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Production and Characterization of Biolubricant from *Cassia Sieberiana* (Decandole) Seed Oil Using Transesterification Reaction

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ABSTRACT: Biolubricant is a biopolymer that can be used as a base oil in a variety of applications such as biomass and bioenergy. Hence, the objective of this paper is the production and characterization of biolubricant from Cassia sieberiana (Decandole) seed oil via two step transesterification reaction between methylesters and trimethylolpropane (TMP) at a molar ratio of 3.5: 1 with a catalyst of 0.8 % w/w of the total reactant at a temperature of 120 C for 2:30 hrs with a percentage yield of 93%. The physicochemical properties of the produced oil were determined by FTIR spectrum and GC-MS analysis. The viscosity (at 40 and 100) are 44.20 cSt and 7.82 cSt with pour point (-9 C), and viscosity index (148.04 cSt) respectively. The GCMS analysis revealed the fatty acid composition of the based oil as palmitic acid, linoleic acid, lineolaidic acid, stearic acid, and that of the biolubricant: methyl oleate, 2-hydroxyl ethyl esters, and 22-tricosenoic acid with other traces of methyl ester. The analysis of the FTIR for the TMP triesters shows superior thermal and oxidative stability due to the ester functional group present. The produced oil was found to confirm with ISOVG viscose grade 32 and 46 for gear oil and other low temperature applications.

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The current increase in population growth in terms of human development and machinery has led to the depletion of world energy reserves and its effects on the climate have affected the geography of the atmosphere in terms of declining environmental health, climatic change and global warming (Dabai *et al*., 2018). By 2020 it was estimated that the global demands of lubricating oil is expected to raise by 2.0 % per year to 45-40 million metric tons with share of biolubricant market raising to 15-30 %. Hence the need to seek alternative form of energy that is renewable, clean, sustainable, chemical stable and environmental friendliness cannot be overemphasize. Currently, the discipline of green chemistry is gaining recognition with respect to biolubricant obtained from green plant (vegetable oil)(Chen *et al*., 2019). Biolubricant are lubricant that are obtained from vegetable oil (esters) via a process known as transesterification reaction. The vegetable are made up of triglycerol with a carbon chain ranging from C4-C26 a double bond and a hydroxyl functional group attached to it which determines the properties of the lubricant in terms viscosity, oxidative stability and pour point (Rios *et al*., 2019*;* André *et al*., 2015). The presence of long fatty acid and polar groups makes them suitable for use as biolubricant, though chemical modifications such as epoxidation, hydrolysis and transesterification reactions can turn them into suitable lubricant (Cerón *et al*., 2018). The triglycerol obtained from the vegetable oil reacts with an alcohol in the presence of a catalyst to produce the corresponding biolubricant. the vegetable oil extracted

from a plant cannot be directly use as lubricant because of its poor fluidity at room temperature and it also lack thermal and oxidative stability due to the presence of double bond (Talib and Rahim, 2016), hence there is need for structural modification in order to meet up with the physiochemical properties of a standard lubricant such as pour point, viscosity index and high flash points (Syahrullail *et al*., 2019). There are three (3) different ways of producing biolubricants: by esterification or transesterification reaction, estolide formation and epoxidation followed by ring opening and acetylation reaction. However, the first method i.e. esterification or trans-esterification reaction is more convenient and conventional the second method requires estolide formation that would require capping fatty acids which are expensive, while the third method requires multiple reaction stages making it unfavorable (Chen *et al*., 2019, Mcnutt and He, 2016). Biolubricant can be classified as either vegetable or animal oil depending on their source. This lubricant, when compared to synthetic lubricant offers a better advantage that include; less toxicity and environmental friendliness, higher flash point, constant viscosity, less mist oil, vapor, are renewable and biogradable (Preferable and Fact, 2011). However, the problem associated with the use of this lubricant is that they have; high viscosity at low temperature, poor oxidative and thermal stability, and emission of unpleasant odor if contaminate are present.

Cassia sieberiana commonly known as drumstick, is a tree that belongs to the family fabaceae which is native to African, commonly known as malgaa in Hausa language. It has a height range of 10-20 m the plant has yellow flower and fruit with a dark brown color. It is widely known for its medicinal properties. The root bark has been reported to have antimalarial therapy with its leave having medicinal properties (Warra, 2016). Hence, the objective of this paper is the production and characterization of biolubricant from *Cassia sieberiana* (Decandole) seed oil via two step transesterification reaction between methylesters and trimethylolpropane (TMP).

MATERIALS AND METHODS

Raw material collection and reagent: The *Cassia sebieriana* seed were obtained from NARICT and the chemicals were source from outlet retailers

Oil extraction: The seed oil was extracted using n-hexane, 50 g of the powder seed sample was put into a porous timble and placed in a soxhlet extractor with 150 cm of n-hexane being at a temperature of 40-60 ˚C as extracting solvent for a period of about 6 hrs repeatedly until a desired quantity was obtained after which it was evaporated with a water bath at a temperature of of 70 ˚C to remove dissolved solvent (Warra, 2016).

Preliminary analysis: Preliminary analysis was conducted to characterize the oil and to check its potential a lubricant base. This include saponification value, free fatty acid, free fatty acid reduction (esterification reaction), kinematic viscosity, density, pour point and flash point

Percentage yield: After the extraction of the seed oil using n-hexane the percentage yield of the oil content was determined by completely evaporating the solvent (n-hexane) for 2 hrs and its volume was recorded and expressed as percentage of oil content giving by the equation bellow (Warra, 2016).

$$\text{Oil content} = \frac{weight\ of\ the\ oil}{weight\ of\ sample}\ x\ 100$$

Oil esterification: The fatty acid content was reduced in-order to prevent high saponification. The reduction of the FFA content was achieved by esterification of the oil using methanol and sulfuric acid as catalyst.50 g of the oil sample was weight and transferred into a two litre three neck round bottom flask. 20 % w/w methanol and 5 % w/w sulfuric acid were mixed together in a conical flask. The mixture of the methanol acid and the oil sample were heated in a water bath at a temperature of 60 ˚C. They were then mixed in the three neck round bottom flask with a mechanical stirrer while the other two neck of the flask was stoppered. The stirrer was set at 700 rpm, while the water bath maintain a constant temperature of 60 ˚C. Timing was started after 60 min, a picking pipette was used to remove the sample and titrated against 0.1N solution of KOH in order to determine the free acid content of the oil. This titration was repeated at 1 hr interval till the fourth hour(Bilal *et al*., *2013*)

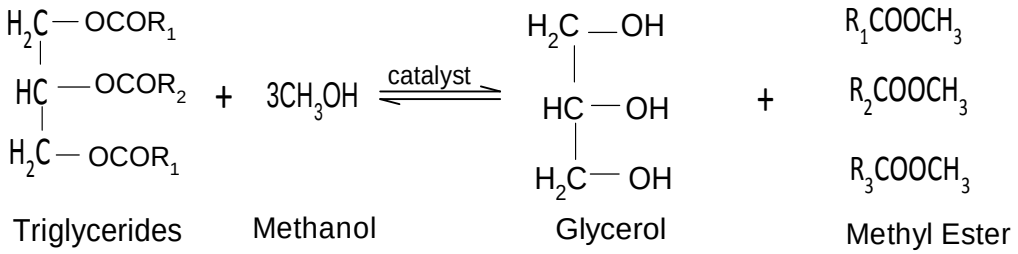
$$\%\ FFA = \frac{(ml\ of\ titrant)(\ N\ of\ titrant)x\ 28.2}{(sample\ weight)}\ x\ 100100$$

Where FFA = free fatty acid

Transesterification: The production of biolubricant can be achieved using two stage transesterification which is described below

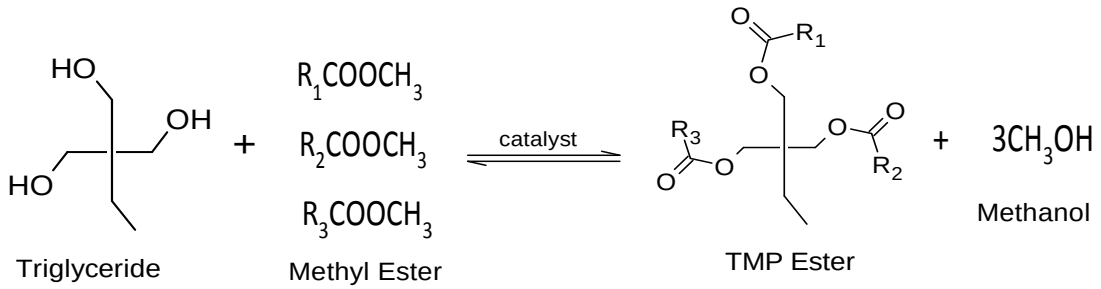
Methyl ester synthesis: This involves the transesterification of the oil sample using methanol and calcium oxide catalyst. 50g (100ml) of the oil was transesterified, the weight ratio of the oil to the methanol was 3:1. The amount of catalyst used was 0.5 % w/w of the oil. The reaction was achieved at a temperature of 60 ˚C for 2hrs (Bilal *et al*., *2013*).

$$\%\text{Methyl ester yield} = \frac{\text{Mass of methyl ester produced (g)}}{\text{Mass of oil sample used (g)}} \times 100$$



Scheme 1: Equation of Methylester synthesis

Poly ester (biolubricant) synthesis: The biolubricant was synthesize by the transesterification of methyl ester with trimethylolpropane (TMP) using calcium methoxide (in 30 % methanol) as catalyst the weight ratio of the sample oil to trimethylolpropane (TMP) was in the ratio 3.5:1 and the amount of catalyst used was 0.8 % w/w of the total reactant which was conducted at a temperature of 120 ˚C for 2:30 hrs (Bilal *et al.,* 2013).



Scheme 2: Equation of Polyester Synthesis

Characterization: The synthesize biolubricant was characterize for the following physico-chemical properties; Density, pour point, flash point, viscosity, iodine value, kinematic viscosity and saponification value.

Density: An empty beaker was weighed and recorded, 50 cm^3 of the seed oil was put into the beaker and weighed. The density of the seed oil was determine using the weight of the oil to know volume (50 cm^3) in SI unit according to the equation bellow (Bilal *et al*., *2013*).

$$\text{Density} = \frac{sample\ weight}{sample\ volume}$$

Pour point: Pour point is a function of low temperature properties, it measures the degree at which a fluid flow at colder operating temperature. I.e. the lowest observed temperature at which the fluid˚ will flow when subjected to. This is one of the main problem associated with biolubricant. (Ahmed., *et al 2014*) the pour point was determined using according to ASTMD97-12 by putting the sample in a test jar an allowed to cool in a cooling bath after which the temperature was measured at an interval of 3 ˚C until it stops to pour (Amdebrhan *et al*., 2015).

Flash point: This measures the lowest temperature at which a fuel vaporizes to form an ignitable mixture in the air. The flash point was conducted in accordance with the American natural standard method (ASTM D56-79) (Salimon *et al*., *2011*).

Viscosity: This was determined at a temperature of 40 ˚C and 100 ˚C by first of all choosing a viscometer spindle three (3) followed by transferring the sample into a large 250 ml beaker that can hold the viscometer spindle. The oil sample temperature was increased to the required value by constant stirring on a heating mantle and then checked by a thermometer. At the required temperature the sample was removed from the heat source in order to measure the viscosity. The spindle was connected to the upper coupling by gripping the coupling between the thumb and fore finger while carefully rotating the spindle counter clock wise. The spindle was immersed into the sample to the middle of the identification in the shaft. The viscometer was turned on and allowed to run until a constant reading was obtained. The viscosity of the sample was then taken in mpas (Bilal *et al*., *2013*).

Viscosity= reading obtained X $\frac{factor\ for\ the\ spindle}{speed\ comb}$

Iodine value: The iodine value was determined according to AOCS method of which 1 g of the sample was poured into 250 ml of conical flask followed by the addition of 10 cm^3 of CCl4 to dissolve the sample. The mixture was stirred and allowed to stand at a dark ambient temperature for about 30 minutes. 15 cm3 of 10 % KI solution was added with 100 cm3 of distilled water to rinse the flask and the solution was then titrated with sodium thiosulphate using starch as indicator. Blank sample was prepared and back titrated

$$IV = \frac{(sample - T - blank - T)xN\ x\ 12.69}{W_{OS}}$$

Where IV = Iodine value; N = normality; Wos = weight of oil sample; Blank-T = blank titre value; Sample-T = sample titre value

Kinematic viscosity: The kinematic viscosity of the lubricant oil was measured at 40 ^0C and 100 ^0C using a rotational viscometer with respect to ASTM D2983. The viscosity index was then calculated using ASTM D2270 equation, where U is the viscosity measured at 40 ^0C, L and H correspond to the viscosity at 100 ^0C of the oil extracted (Syahrullail *et al*., *2019*).

$$\text{VI}= \frac{L-U}{L-H} X\ 100$$

Saponification value: This was done according to the AOCS method cd 325. 2 g of *Cassia sieberiana* oil was poured in a 250 ml conical flask followed by the addition of 50 $cm^3$of 0.5 ethanolic KOH. The mixture was heated to saponify the oil. The unreacted KOH was back titrated with 0.5N hydrochloric acid using 2 drops of phenolphthalein indicator. The saponification value is the amount of KOH in milligram is required to saponify 1 g of the oil, it is a measurement of the molecular weight of the acid.(Bilal *et al*., *2013*).

$$\text{SAP value}= \frac{(titre\ value)x\ (normality of\ NaoH)X\ 56.1}{weight\ of\ sample}$$

Gas chromatography: The analysis of sample by chromatography involved separation of the sample according to molecular mass hence provide the necessary data of the sample composition. The column used by the GC was (GCMS-QP2010, Shimadzu) from Department of Chemistry Ahmadu Bello University, Zaria. The GC column was calibrated by injecting methyl ester standards in order to obtain good separation. The sample was diluted with little quantity of ethyl acetate and the carrier gas was hydrogen and its flow rate was monitored at 41.27 ml/min while the column flows at 1.82 ml/min. the temperature of the oven was calibrated at 80 ^0C prior to ramping up at 6 ^0C/min until 340 ^0C.

RESULTS AND DISCUSSION

The percentage of the free fatty acid obtained from the cassia seed oil was observe to be above 1 % wt as seen from table 1, therefore this value was reduced to 0.54 wt% respectively through esterification after a period of 120minutes, because it was discovered by (El-Diwani *et al*., 2010) that high value of FFA > 1 % poorly affect the transesterification hence, the reduction was carried out in order to avoid saponification during methylester synthesis. At the end of the esterification reaction after 3 hours, the FFA value was found to be closer to 0.44 % which was obtained by(Bilal *et al.,* 2013) which is in line with the specified ASTM standard limit of (<1 %)

Table 1. Free fatty acid (FFA) content of the oil

RUN	Time(min)	Cassia. Wt.%
1	**0**	**2.55**
2	**60**	**1.15**
3	120	0.54

Table 2: Physicochemical properties of the raw oil

Property	Experimental result
Density	0.91 (Kg/m^3)
Pour point	5 (˚C)
Saponification value	172.51 (MgKOH/g)
Iodine value	24.50 (gl/100g)
Acid value	5.18 (MgKOH/g)
Percentage yield	15.1 (%)
Free fatty acid	2.59 (%)

The oil yield of Cassia sebeiriana seed using n-Hexane as the extracting solvent was 15 % and with a density of 0.91 kg/m^3 which was found to be greater than 9 % yield obtained by (Warra, 2016) but closer to 13.97 % obtained by (Ezekoye and Ezekoye, 2016) but the density was however similar to 0.88 kg/m^3 which was obtained by him. This occurs as a result of difference in demographic factors and also species.

Table 3: The percentage composition of various fatty acid present in cassia seed oil

Fatty acid	Symbol	Area (wt. %)
Palmitic acid	C16:0	6.44
Stearic acid	C18:0	3.94
Oleic acid	C18:1	2.34
Linoelaidic acid	C18:2	59.68
Linoleic acid	C18:2	27.56

The GCMS result in table 3 indicates the various percentage composition of fatty acids present, it indicate that the oil contains 87.24 % of unsaturated fatty acid (linoelaidic acid and linoleic acid) than the saturated acid which is 12.72 % (palmitic and stearic acid) this account for higher iodine value of the oil (24.49 of iodine/100 g) of the sample, because the

unsaturated glyceride has the ability to absorbs a definite amount of iodine.

Table 4: Fatty acid composition of the cassia biolubricant oil from GCMS analysis.

Formular	Composition range (%)
Palmitic acid methyl ester	7.10
Cis vaccenic acid	9.01
Erucic acid	1.81
Methyloleate	26.40
Methystearate	1.07
Oleic acid	1.33
Paullinic acid	1.20
Methylester behenate	1.80
2-hydroxyl ethylester	14.15
22-tricosenoic acid	27.24
Others	6.83

The major fatty acid found in Cassia sieberiana was linoelaidic acid and linoleic acid which has significant Importance in surface coating industries and biolubricant base oil application (Hayat and Mendhulkar, 2016). The presence of high degree of unsaturation further confirm why most vegetable oil do not solidify at room temperature making it good oil for lubricants (Abitogun *et al.,* 2009). The oil fatty acid composition will affect it physical and chemical properties. The high content of unsaturated fatty acid can result in low oxidative stability similarly high amount of saturated fatty acid results in low pour point values (Attia *et al.,* 2020). The result of table 4 shows that the produced biolubricant consist primary of fatty acid methylesters; palmatic acid methylester, Euric acid, Methyloleate, methylstearate, cis vaccenic acid, Oleic acid, paullinic acid, methylesters behenate, 2-hydroxylethylesters and 22-tricosenoic acid with other traces of methyl esters. With a percentage area of 7.10 %, 1.81 %, 26.40 %, 1.07 %, 9.01 %, 1.33 %, 1.20 %, 1.80 %, 14.15 %, 27.24 % and 6.83 %. The major constituent of the Cassia sieberiana is the saturated content of palmatic acid methylesters ,2-hydroxy ethylester, methylstearate and 22-tricosenoic acid, which result in significant amount of saturation that help in high thermal-oxidative resistance (Heikal *et al.,* 2016).

Table 5. Physicochemical properties of the produced biolubricant via two step transesterification reaction.

Property	Cassia biolubricant	ISOVG-32	ASTM METHOD
Viscosity	44.20 at 40 (cst)	>28.8	ASTM D445
Viscosity	7.82 at 100 (cst)	>4.1	ASTM D445
Pour point	-9	<-10	ASTM D2270
Viscosity index	148.04	>90	ASTM D97
Yield	93 (%)	-	-
Density	0.92	0.70-0.95	ASTM D1298

Viscosity @ 40 and 100: The kinematic viscosity of cassia biolubricant produced via two step transesterification reaction with TMP were 44.20 cSt at 40 and 7.82 cSt at 100 respectively as seen in table 5, this value obtained were higher than 30 cSt at 40 and 6.5 cSt at 100 reported by(Ghafar *et al.,* 2019) the variation obtain in this work as relates to others may be due to the difference in fatty acid composition of the base oil or the process condition/difference in catalyst and additives. However this value cited seems to be relatively closer to 45.3 cSt at 40 and 9.21 cSt at 100 reported by (Mohammed *et al.,* 2015). The values obtain shows that the biolubricant produced met the ISOVG32 and ISOVG46 viscosity grade requirement as reported by (Bilal *et al.,* 2013).

Viscosity Index: The viscosity index is the property of a liquid that helps to reduce the effect of change in viscosity with increase or decrease in temperature. The viscosity index of the produced biolubricant is 148.04 cSt this value is however higher than 32 cSt and 78 cSt reported by (Dibal and Ibrahim, 2017) but lower than 311 cSt and 179.05 cSt obtained by (Attia *et al*., *2020*) and (Ghafar *et al*., *2019*). It is important to have high viscosity index because lubricant with high viscosity index resist changes in Temperature and also reduce wear and lubrication consumption during application (Mohammed *et al., 2015*). It was also observed that the viscosity index value obtained in this work is closer to 149+_2.11 cSt obtained by (Cerón *et al.,* 2018) but in accordance with the ISOVG 32 and 46 making it having a potential of a good lubricant.

Pour point: The pour point of a biolubricant is the property of that lubricant that determines its low temperature usability, the pour point of the cassia oil was reduced from 5 ˚C to -9 ˚C after the transesterification of the based oil with TMP as seen from table 5, This result shows that the pour point of the produced biolubricant are good enough to permit usage at low temperature. The pour point is however lower than those obtained by (Dibal and Ibrahim 2017) this may be due to difference in additives or the fatty acid composition of the oil. But similar to -9 ˚C and -8 ˚C obtained by (Alang *et al.,* 2018) and (Mohammed *et al., 2015*). It can be observing that the remarkable change in pour point as seen can be attributed to the attachment of an ester side chain to the position of the fatty acid chain of TMP to replace the hydroxyl group. hence high degree of branching of esters leads to higher pour point which are desirable properties of a lubricant (Salimon *et al.,* 2011) similarly the presence

of a large branch chain from the backbone of the TMP triggers a steric hindrance to slow the crystallization process (Samidin *et al.*, 2021).

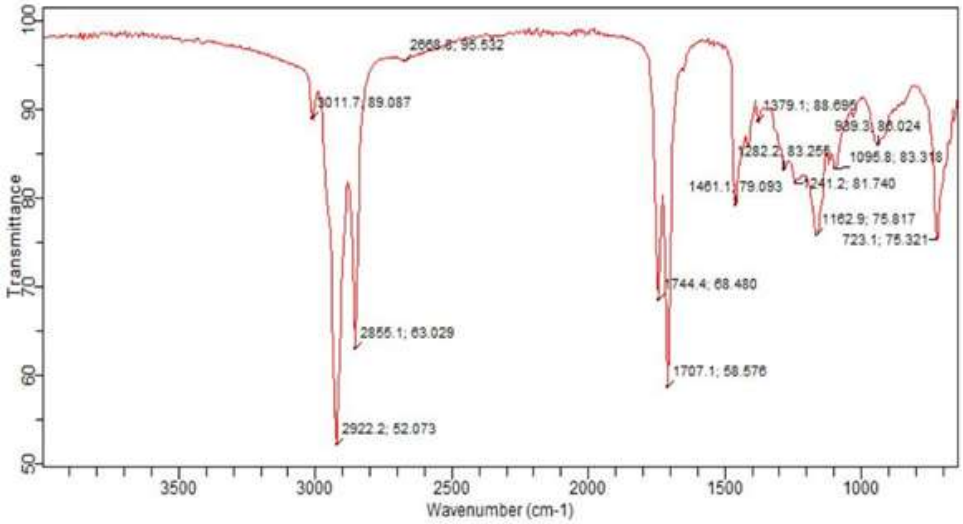


Fig 1. FTIR spectra of the cassia seed oil at different absorption bands

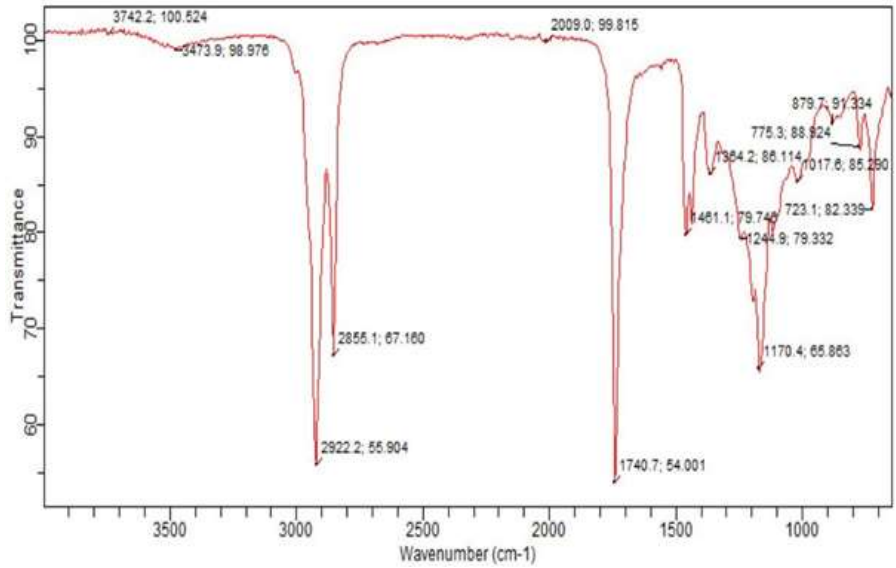


Fig 2. FTIR spectra of produced biolubricant from Cassia sieberiana seed oil

Vibrational frequency of organic compounds is widely used in quantitative and qualitative analysis to identify different absorption band of the functional groups. The FTIR spectrum of the cassia seed oil as seen in figure 4.5.1 shows an absorption band at 2922 and 2855 cm^{-1} which shows the stretching bands of C-H and – CH_2. From the spectrum a peak of relevance occurs at 1744.4 cm−1 which falls in the range of carbonyl (C=O) absorption and the peak located at 1707.1 is indicative of the presence of a C-O functional group.

The produced biolubricant was analyze to confirm the transesterification reaction between the methylesters and trimethylolpropane (TMP). The biolubricant shows an absorption band at 1748.7 cm^{-1} indicative of the presence of carbonyl (C=O) for esters as can be seen in figure 4.5.2 and the peak located at 1170.4 cm^{-1} correspond to C-O signifying the presence of oxygen in the biolubricant. There was a strong absorption band located at 2922 and 2853 cm^{-1} due to anti-symmetric and symmetric axial stretching vibration of C-H in CH_2 and CH_3 for the cassia base oil and biolubricant which was similar to the one

reported by (Dabai *et al.*, 2018) and(Heikal *et al.*, 2016) . The broad peak located at 3473.9 cm^{-1} in the biolubricant indicate the presence of oxygen-containing compounds and water molecules which appear as impurities in the TMP (Asadu and Okolo, 2021).

Conclusion: By bridging the gap between sustainable feedstock utilization, efficient production processes, and thorough characterization, this research contributes valuable knowledge to the field of biolubricant development. The findings have the potential to pave the way for the commercialization of environmentally friendly lubricants, promoting sustainability in the lubricant industry while harnessing the unique properties of Cassia Siberiana seed oil.

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