lower timing mark. The time taken for a given volume of oil to move from the upper timing mark to the lower timing mark was recorded. The 113 114 test, which is in accordance with ASTMD-446, was also carried out at 40 and 100 °C respectively for 115 all the blended oils [24]. The kinematic viscosities were calculated using equation 2.1:

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118 119 **K** is the calibration constant of the viscometer (cSt/s) **T** is time (s)

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Viscosity index (VI) of the oil sample was obtained using values of kinematic viscosity obtained at 40
 and 100 °C with standard measurement table as determined by ASTMD-2270 method.

The pour point test was conducted according to the method discribed in ASTMD-97 with accuracy of ±3 °C with the pour point tester. The tester has a minimun temperature of -68 °C with methanol as cooling agent. 45 cm³ of oil sample was poured into a test jar to the leveled mark. Then the tester was cooled to -37 °C. While cooling the tester, the sample jar was heated to 45 °C using a water bath maintained at 45 °C. The sample jar was cooled with another water bath to a temperature of 27 °C. When the pour point tester had reached -36 °C, the sample jar was placed in a horizontal position in the hole at the top of the tester until the oil showed no movement. The pour point temperature was taken when the oil showed no movement when the test jar was held in a horizontal position for 5 seconds.

The flash point was determined by heating a cup containing the oil sample. A flame was presented at regular interval, starting with atleast 28 °C below the expected flash point to the liquid surface. The bulb of the thermoneter was immersed in the sample. Occurrence of flash in the vessel indicates that the temperature of the tested oil has reached (or exceeded) the flash point. The flash point was measured at room temperatures with Cleavelend "open-cup" and thermometer immersed in it. This test conforms to ASTMD-92 as cited by Janes and Chaineaux [26] and Obasi *et al.* [24].

The sample of oil, 2.0 g, was placed in a dry 250 cm³ conical flask. 50 cm³ of ethanol and few drops (2-3) of phenolphthalein indicator were added. The mixture was heated at 60 °C in a water bath for 10 minutes and then cooled. The mixture was titrated with 0.1M KOH to the end point with consistent shaking, a dark pink colour was observed and the volume of 0.1M KOH was recorded as the titre value [27,28]. Acid value was calculated using equation 2.2:

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Acid value $\left(\frac{mgKOH}{g\,sample}\right) = \frac{Volume\,KOH(cm^2) \times N\,KOH(mmol/cm^2) \times 56.1(mg/mmol)}{sample\,Weight(g)}$2.2

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Sample of 2.00 g of oil was weighed into a conical flask and 25 cm³ of carbon tetra chloride (CCl₄) 148 was added to dissolve the oil. Then 25 cm³ of Dam's reagent was added to the mixture using a safety 149 150 pipette in fume chamber. Stopper was inserted and the content of the flask was vigorously swirled. The flask was placed in the dark for 1 hour. Then, 20 cm³ of 10% aqueous potassium iodide (KI) and 151 152 125 cm³ of water were added using a measuring cylinder. The solution was titrated with 0.1M sodium 153 thiosulphate (Na₂S₂O₃) solutions until the vellow color almost disappeared. Few drops of 1% starch 154 solution indicator were added and titration continued by adding sodium thiosulphate drop wise until 155 blue coloration disappeared after vigorous shaking. The same procedure was used for blank test 156 [27,28]. The iodine value (I.V) was determined by using equation 2.3: 157

$$Iodine \ Value = \frac{12.69 \ x \ Nof \ Sodiun \ Thiosulphate \ x \ (V_0 - V_1)}{Wt \ of \ Sampl \ (g)} \dots \dots \dots 2.3$$

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 V_0 = Volume of sodium thiosulphate used for blank

V₁ = Volume of sodium thiosulphate used for determination

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163 **2.4 Fourier Transform Infrared Spectroscopy Analyses of the Oils**

Functional group (esters) analyses for the oils were carried out using FTIR assay method, with Agilent Technologies, at the Multi Users Science Research Laboratory (MUSRL), Ahmadu Bello University, Zaria. The sample of oil was placed on sodium chloride (NaCl) plate (sample holder) forming thin layer of the sample. Second sodium chloride was taken and mounted on the first sodium chloride plate. All the assay were carried with wave number set at a range of 4000 cm⁻¹ to 650 cm⁻¹ [29].

169 2.5 Gas Chromatography Mass Spectroscopy Analyses of the Oils

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The oils were analysed by gas chromatograph equipped with mass spectrometer at the Multi Users
Science Research Laboratory (MUSRL), Ahmadu Bello University, Zaria.

173 The GC-MS system was equiped with an Econo-Cap EC-WAX capillary column (30.0 m in length x 250µm in diameter x 0.25 µm in film thickness). The oven temperature was set initially at 50 °C for 3 minutes, and then increased at 10 °C/minute to 210 °C and was held at 210 °C for another 9 minutes. The temperature for front inlet (splitless mode) was set at 255 °C. Helium was used as carrier gas with a flow rate of 12 cm³/minute. The split ratio was set at 1:1, and 1.0 µL of the sample was injected into the GC system [21].

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181 **3. RESULTS AND DISCUSSION**

182 183 The chemical compositions of the crude waste cooking palm oils were presented in Table 3.1. It was 184 found that unsaturated fatty acids of carbon chain length C₁₈ were found more in the waste cooking palm oil (50.96 %) compared to saturated fatty acid of carbon chain length C_{16} - C_{18} (27.25 %). In order 185 186 reviews, palm oil was found to contain 53.87 and 43.60 % of unsaturated and saturated fatty acids 187 respectively, as reported by Ebtisam et al. [22], and waste cooking oil was found to contain 58.55 and 29.84 % of unsaturated and saturated fatty acids respectively, as reported by Hassani et al. [20]. This 188 189 indicates that waste cooking palm oil is susceptible to oxidation reactions due to the degree of 190 unsaturation of the carbon atoms.

The quality assessment parameters of the crude waste crude palm (Table 3.2) showed that the crude sample has kinematic viscosities at 40 and 100 °C of 36.98 and 8.26 cSt respectively, while the viscosity index was found to be 206. This indicates that the crude waste cooking palm oil could be used for the production of multigrade motor car engine oils (Appendix I)

195 In order to achieve formulations that could compete with commercially available biolubricating oils, the 196 crude waste cooking palm oil (68.75 %) was contaminated with mineral based SN 500 (23.75) and 197 additives (7.5), as a modification of a method described by Obasi *et al.* [24].

198 The kinematic viscosity of waste cooking palm oil biolubricant was found to be 59.32 cSt at 40 °C and 199 10.72 cSt at 100 °C (Table 3.2). Similar results were found for Palm oil TMP lubricating oil as 50.33 200 cSt at 40 °C and 10.87 cSt at 100 °C [22], Palm kernel TMP ester as 34.90 at 40 °C and 7.8 cSt at 100 °C [23], and waste cooking oil as 36.7 cSt at 40 °C and 8.50 cSt at 100 °C [6]. This indicates that 201 202 WCPO biolubricant has high internal resistance to flow compared to palm kernel TMP ester [23] and 203 waste cooking oil [6], and low internal resistance to flow compared to Palm oil TMP lubricating oil [22]. 204 The viscosity index of WCPO biolubricant was found to be 173 (Table 3.2), which is lower compared 205 to viscosity index of palm oil TMP ester, 214 [22], palm kernel TMP ester, 210 [23], and waste cooking 206 lube oil, 220 [6]. This shows that WCPO biolubricant will experience greater changes in its viscosity 207 with change in temperatures compared to palm oil TMP, palm kernel TMP ester, and waste cooking 208 lube oil. The viscosity index of the produced WCPO biolubricating oils was found to be within standard 209 specification for engine oils, >90 [30,31] which indicates that waste cooking palm oil biolubricant could 210 be used as ecofriendly biolubricating oils.

The flash point of the produced biolubricant was found to be 234 °C (Table 3.2). In similar reviews, flash point of palm kernel TMP ester was found to be 322 °C [23], while that of palm oil TMP ester was found to be 253 °C [22]. This indicate that palm kernel TMP ester and palm oil TMP ester have more carbon atoms in their molecular structures compared to the produced WCPO biolubricating oils. The produced WCPO biolubricating oil could be safely used in vehicles without presumable vehicular or engine failures owing to its conformity with standard specification [30,31].

The pour point of WCPO biolubricating oil was found to be -31 °C (Table 3.2). In similar reviews, pour 217 point of palm oil TMP ester was found to be 5 °C [22]; that of palm kernel TMP ester was found to be -218 15 °C [23]; while that of WCO was found to be -2 °C [6]. The large differences in the pour points of 219 220 those reviewed compared to that of the produced WCPO biolubricant could be as a result of none 221 formulation of additives (pour point depressants) incorporated in the production of WCPO 222 biolubricating oils which were absent in those reviewed biolubricants. This indicates that formulation of 223 pour point depressants (PPDs) in vegetable oils could boost their usefulness for various applications 224 at very low temperatures.

Acid value of WCPO biolubricating oil was found to be 21.04 mgKOH/g (Table 3.5). The acid value is higher compared to acid value of 0.05 mgKOH/g for palm kernel TMP ester reported by Robiah *et al.* [23], acid value of 1.56 mgKOH/g for WCO lube oil reported by Weimin and Xiaobo [6]. Though the acid value of WCPO biolubricant is within standard specifications of acid value of 0.20 – 50.00 mgKOH/g [30,31], calculated amounts of anti-corrosion and anti-oxidants are required as additives in order to enhance the usefulness of the produced WCPO biolubricant for any particular application. These additives will inhibit the negetive effects of corrosion and oxidation during their usages.

The iodine value of WCPO biolubricant was found to be $127.85 l_2/100g$ (Table 3.2). The iodine value of the produced WCPO biolubricant is higher compared to that of palm kernel TMP ester of 89.60 $l_2/100g$ reported by Robiah *et al.* [23]. This shows that WCPO biolubricant has more methylene interrupted double bonds and it is more susceptible to oxidation reactions than palm kernel TMP ester.

The absorption bands for C-H and $-CH_2$ for the crude WCPO and WCPO biolubricant occur at similar wavenumbers of 2922 cm⁻¹ and 2855 cm⁻¹ (Table 3.3) respectively. This is an indication of alkane functional group in the samples. Alkane functional group was found for biodiesel within the range of 3000-2855 cm⁻¹ as reported by Ebtism *et al.* [22]. The methyl group, $-CH_3$ bend, occurs at

wavenumber of 1375 cm⁻¹ for both crude WCPO and WCPO biolubricant (Table 3.3). Alkene out of 241 plane, =C-H bend, was observed at 969 cm⁻¹ for WCPO biolubricant and at 961 cm⁻¹ for crude 242 243 WCPO. The alkene functional group is an indication of unsaturation of the crude WCPO and WCPO biolubricant. The carbonyl functional group, C=O, was observed at 1744 cm⁻¹ for both samples (Table 244 3.3). The C-O stretching vibration occurring at 1159 cm⁻¹ for both WCPO and WCPO lube is an 245 indication that the carbonyl group, C=O, is for ester since there is no visible O-H band for both 246 247 samples. In similar reviews, ester group was reported at 1744 cm⁻¹ for palm oil based TMP ester [22], and 1745 cm⁻¹ for waste cooking oil [32]. The wavenumber at 1710 cm⁻¹ observed in both crude 248 249 WCPO and WCPO biolube suggests C=O stretching vibration. This is a possible indication of the 250 presence of carboxylic acid functional group in both crude WCPO and WCPO biolubricant samples 251 even though their corresponding O-H functional group was not observed (Table 3.3): and the high 252 acid values observed in both samples (Table 3.2) attest to this claim. A peak was observed at 1975 253 cm⁻¹ for WCPO lube (Table 3.3). This unique peak occurring with the range of 2270-1950 cm⁻¹ 254 suggests the presence of X=C=Y functional group of alkenes, isocyanates, or isothiocyanates. Details 255 of the infrared spectra of crude WCPO and WCPO biolubricant are found in Figure 3.1 and Figure 3.2 256 respectively.

The gas chromatography mass spectra indicated the presence of palmitic acid (16:0); stearic acid (18:0); oleic acid (18:1); linoleic acid (18:2); and linolenic acid (18:3), in both waste cooking palm oil samples (Table 3.1)

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262 Table 3.1: Fatty Acid Composition of Crude Vegetable Oils

	Oils	Palmitic acid (16:0)	Stearic Acid (18:0)	Oleic Acid (18:1)	Linoleic Acid (18:2)	Linolenic (18:3)
	Crude WCPO	25.75	1.53	23.11	25.05	2.80
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263 Keys: (-) = Not Applicable; **CSO** = Calabash Seed Oil; **WCPO** = Waste Cooking Palm Oil

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265 Table 3.2: Quality Assessment parameters of crude WCPO and biolubricant

Crude Oils	KV @10 0°C (cSt)	KV @40°C (cSt)	VI	Density @15°C (g/cm³)	FP (°C)	PP (°C)	AV (mgKOH/ g)	IV (l₂g/ 100g)
Crude WCPO	8.26	36.98	206	0.91	153	-9	39.91	135.40
WCPO Biolube	10.7	59.32	173	-	234	-31	21.04	127.85
Standard ^[30,31]	2 >4.1	>28.80	>90	<1.00	>150	<-5	0.20-50	50-140

Keys: (-) = Not Applicable; CSO = Calabash Seed Oil; AV = Acid Value; FP = Flash Point; KV =
Kinematic Value; IV = Iodine Value; PP = Pour Point; VI = Viscosity Index; WCPO = Waste Cooking
Palm Oil

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272 Table 3.3: FTIR Analyses of Crude Extracts and Biolubricants

Bonds	Wave Number (cm ⁻¹)	Bond Description	Functional Group	Samples
C-H	2922	Alkane stretch	Alkane	WCPO, WCPO biolube
-CH ₂ -	2855	Alkane stretch	Alkane	WCPO, WCPO biolube
-CH ₂ -	1461	Alkane bend	Alkane	WCPO, WCPO biolube
$-CH_3$	1375	Methyl bend	Alkane	WCPO, WCPO biolube
-(CH ₂)4-	723	4 or more –CH2- (chain)	Alkane	WCPO, WCPO biolube
C=O	1710	Carbonyl stretch	Carboxylic acid	WCPO, WCPO

UNDER PEER REVIEW



Formulation waste cooking palm oil with mineral based oil, SN 500, and additives, confirmed the usefulness of WCPO lube oil for the production of multigrade ecofriendly biolubricant that is comparable to the available commercially synthesized multigrade biolubricants. The viscosity index and pour point of the produced waste cooking palm oil biolubricant (173 & -31 °C respectively) are good indication of the oil's potential to be used for operations that requires variation of temperature ever as low as -30 °C. FTIR analyses of the produced waste cooking palm oil biolubricant confirmed the presence of ester functional group: esters are good starting materials for the formulation of lubrication oil because of their good lubricity, while GC-MS revealed both the presence of saturated and unsaturated fatty acids in the sample of the waste cooking palm oil. The biolubricant produced

from watse cooking palm oil is renewable, biodegradable and is ecofriendly.

296 CONSENT (WHERE EVER APPLICABLE)

298 Not applicable

301 ETHICAL APPROVAL (WHERE EVER APPLICABLE)

303 Not applicable

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382 **DEFINITIONS**

- 383384 CSO = Calabash Seed Oil
- 385 **FTIR** = Fourier Transform Infra-red Spectroscopy
- 386 **GC-MS** = Gas Chromatography Mass Spectroscopy
- 387 WCPO = Waste Cooking Palm Oil
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390 APPENDIX

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- 393 Appendix I: Multigrade engine oil specification

TYPICAL SPECIFICATIONS

SAE Viscosity Grade	5W-20	5W-30	10W-30
API Service	SN/GF-5	SN/GF-5	SN/GF-5
API Gravity Ibs/gal	32.5/7.18	33.2/7.15	32.4/7.19
Viscosity @ 40°C, cSt	51	57	67
Viscosity @ 100°C	8.8	10.5	10.6
Viscosity Index	152	176	147
CCS Vis, cP @ °C	5,700 @ -30	5,600 @ -30	5,900 @ -25
MRV-TP1 Vis, P @ °C	21,500 @ -35	25,000 @ -35	20,000 @ -25
Pour Point, °F (°C)	-45 (-49)	-42 (-44)	-39 (-38)

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