



Use of Lagenaria Siceraria Seed Oil for the Production of Environmentally Friendly Biolubricant

Owuna Friday Junior^{1,*}, Dabai Musa Usman¹, Sokoto Muhammad Abdullahi¹, Muhammad Chika¹, Abubakar Aminu Lailaba²

¹Department of Pure and Applied Chemistry, Faculty of Science, Usmanu Danfodiyo University, Sokoto, Nigeria

²Department of Biochemistry, Faculty of Science, Usmanu Danfodiyo University, Sokoto, Nigeria

Email address:

Jzr4do@gmail.com (O. F. Junior)

*Corresponding author

To cite this article:

Owuna Friday Junior, Dabai Musa Usman, Sokoto Muhammad Abdullahi, Muhammad Chika, Abubakar Aminu Lailaba. Use of Lagenaria Siceraria Seed Oil for the Production of Environmentally Friendly Biolubricant. *American Journal of Applied and Industrial Chemistry*. Vol. 2, No. 1, 2018, pp. 1-7. doi: 10.11648/j.ajaic.20180201.11

Received: September 3, 2018; **Accepted:** September 14, 2018; **Published:** October 12, 2018

Abstract: The needs for environmentally friendly energy sources, coupled with the depleted fossil oil reserves, have generated interests in the search for world's alternative energy sources, and vegetable oils seem to be the best options. This study assesses the use of Lagenaria siceraria seed oil (LSSO) as a potential source of environmentally friendly biolubricant. The LSSO was extracted from the Lagenaria siceraria seed (LSS) using Soxhlet apparatus, and a yield of 41.70% was obtained, with n-hexane as the extracting solvent. The physicochemical parameters of LSSO were assayed to confirm its potential for the production of environmentally friendly biolubricant. The result showed that the kinematic viscosities of 6.24 ± 0.10 cSt (at 100°C) and 21.74 ± 0.02 cSt (at 40°C) were obtained, while the viscosity index (266 ± 0.00) was also obtained. An experiment was designed (Mixture Design Method using Minitab 17) to obtain the proportion of LSSO (47.50%), SN 500 (47.50%), and additives (5.00%) that gave the mixture with the optimum physicochemical parameters of the produced biolubricant. The produced LSSO biolubricant had kinematic viscosities 9.59 ± 0.02 cSt (at 100°C) and 49.62 ± 0.14 cSt (at 40°C), a viscosity index of 181 ± 0.01 , flash point of $230 \pm 1.20^\circ\text{C}$, pour point of $-30 \pm 0.03^\circ\text{C}$, acid value of 2.72 ± 1.03 mg KOH g^{-1} , and iodine value of 1.28 ± 1.40 mg I_2 g^{-1} . The LSSO biolubricant has physicochemical parameters that are within the standard for engine oils and could compete favourably with the synthesised environmentally acceptable lubricating oils.

Keywords: Production, Environment, Biolubricant, Lagenaria Siceraria, Viscosity

1. Introduction

Global attention is gradually shifting to the use of vegetable oils as promising alternative sources of energy due to depletion of fossil oil reserves, environmental challenges caused by the use of fossil oils, and non-renewability of the conventional petroleum energy sources [1-7]. In addition to the renewability and environmentally friendly nature of the vegetable oils, they can also be produced locally [5, 7] thereby serving as sources of income to rural communities.

Vegetable oils are gaining recognition as the most promising energy sources and a blend of small quantity of vegetable oils with fossil oils has significant impact on the environment and economy [5, 7-9].

Lagenaria siceraria is a climbing ornamental plant planted

largely in Northern Nigeria. It is a white flowered gourd known as long melon, and a vine grown for its fruits which can be used as vegetable when harvested young or used as bottle (utensil or pipe) when harvested dried, while the fresh fruit has a light-green smooth skin and white flesh. The plant is commonly found around Gombe, Jigawa, Sokoto, Kebbi and Zamfara States of North-western Nigeria [10-13]. After harvesting, only few quantities of the Lagenaria siceraria seeds (LSS) are preserved for planting. The rest non-edible seeds are wasted. These non-utilised seeds could find relevance as renewable and ecofriendly sources of energy [13].

This study was focused on the utilisation of LSSO as potential sources of environmentally friendly biolubricating oil.

2. Methods

2.1. Sample Collection and Treatment

The Lagenaria siceraria seeds (LSS) were purchased from Sokoto Metropolis, Sokoto State, North-western Nigeria, and were authenticated by Botany Unit, Department of Biological Science, Faculty of Sciences, Usmanu Danfodiyo University, Sokoto. The seeds were dehulled, dried, grounded into powder, and sieved to obtain a homogeneous powder. The LSS (300.00 g) was weighed and preserved for oil extraction [12].

2.2. Oil Extraction

A Soxhlet extractor was set up with n-hexane as the

extracting solvent (in 500 cm³ round-bottomed flask). The powdered-LSS (50.00 g per each extraction) was placed in a thimble. The volume of the n-hexane used was determined by the ratio 6:1 (w:w% of solvent and sample). The solvent was boiled gently (using heating mantle), a reflux condenser was fitted (to cool the hot solvent), the mixture was digested at 60°C for 5 hours, while the condensed hot-solvent soaked the thimble containing the sample. The solvent siphoned into the flask when it reached the top of the siphon tube of the Soxhlet apparatus. The resulting oil and solvent mixture was filtered to remove the suspended solids [3, 5]. The mixture was placed in a rotary evaporator to evaporate the solvent and thus, LSSO was obtained for subsequent analyses. The percentage yield of the LSSO was calculated using equation (1):

$$\% \text{ Yield} = \text{Weight of Oil(g)} \times 100 / \text{Weight of the Sample(g)} \quad (1)$$

2.3. Biolubricant Production

The LSSO was a poor base oil to be used for the production of biolubricant its raw form due to its poor thermal and oxidative stability [7, 14], therefore, it was mixed with mineral-based oil and additives [7] for the produced biolubricant. In order to determine the best proportion of variables (LSSO, SN 500, and additives) that gave the LSSO biolubricant with the optimum physicochemical parameters, an experiment was designed (Mixture Design method of Minitab 17) and carried out in two different levels and two replicates. The mixture of LSSO, SN 500, and additives (equal amount of zinc dialkyldithiophosphate, poly alkylmethacrylate, and triethylenetetramine) was done in a conical flask at a temperature of 45°C, and stirred at 600 rpm for 15 minutes. A heating mantle equipped with a magnetic stirrer was used to attain a homogeneous mixture [7, 15, 16].

2.4. Physicochemical Analysis of LSSO and Biolubricant

2.4.1. Determination of Kinematic Viscosity

The oil was poured into a viscometer and mounted upright in the viscometric bath (maintained at 40 or 100°C). The oil in the tube was allowed to stabilise for 15 minutes. When the equilibrium temperature was attained, the oil level was adjusted, using a suction pump, to 7 mm above the upper mark of the viscometer tube. The time taken for the oil to move from the upper mark to the lower mark of the viscometer tube was recorded [7, 15]. The kinematic viscosity (KV) was calculated using equation 2:

$$KV \text{ (cSt)} = C \text{ (cSt s}^{-1}\text{)} \times t \text{ (s)} \quad (2)$$

Where,

KV is the kinematic viscosity

C is the calibration constant of the viscometer

t is the time taken for the oil meniscus to move from the upper timing mark to the lower timing mark of the viscometer tube.

2.4.2. Determination of Viscosity Index

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

2.4.3. Determination of Pour Point

The pour point of the oil was obtained with accuracy of $\pm 3^\circ\text{C}$ with the pour point tester. The tester has a minimum temperature of -68°C with methanol as cooling agent. The oil (45 cm³) was poured into a test jar to the levelled mark. Then the tester was cooled to -37°C . While cooling the tester, the oil jar was heated to 45°C using a water bath. The oil jar was cooled with another water bath to a temperature of 27°C . When the pour point tester had reached -36°C , the oil jar was placed in a horizontal position in the hole at the top of the tester and the pour point temperature was taken after 5 seconds when the oil showed no movement [7].

2.4.4. Determination of Flash Point

The flash point of the oil was determined by heating a cup holding the oil while presenting a flame on the surface of the oil at regular temperature, starting with a temperature of 28°C below the expected flash point of the oil. The bulb of the thermometer was immersed in the sample. The flash occurred in the cup containing the LSSO when the temperature of the oil had reached its flash point [15, 17].

2.4.5. Determination of Acid Value

The oil (2.00 g) was placed in a dry 250 cm³ -conical flask. 50 cm³ of ethanol and 2 drops 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 endpoint (with consistent shaking). A dark pink colour was observed and the volume of KOH used for the titration was recorded as the titre value [5, 7]. The acid value of the LSSO was calculated using equation 3:

$$\text{Acid value} \left(\frac{\text{mgKOH}}{\text{g sample}} \right) = \frac{\text{Volume KOH}(\text{cm}^3) \times N \text{ KOH}(\text{mmol}/\text{cm}^3) \times 56.1 (\text{mg}/\text{mmol})}{\text{sample weight} (\text{g})} \quad (3)$$

Where,
KOH is potassium hydroxide
N is the molar concentration of KOH
56.1 is the molecular weight of KOH

2.4.6. Determination of Iodine Value

The oil (2.00 g) was placed in a dry 250 cm³-conical flask. 25 cm³ of carbon tetrachloride (CCl₄) was added to dissolve the oil. Then 25 cm³ of Wijs' reagent was added (in the fume chamber) to the mixture using a safety pipette. The flask was stoppered and the content of the flask was vigorously shaken.

The flask was placed in the dark for 1 hour. Then, 20 cm³ of 10.00% aqueous potassium iodide (KI) and 125 cm³ of water were added using a measuring cylinder. The solution was titrated with 0.1M sodium thiosulphate (Na₂S₂O₃) solutions to a faint-yellow colour, 2 drops of 1.00% starch solution indicator were added, and titration continued by adding sodium thiosulphate drop-wise until blue colouration disappeared after vigorous shaking. The same procedure was repeated for the blank test [5, 7]. The iodine value (I.V) of the LSSO was determined using equation 4:

$$I.V = \frac{12.69 \times 0.1 \text{ M Na}_2\text{S}_2\text{O}_3 (\text{Blank cm}^3 \text{ Na}_2\text{S}_2\text{O}_3 - \text{Sample cm}^3 \text{ Na}_2\text{S}_2\text{O}_3)}{\text{Wt of Sampl} (\text{g})} \quad (4)$$

Where,
I. V is the iodine value
12.69 is the mass of iodine in 0.1M solution of iodine

2.5. Characterization of LSSO and LSSO Biolubricant

2.5.1. Fourier Transform Infrared Spectroscopy

Functional group (esters) analysis for the LSSO and LSSO biolubricant were carried out using FTIR assay method. The oil was placed on sodium chloride (NaCl) plate (sample holder) forming thin layer of the sample. A second sodium chloride layer was mounted on the first sodium chloride plate. All the analyses were carried with wave number set at a range of 4000 cm⁻¹ to 650 cm⁻¹ [7, 18].

2.5.2. Gas Chromatography Mass Spectroscopy

The oils were analysed by gas chromatograph equipped with mass spectrometer. The GC-MS system was equipped 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, increased at 10°C/minute to 210°C and held at 210°C for

another 9 minutes. The temperature for front inlet (splitless mode) was set at 255°C and helium was used as carrier gas with a flow rate of 12 cm³ min⁻¹, while the split ratio was set at 1:1. The analysis of the chemical compositions of the oil sample was carried out by injecting 1.0 µL of the sample solution (blending the oil sample with a prepared internal standard of GC i.e. methyl heptadecanoate) [7]. The percentage composition by weight of the oil sample was determined using equation 5:

$$\text{Weight percent} (\%) = \left[\frac{\sum(A_i - A_R)}{A_R} \right] \frac{C_R V_R}{w} \quad (5)$$

Where,
A_i is the peak area from chromatogram of sample
A_R is the peak area from chromatogram of internal standard
C_R is the concentration of the internal standard
V_R is the volume of the internal standard
W is the total weight of the oil sample

3. Results

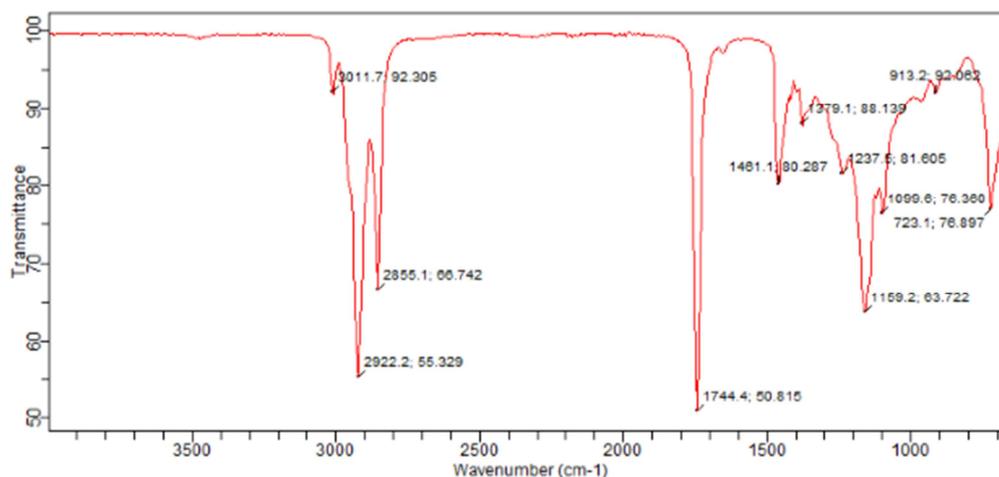


Figure 1. FTIR Spectrum of the LSSO.

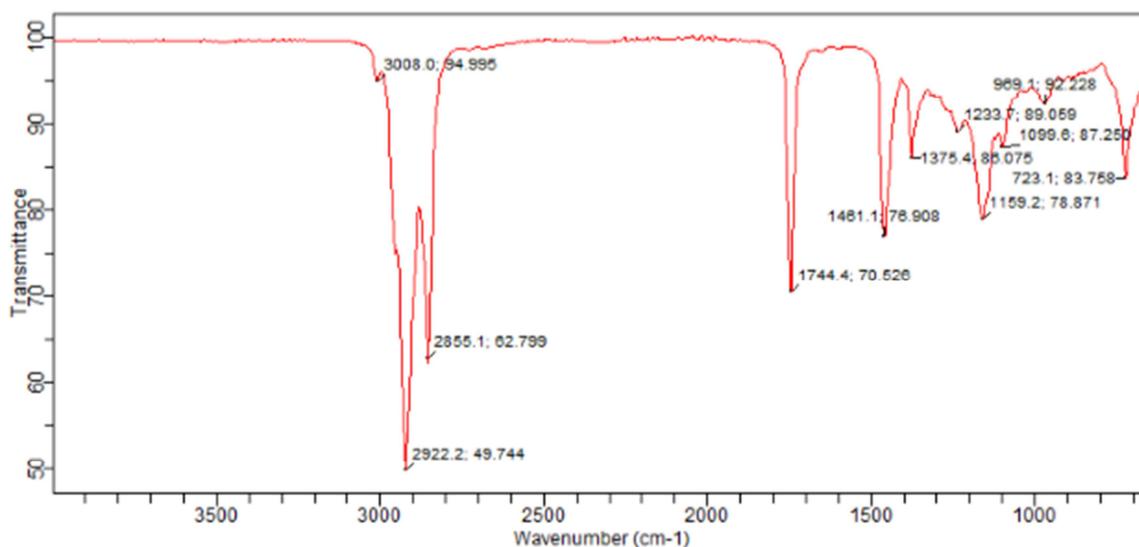


Figure 2. FTIR Spectrum of the Biolubricant.

Table 1. Optimisation Mixture for Biolubricant Production.

RunOrder	LSSO	SN500	Additive	KV @ 100°C	KV @ 40°C	VI	FP	PP	AV	IV
1	0.00	90.00	10.00	10.90	99.60	106	238	-11	2.61	0.94
2	68.75	23.75	7.50	8.30	36.41	252	145	-12	3.01	1.38
3	23.75	68.75	7.50	10.50	60.10	152	239	-15	2.88	1.05
4	90.00	0.00	10.00	7.10	21.74	270	151	-27	2.08	1.46
5	28.75	68.75	2.50	8.10	49.60	158	241	-18	2.85	1.16
6	68.75	28.75	2.50	7.90	40.30	178	142	-14	3.53	1.39
7	10.00	90.00	0.00	10.70	66.90	174	237	-13	1.70	0.98
8	28.75	68.75	2.50	8.10	49.51	159	240	-21	3.00	1.16
9	90.00	0.00	10.00	7.10	21.74	270	152	-27	2.05	1.45
10	23.75	68.75	7.50	10.55	60.10	151	237	-15	2.80	1.05
11	90.00	10.00	0.00	8.60	44.90	170	166	-28	3.77	1.47
12	68.75	28.75	2.50	7.90	40.29	177	143	-14	3.49	1.40
13	68.75	23.75	7.50	8.37	36.41	250	144	-12	3.91	1.38
14	47.50	47.50	5.00	9.59	49.62	181	230	-30	2.72	1.28
15	0.00	90.00	10.00	10.10	99.60	107	239	-11	2.61	0.95
16	90.00	10.00	0.00	8.60	44.90	170	164	-27	3.35	1.47
17	47.50	47.50	5.00	9.59	49.62	181	228	-29	2.74	1.28
18	10.00	90.00	0.00	10.70	66.90	174	237	-13	1.70	0.98

Key: LSSO = Lagenaria sinceraria Seed Oil; KV = Kinematic Viscosity (cSt); VI = Viscosity Index; FP = Flash Point (°C); PP = Pour Point (°C); AV = Acid Value (mg KOH g⁻¹); IV = Iodine Value (mg I₂ g⁻¹)

Table 2. Physicochemical Parameters of LSSO and the LSSO Biolubricant.

Parameters	Units	LSSO	Biolubricant	Engine oil range*
Kinematic Viscosity @ 40°C	cSt	6.24±0.10	9.59±0.02	> 4.10
Kinematic Viscosity @ 100°C	cSt	21.74±0.02	49.62±0.14	> 28.80
Viscosity Index		266±0.00	181±0.01	> 90.00
Flash Point	°C	145±1.01	230±1.20	> 150.00
Pour Point	°C	-10±0.01	-30±0.03	< -5.00
Acid Value	mg KOH g ⁻¹	3.08±1.20	2.72±1.03	0.2 – 50
Iodine Value	mg I ₂ g ⁻¹	1.44±0.22	1.28±1.40	50 – 140

Key: LSSO = Lagenarias inceraria Seed Oil; (±) = Mean Value Plus or Minus Standard Deviation (n = 3); (*) = Danjuma and Dandago, 2009; Muhammad *et al.*, 2015

4. Discussion

The optimum mixture for the produced LSSO biolubricant with the best physicochemical parameters was obtained from run number 14 (Table 1) as LSSO (47.50% wt), SN500 (47.50% wt), and additives (5.00% wt). The physicochemical parameters of the produced biolubricant were replicated three (3) times, and average values were recorded (Table 2).

The percentage yield of LSSO was found to be $41.70 \pm 0.43\%$ using n-hexane as the extracting solvent. This yield is higher compared to the yield of 22.32% obtained with petroleum ether as extracting solvent [19]. The yield is also higher compared to the yield of 29.33% obtained with n-hexane [20]. This shows that n-hexane is a better solvent for the extraction of LSSO than petroleum ether.

The kinematic viscosities of the produced LSSO biolubricant were found to be 49.62 ± 0.14 cSt at 40°C and 9.59 ± 0.02 cSt at 100°C (Table 2). Though the kinematic viscosities of the biolubricant are comparable to the value of 55.17 cSt at 40°C and 10.96 cSt at 100°C for Jatropha biolubricating oil [3], they are lower compared to a value of 11.22 cSt at 40°C for modified jatropha biolubricant [21], and a value of 20.15 cSt at 40°C for modified pongamia biolubricant [21]. The kinematic viscosity (at 40°C) of the biolubricant suggests its higher internal resistance to flow compared to that of modified jatropha biolubricant and modified pongamia biolubricant [21] even though the kinematic viscosities at 40 and 100°C were within the standard for engine oil (Table 2).

The viscosity index of the produced LSSO biolubricant was found to be 181 ± 0.01 (Table 2), and is lower compared to a value of 195.22 for jatropha biolubricant [3], a value of 225.36 for modified jatropha oil [21], and a value of 219.00 for modified pongamia oil [21]. Though the viscosity index was within the standard for engine oils [12, 19], LSSO biolubricant will experience greater changes in its viscosity with change in temperatures compared to jatropha lube oil, modified jatropha oil, and modified pongamia oil.

The flash point of the produced LSSO biolubricant was found to be 230 ± 1.20 (Table 2). The high flash point confirmed that LSSO biolubricant has oxygen atoms in its molecular structure, and hence has low risk associated with vapourisation during transportation and storage [7].

In addition, the pour point of the produced LSSO biolubricant was found to be -30 ± 0.03 (Table 2), and it is lower compared to a value of -7°C for jatropha biolubricant [3], a value of -03.00°C for modified jatropha oil [21], and a value of -02.00°C for modified pongamia oil [21]. The large differences in the pour points of those reviewed biolubricants and the produced LSSO biolubricant was as a result of additives formulated with the produced LSSO biolubricant which were absent in the others [7]. The pour points of the produced LSSO was also found within the standard for engine oils [12, 19].

The acid value of the LSSO biolubricant was found to be

2.72 ± 1.03 mg KOH g^{-1} (Table 2), and it is higher than a value of 0.05 mg KOH g^{-1} for modified jatropha oil [21] and a value of 0.13 mg KOH g^{-1} for modified pongamia oil [21]. This shows that requisite additives are necessary to enhance the usefulness of the produced biolubricant for any particular application [7, 9, 23].

The iodine value of the produced LSSO biolubricant was found to be 1.28 ± 1.40 mg I_2 g^{-1} (Table 2), and it is lower compared to a value of 22.00 mg KOH g^{-1} for modified jatropha oil [21] and a value of 21.41 mg KOH g^{-1} for modified pongamia oil [21]. This shows that the produced biolubricant has fewer methylene interrupted double bonds in its molecules and has high resistance to oxidation reactions than modified jatropha oil, and modified pongamia oil [7, 23].

The absorption bands for C-H and $-\text{CH}_2$ for LSSO and LSSO biolubricant were observed at 2922 cm^{-1} and 2855 cm^{-1} respectively, this is an indication of alkane functional group in both oils. The peaks occurring at 1461 cm^{-1} and 723 cm^{-1} were observed for both oils, indicating $-\text{CH}_2$ bends and $-\text{CH}_2$ long chains respectively. The methyl group ($-\text{CH}_3$ bend) was observed at 1379 cm^{-1} for both oils. Alkene stretching vibration ($=\text{C}-\text{H}$) was observed for LSSO and LSSO biolubricant at 3011 cm^{-1} and 3008 cm^{-1} respectively. Alkene stretching vibration ($=\text{C}-\text{H}$) was also observed at 3008 cm^{-1} for Calabash seed oil [24]. Alkene out of plane ($=\text{C}-\text{H}$ bend) was observed at 969 cm^{-1} for LSSO biolubricant and 913 cm^{-1} for LSSO. The alkene functional group is an indication of unsaturation of both oils [7, 14]. The carbonyl functional group ($\text{C}=\text{O}$) was observed at 1744 cm^{-1} for both oils, while C-O stretching vibration occurred at 1155 cm^{-1} , indicating that the $\text{C}=\text{O}$ is for ester since there is no corresponding O-H absorption [7]. Ester group was also found at 1744 cm^{-1} for Rubber Seed Oil TMP [18].

The gas chromatography mass spectra of the oils revealed the degree of unsaturation of the carbon atoms in various compounds, such as 1-O-pentadecyl 2-O-prop-2-enyl oxalate ($\text{C}_{20}\text{H}_{36}\text{O}_4$), 1-O-hexadecyl 2-O-prop-2-enyl oxalate ($\text{C}_{21}\text{H}_{38}\text{O}_4$), and Octadecyl prop-2-en-1-yl oxalate ($\text{C}_{23}\text{H}_{42}\text{O}_4$). Unsaturated compounds were also found in calabash seed oil [24], grape seed oil and pomegranate seed oil [25], and blighia sapids seed oil [26]. While ester groups are good for boundary lubrication [7, 26], chemical and physical properties of vegetable oils depend on the degree of saturation and unsaturation of the molecules of the compounds present in the vegetable oils [27]. This confirmed why vegetable oils are not relatively good for the production of biolubricant in their crude forms [7, 9, 14].

5. Conclusion

This research was conducted in order to confirm the use of LSSO for the production of environmentally friendly biolubricating oil. Oil yield of 41.70% was obtained from the

LSS. Formulations of LSSO biolubricant confirmed its potentials as good ecofriendly biolubricant. FTIR and GC-MS analyses of the oils confirmed the presence of ester functionality which indicates that LSSO is good starting material for the formulation of biolubricating oils. Physicochemical analysis of the produced LSSO biolubricating oil confirmed quality parameters that are within the standard for engine oils, and could be useful for any application. The produced biolubricant is environmentally friendly and it is applicable for any operation where commercially synthesised ecofriendly lubricants are in use.

6. Recommendations

Based on the findings of this analysis, the following recommendations were made:

- (1) There is need to explore the use of other extraction methods, other than soxhlet extraction, for the extraction of LSS in order to optimise its percentage yields.
- (2) Biodegradable tests and other tests such as anti-wear, corrosion test, anti-oxidant test, thermal stability test, should be carried out on the produced biolubricant in order to ascertain its degradability, oxidative stability, thermal stability, and usefulness in extreme pressure and temperature conditions during operations.
- (3) Optimisation of the interactions of various variables of the mixture (LSSO, SN 500, and additives) should be carried out in order to determine the variable of the mixture that has more influence on the Physicochemical parameters of the produced biolubricant.

Acknowledgements

The authors wish to acknowledge OVH Energies and Marketing (Kaduna, Nigeria) for the use of their laboratory facilities for physicochemical analysis, Sokoto Energy Research Commission (Sokoto, Nigeria) for the use of their laboratory for LSSO extraction, and TheRoyalFamily for the financial support.

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