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PRODUCTION OF ECO-FRIENDLY BIOLUBRICANT FROM *Sesbania herbacea* SEED OIL USING POTASSIUM HYDROXIDE CATALYST

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Abstract: The increasing environmental concerns and depletion of fossil fuel reserves have necessitated the search for sustainable and eco-friendly alternative biolubricants. This study focus on the production and characterisation of biolubricant produced from Sesbania herbacea seed oil using homogeneous catalyst. The process of converting Sesbania herbacea seed oil into biolubricant is being studied in this work. Oil extraction by Soxhlet method yielded 20.0%, and the oil exhibited suitable properties for tranesterification, including low acid value (2.70 mgKOH/g) and free fatty content (1.35 mmol/L). Biolubricant production was achieved via double transesterification, involving initial biodiesel production followed by double transesterification with trimethyolpropane (TMP). The targeted Biolubricant was produced under reflux at various reaction conditions, and it was then characterized using FT-IR and GC-MS. The biolubricant's physicochemical properties were examined using techniques approved by the American Society for Testing and Materials (ASTM). The analysis of parameters like Kinematic viscosity (51.20), cloud point (2.90), pour point (-9.80), density (0.90) and viscosity index (180) of the Biolubricant revealed results were in line with the ASTM standards for lubricant.

Keywords: Sesbania herbacea seed oil, Methanol, Potassium hydroxide, TMP.

1.0 INTRODUCTION

The search for sustainable development due to increasing environmental challenges have increased the concern in the search of renewable, biodegradable and environmentally friendly products and processes development (Yang *et al.,* 2012). The increasing demands depleting fossil fuel energy reserves, cost of production and the overall damage to the environment fuelled the search for renewable sources of energy and other products which has resulted in significant discoveries. The bio-degradable fuel and lubricants made from vegetables and cereals based oils usually comprise of 60–99% base oil, and the remaining are additives which varies depending on the desired level of performance (Wagner *et al.,* 2001). The use of biodegradable and environmentally accepted lubricants from vegetable oil has increased over the past 25 years (Willing, 2001), however, dependence on consumable vegetables and cereals for biodegradable fuel and lubricants has far reaching implications on sustainable development.

Biolubricants 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). 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.

Transesterification and esterification are two main chemical processes for biolubricant production from lipid. In the transesterification process, triglycerides in plant or animal-based feedstocks react with an alcohol in the presence of a catalyst to produce fatty acid methyl ester (FAME). FAME is then esterified again with alcohols to form stable esters that can be used as biolubricants. Carbohydrates, particularly polysaccharides, can also be extracted and directly utilized as biolubricants due to their inherent lubricating properties.

2. Materials and Methods

2.1 Materials

Methanol (99.5%), potassium hydroxide (85.0%), Trimethylolpropane (98.0%), and sodium bicarbonate (95.0%) were all of the analytical grades and were purchased from Sigma-Aldrich (Merck).

2.2 Methods

2.2.1 Sample collection and Preparation

The Seeds of *Sesbania herbacea* were collected from Bakassi Postgraduate Hostel, Usmanu Danfodio University, Sokoto, Nigeria. The seeds were authenticated and identified by a Plant scientist (Botanist) in the Department of Plant Science, Usmanu Danfodiyo University Sokoto. The seeds were washed properly with water and shade dried in the open place for 7 days and the seeds were manually separated from hulls by cracking then milled to powder using electric powered milling machine which was be further placed on a solar dryer for a day to remove residual moisture.

2.2.2 Extraction of Oil from *Sesbania herbacea* Seeds

Oil from *Sesbania herbacea* seeds was extracted using Soxhlet extractor with n-hexane. The grounded seeds sample (50g) was placed in thimble plugged with a piece of cotton wool and inserted into Soxhlet extractor. A reflux condenser was fitted onto Soxhlet extraction column and the set up was placed on heating mantle, heated to a temperature of 60˚C to vaporize the solvent. The extraction lasted for six hours after which the flask was allowed to cool and the thimble was removed. The set up was reassembled to recover the solvent. The flask containing the extracted oil was dried in air oven at 65˚C for 30 minute to expel the remnant solvent (Muhammad *et al.,* 2021). The flask was then weighed and the percentage oil yield calculated using equation.

$$oil\ yield\% = \frac{Weight\ of\ extracted\ oil}{Weight\ of\ of\ sample} \times 100$$

2.2.3. Synthesis of methyl esters

Sesbania herbacea seed oil (400ml) was transesterified with methanol using Potassium hydroxide as catalyst. The weight ratio of oil to methanol was 3:1; (i.e. 48ml of methanol and 400ml of oil) the amount of catalyst was 0.5% w/w of the oil (i.e. 2g of the catalyst). The reaction was conducted at a temperature of 60°C for one hour to produce biodiesel and glycerol. The methyl esters (biodiesel) was allowed to be separated by gravity from the glycerin using separating funnel after leaving for about 20 hours (Muhammad *et al*., 2019).

2.2.4. Biolubricant Production

Sesbania herbacea methyl esters (20cm^3) was placed into 250cm^3 three neck round bottom flask and heated on a hot plate to 70°C for 10 minutes. 0.9g of Potassium hydroxide catalyst was dissolved in 5cm^3 of methanol. The catalyst mixture was heated for 10mins before being added to the reaction vessels. Then 1g of TMP crystals was added to the reaction vessels and reaction was allowed to proceed under reflux at 100°C for 4 hours and the reaction mixture was allowed to cool to room temperature. The mixture was then transferred into a separatory funnel for gravity separation after leaving for 20 hours. The same procedure was repeated at different reaction condition. (Alang *et al*., 2018; Mustapha *et al.,* (2023).

3.0 Results and Discussions

The results obtained from the study are presented in the Tables below. The analysis of *Sesbania herbacea* seed oil and the produced biolubricant reveals critical insights into their performance and potential as a sustainable lubricant base stock.

3.1. Physicochemical Parameters of the Extracted Oil (*Sesbania herbacea* seed oil)

The physicochemical properties provide valuable insight into the characteristics and potential applications of both the oil and the synthesized biolubricant. The physicochemical characteristics of the oil and the biolubricant produced, compared with the ASTM D5 standard specifications are presented in (Table 1), (Table 2) respectively.

Table 1 : Physicochemical Parameters of Sesbania herbacea Seed Oil

Properties	*Sesbania herbacea* oil
Percentage Yield (%)	20.0
Density (g/cm^3)	0.92±0.002
Viscosity (mPa.s)	66.6±1.601
Specific gravity -	0.923±0.003
Saponification Value (mg$_{KOH}$/g)	183.0±0.420

Iodine Value ($gI_2/100g$)	77.2±1.200
Acid Value (mg_{KOH}/g)	2.70±0.021
Free Fatty Acid (mmol/L)	1.35±0.030

Table 1 shows that, the oil yield of *Sesbania herbacea* seed oil was found to be 20.0% and was similar to the yield of *sesbania* seeds oil 20% reported by Mohammad *et al.* (2021) and higher than the range (19.10%) reported previously Salgin *et al.* 2016). In this study, the *Sesbania herbacea* seed oil yield was found to be similar to those reported for Soyabean, (18 -20%) reported by Liu (1997) and also found to be lower than Jatropha seed oil (25-40%) reported by Belewu *et al.* (2010). The oil high percentage makes this seed a distinct potential for the oil industry. The extracted *Sesbania herbacea* seed oil is liquid at room temperature, this makes it noble for biolubricant production. According to Sani *et al.* (2018) and Muhammad *et al.* (2018), variation in oil yield may be due to differences in variety of plant, soil type, duration of oil extraction, ripeness, harvesting time of seeds and the extraction method used.

Density is an important physical property of oils that determines their volume-to-weight ratio. The density of a substance is defined as its mass per unit volume. The density of the extracted oil is 0.90g/cm^3 which is similar to 0.92g/cm^3 reported by Belewu *et al.* (2010) and higher than the range 0.918gm/cm^{3}reported by Mohammad *et al.,* (2021). However, it's worth noting that the exact density of a given oil can vary depending on factors such as the source of the oil, the method of production, and the moisture content. Viscosity is a measure of resistance of a fluid to deform under shear stress. It is commonly perceived as thickness, or resistance to pouring. Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction (Usman *et al.*, 2017). The oil was measured at 40°C and found to be 62.60mPa.s which is slight higher than 55.12mPa.s reported for *Sesbania* seed oil by Mohammad *et al.* (2021).

Specific gravity is a measure of the density of an object compared to that of reference material and it is a dimensionless quantity. The higher the specific gravity of a fuel the greater the mass of fuel injected into the engine and hence more power and emission (Orhan *et al.,* 2010). The specific gravity of the oil is 0.923 which is slightly similar to 0.922 reported by Adebowale and Lawal, Saponification Value (SV) is defined as the number of milligrams of potassium hydroxide (KOH) required to saponify one gram of fat or oil. Saponification Value is used in checking adulteration. Oils with higher saponification values contain higher proportion of lower fatty acids (Muhammad *et al*., 2018). The saponification value of the oil is 183 mg_{KOH}/g which is slightly similar to the result 185.2 mg_{KOH}/g for *Sesbania* oil reported by Ahmad *et al.* (2019) and is lower than 187.4 mg_{KOH}/g obtained by Mohammed *et al.* (2021). This shows the oil is normal triglycerides and is useful in production of liquid soap in shampoo industries (Muhammad *et al.,* 2018).

Iodine Value is a measure of the degree of unsaturation of oil and fat, it is an identity characteristics nature of oil. The iodine value could be used to quantify the amount of double bond present in the oil which indicate the suitability of oil for production of biolubricant. The iodine value of the oil was found to be 78.21 gI_2 /100g; this value is higher than 76.80 gI_2 /100g reported by Ahmad *et al*. (2019) and is slightly similar with 78.4 gI_2/100g obtained by Mohammad *et al.* (2021).

Acid value is defined as the measure in milligram of potassium hydroxide per gram of fat/oil required to titrate a sample to a specified end point. It is a direct measure of FFA present in the oil. The acid value of the oil was found to be 2.7mg_{KOH}/g which is similar to 2.4mg_{KOH}/g reported by Kaur *et al.* (2012). Low acid value lowers the choice of oil for biodiesel production, production of paints, liquid soap and shampoo (Aremu *et al.,* 2006).

Free Fatty Acid Value (FFA Value) is the measure of the amount of free fatty acids present in a fat or oil. The amount of FFA in the oil was found to be 1.35mg_{KOH}/g which is slightly similar to 1.10mg_{KOH}/g reported by Ramalingam *et al.* (2018). Studies have shown that high FFA reduces catalyst effectiveness and decreases the production yield; therefore, the recommended amount of FFA in vegetable oil should not exceed 2wt%, (Ngoya *et al*., 2017).

Physicochemical Parameters of Biolubricant produced

The physicochemical properties provide valuable insight into the characteristics and potential applications of both the oil and the synthesized biolubricant. The physicochemical qualities are crucial criteria that provide valuable information. Kinematic viscosity, pour point, cloud point, viscosity index, and density are a few of these characteristics.

Parameters	Biolubricant Produced	ASTM Limits D5
Kinematic viscosity(mm^2/s)	51.20±0.020	> 41.1
Viscosity index -	180.0±1.001	> 90
Pour point (ºC)	-9.80±0.840	< -10
Cloud point (ºC)	2.90±0.160	-10 to 5
Density (g/cm^3)	0.90±0.001	0.86- 0.93

Table 2: Physicochemical Parameters of Biolubricant in Comparison with ASTM Standard

Kinematic Viscosity

Kinematic viscosity is the most important property of the oil because it affects the fluidlity, lubricity and atomization of fuels (Sattanathan, 2013). Viscosity is very important in determining the fluidity of lubricating oil at low and high temperatures (Bilal *et al*., 2013). Fuel with low viscosity may not provide sufficient lubrication resulting in wear and high viscosity causes poor combustion and also increases exhaust emission (Rengasamy *et al.,* 2014). The kinematic viscosity of the biolubricant produced was found to be 51.20 mm^2/s which is similar result was obtained by Salih *et al.* (2011). The kinematic viscosity result obtained is highly favourable for ignition, as the higher viscosity enhances the oil's characteristics and enables more efficient engine performance.

Viscosity Index

Viscosity index shows the properties of the lubricant viscosities when temperature changes are applied. The viscosity index obtained from the biolubricant was found to be 180 which is similar to the result obtained by Rajendra *et al.* (2022). The obtained result was also in accordance with the ASTM standard.

Pour point

Pour point refers to the lowest temperature at which fluid continue to flow and maintain it is lubricity properties or is the property of bio-lubricant that determines its low temperature usability. The pour point was carried out in this research to determine the freezing point of the sample. The pour point obtained from the biolubricant was found to be -9.8 which is similar with the result obtained by Savwede and Tarbuka, (2024) and the

ASTM allowed limit of -9.8 falls within the range of ASTM standards for pour point for lubricants.

Cloud point

The cloud point is the temperature at which dissolved solids are no longer completely soluble; precipitating as a second phase giving the fluid a cloudy appearance (Rengasamy *et al.* (2014). The ASTM standard for maximum allowed values of cloud point (<-10ºC) are within the range of -15 to 5 ºC. The cloud point obtained from the biolubricant was found to be 2.9 ^{0}C similar to the result (3.0ºC) obtained by Zayyanu *et al*. (2024). The result obtained in this work fall within the ASTM standards for lubricants oil.

Density

The density, the higher the density of a lubricant, the thicker it becomes; which increases the amount of time it takes for particles to settle out of suspension (Areej *et al.,* 2019). The ASTM specification allowed for biolubricant are within the range of 0.86gm/cm^3 to 0.93g/cm^3. The density of the obtained biolubricant was found to be 0.90gm/cm^3 similar to the result obtained by Hadiza *et al.* (2024). The result of density obtain in this work was also in complies with the ASTM Standard.

Characterization of the Biodiesel and Biolubricant produced

Table 3: Characterization of the Biodiesel and Biolubricant produced

Absorption band of the Biodiesel (cm^{-1})	Bond	Absorption band of the Biolubricant (cm-^{1})	Bond
3411	O-H	3384	O-H
1738	C=O	2921	C-H
1237	C-O	1743	C=O
2926	C-H	1378	CH3
721	C-H	1238	C-O

Agilent Technologies

Sample ID:Hussein bio biesel

Method Name:C:\Users\Public\Documents\Agilent\MicroLab\Methods\Aminu Method.a2m

Sample Scans:32

User:Central Lab

Background Scans:4

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Range:4000 - 650

System Status:Good

Apodization:Happ-Genzel

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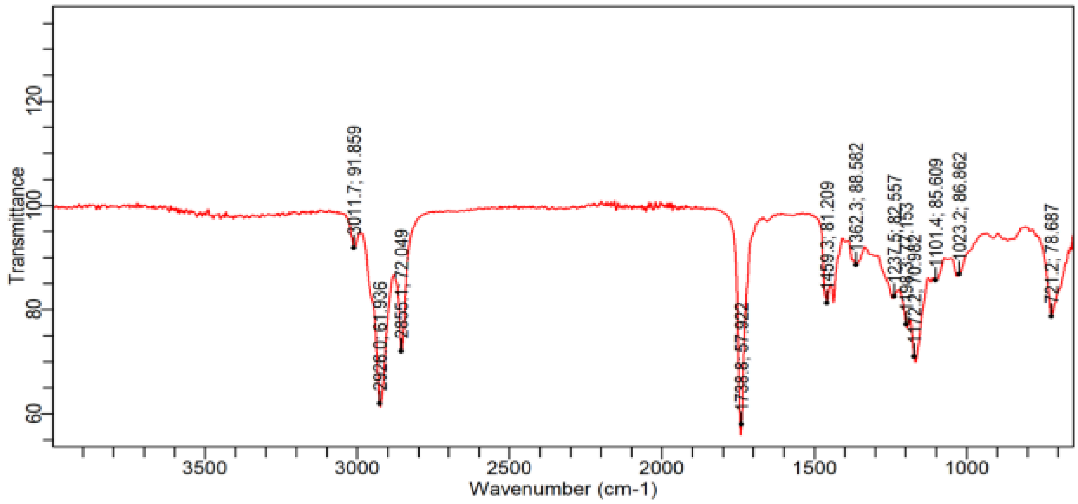

Figure 1: showing the FTIR spectrum of biodiesel

The FT-IR analysis of the obtained biodiesel (Appendix i) revealed a major absorption band at 2926cm-[1], corresponding to the C-H stretching vibration of alkanes. A strong absorption also around 1738cm-[1] represents the characteristic C=O stretching of an ester group. Additionally, an absorption with 1237cm-[1] was identified as characteristic of C-O stretching band for alcohols and a distinct absorption at 721cm-[1] attributed to C-H aromatic bending vibrations. The absorption bands obtained in this analysis are consistent with those observed by Banik *et al.* (2018). The spectrum obtained from this analysis revealed major peaks corresponding to C=O absorption of carbonyl groups, C-H stretching of alkyl groups and C-O absorption bands characteristic of ester. This suggests that the analyzed spectrum represents an aromatic ester compound.

Agilent Technologies

Sample ID:Hussain Biolubricant

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User:Central Lab

Background Scans:4

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Range:4000 - 650

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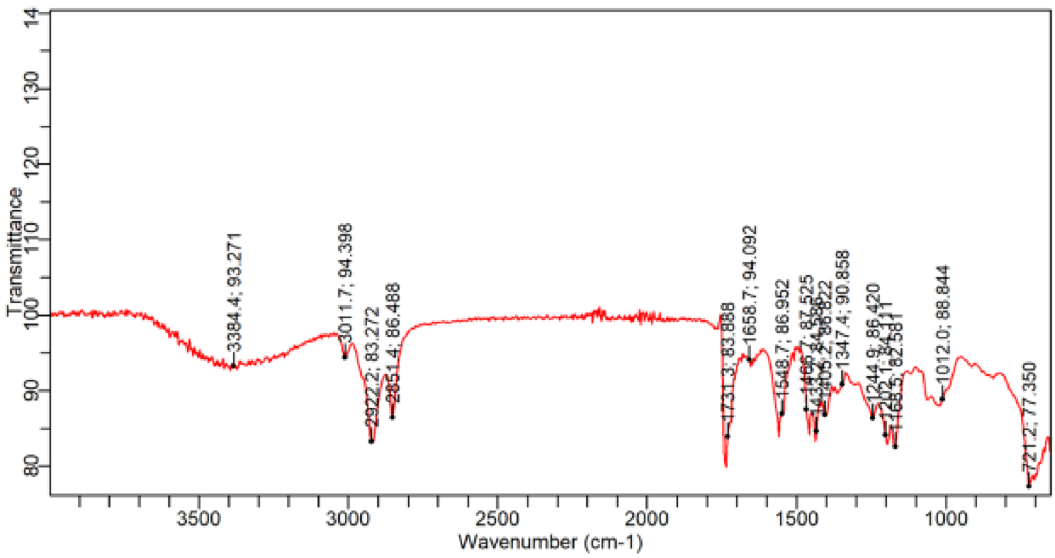

Figure 2: showing the FTIR spectrum of biolubricant

The FT-IR analysis of the biolubricant obtained in table 3 shows the frequency absorption band at about 3384cm-[1] which indicates the O-H bond for alcohols. There is also an absorption at 2921cm-[1] which illustrates the presence of C-H bonds. A stretching absorption C=O seen at 1743cm-[1] designates a characteristic absorption of the carbonyl group and a sharp stretching absorption band at 1238cm-[1] which illustrate C-O bond. However, the spectrum obtained from the analysis of the produced biolubricant revealed major peaks corresponding to O-H stretching (alcohols), C=O absorption (carbonyls) and C-O absorption band (esters) The absorption bands obtained in this analysis are similar with those reported by Mobarak *et al.* (2022). This indicates that the analyzed spectrum represent an alcohol compound attached to an ester group. Furthermore, the absorption bands and their corresponding functional groups are presented in Table 3.

GC-MS analysis of the biodiesel and biolubricant Product

The products (biodiesel and biolubricant) were further analyzed using GC-MS analysis and the results (Table 4) shows the distribution of the obtained esters from esterification of methyl esters with TMP using potassium hydroxide as catalyst. Moreover, the GC-MS analysis of the products revealed that most of the organic compounds detected were by-products of the reaction.

Biodiesel	Molecular formula	Biolubricant	Molecular formula
Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	Linolenic acid, methyl ester	$C_{19}H_{32}O_2$
9,12-Octadecadienoic acid, methyl ester	$C_{19}H_{34}O_2$	Octadecanoic acid, methyl ester	$C_{19}H_{38}O_2$
Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$
Pentadecanoic acid, 14-methyl-methyl ester	$C_{17}H_{34}O_2$	Hexadecanoic acid, ethyl ester	$C_{18}H_{36}O_2$
Methyl stearate	$C_{19}H_{34}O_2$	(Z), 9-Octadecenoic acid, ethyl ester	$C_{20}H_{38}O_2$

Table 4: Names of Compounds Present in Biodiesel and Biolubricant produced using GC-MS.

4.0 CONCLUSION

This study demonstrated that an environmentally sustainable biolubricant can be derived from the oil of *Sesbania herbacea* seeds through the esterification of methyl esters using trimethylopropane (TMP) in the presence of homogenous catalyst (Potassium hydroxide). By employing a Soxhlet extractor with n-hexane as the solvent, 20.0% of the oil was effectively obtained from the *Sesbania herbacea* seeds. The physicochemical properties of the extracted oil and the biolubricant produced showed that all the parameters analyzed are in agreement with the ASTM Standard. FT-IR analysis confirmed ester formation through characteristic C=O and C-O absorption bands, while GC-MS revealed the presence of long-chain fatty acid esters.

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