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Production of biolubricant from *Jatropha curcas* seed oil

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Extracted *Jatropha oil* was analyzed for their chemical and physical properties such as density, acid value, % FFA, saponification value as well as viscosities at 40 and 100°C and viscosity index. The result of the analyses of the Jatropha oil reveals that it has a very high %FFA (14.6%). Reduction was achieved by esterification with methanol (to 0.44%). The method employed for the production of biolubricant involved two stages transesterification process, the first stage produced methyl ester of the oil and in the second stage; the methyl ester was transesterified with ethylene glycol to produce the biolubricant. Major lubricating properties of the product such as pour point, viscosities at 40°C and at 100°C, and the viscosity index were analyzed and found to have value of -7°C, 55.22 cSt, 10.96 cSt and 195.22 respectively. It was found that the biolubricant produced is comparable to the ISO VG-46 commercial standards for light and industrial gears applications and other plant based biolubricant.

Key words: *Jatropha oil,* esterification, transesterification, biolubricant.

INTRODUCTION

The depletion of the world's crude oil reserve couple with the consumption rate, increase in petroleum prices and scarcities, and issues related to conservation have brought about renewed interest in the use of bio-based materials. Emphasis on the development of renewable, biodegradable, and environmentally friendly industrial fluids, such as diesel, lubricants and other fuels have raised the need to search for alternative renewable fuels (Sahoo et al., 2007; Basha et al., 2009; Demirbas, 2009; Refaat, 2010; Yang et al., 2012). It has been estimated that the global transportation energy use is expected to increase by an average of 1.8% per year from 2005 to 2025. Nearly all fossil fuel energy consumption in the transportation sector is from crude oil (97.6%), however, the expected depletion of fossil fuels and environmental problems associated with burning them have encouraged many researchers to investigate the possibility of using alternative fuels, in which biodiesel is considered to be a very promising resource (Shah et al., 2013).

The International Energy Agency (IEA) report (2007), and Shahid and Jamal (2011) have also indicated that the world will need 50% more energy in 2030 than now, of which 45% will be accounted for by China and India. Biolubricant production is necessary to serve as a substitute lubricant to supplement or replace conventional lubricants due to its numerous advantages such as renewability, biodegradability and lower gaseous emission profile.

A lubricant is a substance (often a liquid) introduced between two moving surfaces to reduce the friction

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between them, improving efficiency and reducing wear (Jumat et al., 2010). Wear and heat cannot be completely eliminated but can be reduced to negligible or acceptable levels. Lubrication reduces friction between moving surfaces by substituting fluid friction for mechanical friction (Marth, 2007). Other functions include heat transfer, liquid sealing, contaminant suspension, and corrosion protection. In this study, the production of biolubricant using Jatropha oil as raw material is the main task.

Lubricants have been classified into two major categories based on their sources; the mineral oil lubricant and the biolubricant. The mineral oil lubricants are obtained from crude oil sources and are believed to be harmful to the environment and by extension to human life. For this and other reasons, lubricants were synthesized from plant oils and other environmentally friendly sources which are referred to as biolubricants and these are primarily triglyceride esters derived from plants and animals. There is an increasing demand for environmentally compatible lubricants, particularly in areas where they can come into contact with water, food or people (Askew, 2004). *Lubricants are generally composed of a majority of base oil plus a variety of additives to impart desirable* characteristics. Although generally lubricants are based on one type of base oil, mixtures of the base oils also are used to meet performance requirements.

Jatropha is a non edible plant that was recently discovered to have great potential as feedstock for biodiesel and also biolubricant production (Banerji et al., 1985). Plant oil based lubricants can be attractive to global consumers because of its environmental benefits and the fact that it is made from renewable resources. Jatropha oil is considered non-edible oil due to the presence of toxic esters (Shah et al., 2004). Thus, it could provide an alternative of sufficient supplies of low cost feedstock for fuel oil and its derivatives with no competition with food uses (Ghazi et al., 2010).

Jatropha curcas grows in tropical and subtropical climates across the developing world, and is often cultivated as a hedge crop. It grows rapidly and requires minimal water and nutrients, and is able to grow on barren land under harsh conditions and poor quality or degraded land (Kandpal and Madan, 1995).

The quest for renewable energy sources has since dominated most manufacturing industries with much emphasis on bio products. Several researchers have agreed on the possibility of obtaining more efficient lubricants from such (bio products) sources. Hence, there is need to investigate the possibility of obtaining an environmentally friendly and economically viable lubricant from one of such sources (Nigerian Jatropha curcas seed oil).

This study was carried out with the objective of investigating the feasibility of producing biolubricant from Jatropha oil by conducting chemical modifications on the Jatropha crude oil. The modification involved improving some of the lubricating properties of the Jatropha crude oil. The physicochemical properties of Jatropha biolubricant were also compared with a certain standard properties of lubricants.

Advantages of biolubricant

A number of advantages have been exhibited by biolubricants which give them an edge over the conventional lubricants. These advantages have been highlighted by a number of researchers (Bob and Dwight, 2010; Rahul and Karmarkar, 2013). Biolubricants have excellent lubricity of about 2 to 4 times their petroleum based corresponding lubricants. This is enhanced with the polar nature of the lubricants and also enhances the affinity towards the metal surface resulting in substantially increased thin film strength as indicated in Figure 1. This wetting tendency helps in reduction in friction and energy saving in the range from 5 to 15% of the equipment operation. Biolubricants have higher viscosity index: The viscosity does not vary with temperature as much as the petroleum based lubricants. This makes bio-lubricants suitable for high temperature applications, typically 250°C and above. They also produce fewer emissions due to higher boiling temperature ranges of esters. This can be an advantage when designing lubricants for use over a wide temperature range (Askew, 2004). Biolubricants possess lower volatility, higher flash/ fire points, less vapour emissions and oil mist, and constant viscosity that make them offer better safety. Biolubricants exhibit better skin compatibility and less dermatological problems. Biolubricants also have biodegradability and non-water polluting characteristic which reduces the costs of disposal in addition to making them ecofriendly. Biolubricants are also cost saving on account of less maintenance, due to longer intervals between re-lubrication.

Biolubricant excellent lubricity, minimizing of corrosion of metal surfaces, non-toxicity, and biodegradability, coupled with energy saving makes them suitable to be used in all fields of industry. The increased use of bio-based products will also be expected to reduce petroleum consumption, increase the use of renewable resources, better manage the carbon cycle, and may contribute to reducing adverse environmental and health impacts.

Disadvantages of biolubricant

A study has equally been carried out to indicate the demerits of biolubricants (Environmentally Preferable

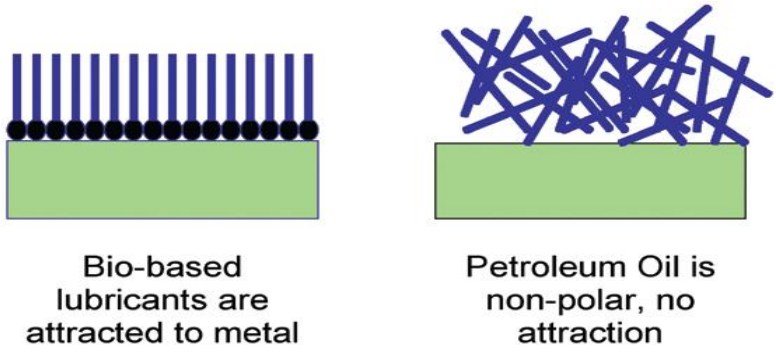

Figure 1. Bio-based lubricants possess a polar attraction to metal, while petroleum-based fluids have no polarity and therefore no affinity to metal (Bob and Dwight, 2010).

Purchasing Fact Sheet, 2011); biolubricants have several disadvantages in the use phase of the product life cycle, including: some bad odours if contaminants are present, high viscosity at low temperatures, poor oxidative stability at high temperatures, although additives designed specifically for plant-based lubricants eliminate stability issues related to extreme high and low temperatures. If biolubricant is untreated, it lacks oxidation stability and will have high pour points (Lou, 2008). There are some challenges associated with the production of biolubricant which include the high cost and limited availability of biolubricant feedstock. There are various factors contributing to the cost of biolubricant. These factors include feedstock prices, plant's capacity, feedstock quality, processing technology, net energy balance nature of purification and its storage, etc (Haas, 2005). However, the two main factors are the costs of feedstock and the cost of processing into biolubricant. It has been found that the cost of feedstock accounts for 75% of the total cost of biolubricants and biofuels in general (Miao and Wu, 2006).

MATERIALS AND METHODS

The materials and reagents used in carrying out the research are as follows: Crude Jatropha oil, methanol, potassium hydroxide, sodium hydroxide, hydrochloric acid, sulphuric acid, isopropyl alcohol, sodium methoxide (30% in methanol) and phenolphthalein indicator.

The instruments and equipment used in carrying out the study are: Mechanical press machine, Soxhlets apparatus, Heating mantle, Brookfield Viscometer (Manufacturer: Brookfield, Model:RVT-43329), water bath, refrigerator, analytical balance (Manufacturer: Adam, Model: PW 124) capable of determining weights to four decimal places, conical flasks, Graduated cylinders, 25 and 50 mL. Beakers, three neck round bottom flasks, magnetic stirrer, retort stand and clamps, Liebig condensers, with ground glass joints, Thermometer capable of measuring both negative and positive temperatures, pipettes and burette, mechanical stirrer and test tubes.

On the basis of methodology, the research is subdivided into three categories; extraction of the oil from the oil bearing seed, characterization of the crude oil and finally, chemical modifications were conducted to produce synthetic esters (biolubricant) of the oil. The following steps were followed as methodology.

Oil extraction

The oil was extracted via solvent extraction method. The raw material (Jatropha seed) was prepared to ensure maximum extraction of the oil.

Raw material preparation

For high oil content materials (oil content of 15% or more), the following preparation steps were employed to make the material suitable for solvent penetration into the oil cells as well as for best percolation.

(i) Passing the seeds through corrugated roller mills with 3 mm flutes to reduce the size to about 3 mm.
(ii) Heating the broken material to about 80°C with open steam in and humidifying the material to raise the moisture content to about 11 to 12%.
(iii) Flaking of the humidified material between a pair of plain rolls to 0.25 mm thickness or lower.
(iv) Conveying the flakes to the extraction system after crisping them firm.

Process of extraction

A standard weight of crushed Jatropha seed was placed in a 5 L three neck flask. Hexane was used as solvent to extract oil. The volume of hexane needed was determined by the ratio of 6:1. A reflux condenser was fitted and the mixture was heated at 60°C and stirred for about 8 h. The resulting oil and solvent mixture were filtered to remove the suspended solids. Then, the mixture was placed in a rotary evaporator to evaporate the solvent and thus,

Jatropha oil was obtained.

Characterization of Jatropha crude oil

Density

An empty beaker was weighed and the weight recorded, then 50 cm^3 of the sample (Jatropha oil) was poured into the beaker and weighed. From the sample weight obtained, the density was determined by taking the ratio of the weight of the oil to the known volume (50 cm^3) in SI units according to the equation below:

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

Viscosity

The viscosity of the oil sample was determined at temperatures of 40 and 100°C, in the following way; first, spindle 3 was selected, the sample was then transferred to a 250 ml beaker. The temperature of the oil sample was raised to the desired value by heating on a heating mantle with constant stirring. The spindle is attached to the upper coupling by holding the coupling between the thumb and forefinger while cautiously rotating the spindle counterclockwise. The spindle was immersed into the sample up to the middle of the indentation in the shaft. The viscometer was then turned on and allowed to run until a constant reading is attained; this reading was taken as the viscosity of the sample in mPas.

Saponification value

This is the milligram of KOH required to saponify 1 g of fat or oil. Saponification value is the measure of the molecular weight of the fatty acid. The AOCS method Cd 3-25 was employed. 2 g of the sample (Jatropha oil) was weighed and transferred into a conical flask and 50 cm^3 of 0.5 N ethanolic KOH (that has stayed overnight) was added to the sample. The mixture was then heated to saponify the oil. The unreacted KOH was then back titrated with 0.5 N hydrochloric acid using 2 to 3 drops of phenolphthalein indicator (Mohammed-Dabo et al., 2012). The SAP value of the samples analyzed was calculated thus:

$$\text{SAP value} = \frac{(\text{Titre value})(\text{Normality of NaOH})(56.1)}{(\text{Weight of sample})}$$

Pour point

The oil Sample was poured into a medium sized test tube and (the test tube with its content) placed in a test tube holder. The set up was placed in a refrigerator and allowed to solidify. After it solidifies, the test tube was removed and a thermometer was used to read the temperature at which the solidified sample begins to melt and flow. This temperature was noted and recorded as the pour point of the oil sample.

Percentage free fatty acid (%FFA)

This is the percentage by weight of specified fatty acid in the oil. The method applied for this analysis, is the American Oil Chemists' Society (AOCS) method 5a-40.1 g of the sample was weight and transferred into a conical flask, 25 mL of isopropyl alcohol and 3 drops of (phenolphthalein) indicator solution were added. The mixture was titrated against 0.1N sodium hydroxide solution shaken constantly until a pink colour persisted for 30 s. The acid value was calculated according to the equation below: (Mohammed-Dabo et al., 2012).

$$\%\ \text{FFA} = \frac{(\text{mL of titrant})(\text{Normality of NaOH}) * 28.2}{(\text{sample wt.})} \times 100$$

Acid value

This is the number of milligram of KOH required to neutralize the free fatty acid in 1 g of the sample. 1 g of the sample (Jatropha oil) was weighed and transferred into a conical flask. The weight was recorded. 25 ml of isopropyl alcohol and 3 drops of the (phenolphthalein) indicator solution were added. It was then titrated with 0.1 N potassium hydroxide solution with constant stirring until a faint, pink end point appears and persisted for 30 s. The volume of titrant used to reach this endpoint was recorded and from the readings obtained, the acid value is evaluated using the equation below (Mohammed-Dabo et al., 2012):

$$\text{Acid Value} = \frac{(\text{Titre value})\ (\text{Normality of KOHt})(56.1)}{(\text{Sample weight})} \times 100$$

Oil esterification

This is required to reduce the free fatty acid content of the oil as it may lead to high saponification. The high FFA content of the oil was reduced by esterification of the oil with methanol using sulphuric acid as catalyst. 100 g of the oil was weight and transferred into a two liter three necks round bottom flask. 20% w/w methanol and 5% w/w sulphuric acid were weighted and mixed in a conical flask. Both the methanol acid mixture and the oil sample were placed in a water bath and heated to a temperature of 60°C. They were then mixed in the three necks round bottom flask as shown in Figure 2; a mechanical stirrer was inserted through one of the necks while the other two necks were stoppered. The stirrer was set at 700 rpm and the temperature of the bath maintained at 60°C. At this moment, timing was started. After 60 min, a picking pipette was used to withdraw the sample and it was titrated against 0.1 N solution of KOH to determine the free fatty acid content of the oil. The titration was repeated at one hour intervals up to the fifth hour.

Transesterification of the oil

Transesterification is the reaction of triglycerides to fatty acid alkyl esters (FAAE) and low molecular weight alcohols such as methanol and ethanol in the presence of catalyst (Demirbas, 2011; Sharma and Singh, 2009; Demirbas et al., 2009) Production (synthesis) of biolubricant involves a double transesterification; the first one is aimed at producing and intermediate product- methyl ester of the oil, and the second uses the methyl ester as reactant to produce the desired product a polyol ester. The two processes proceed as follows:

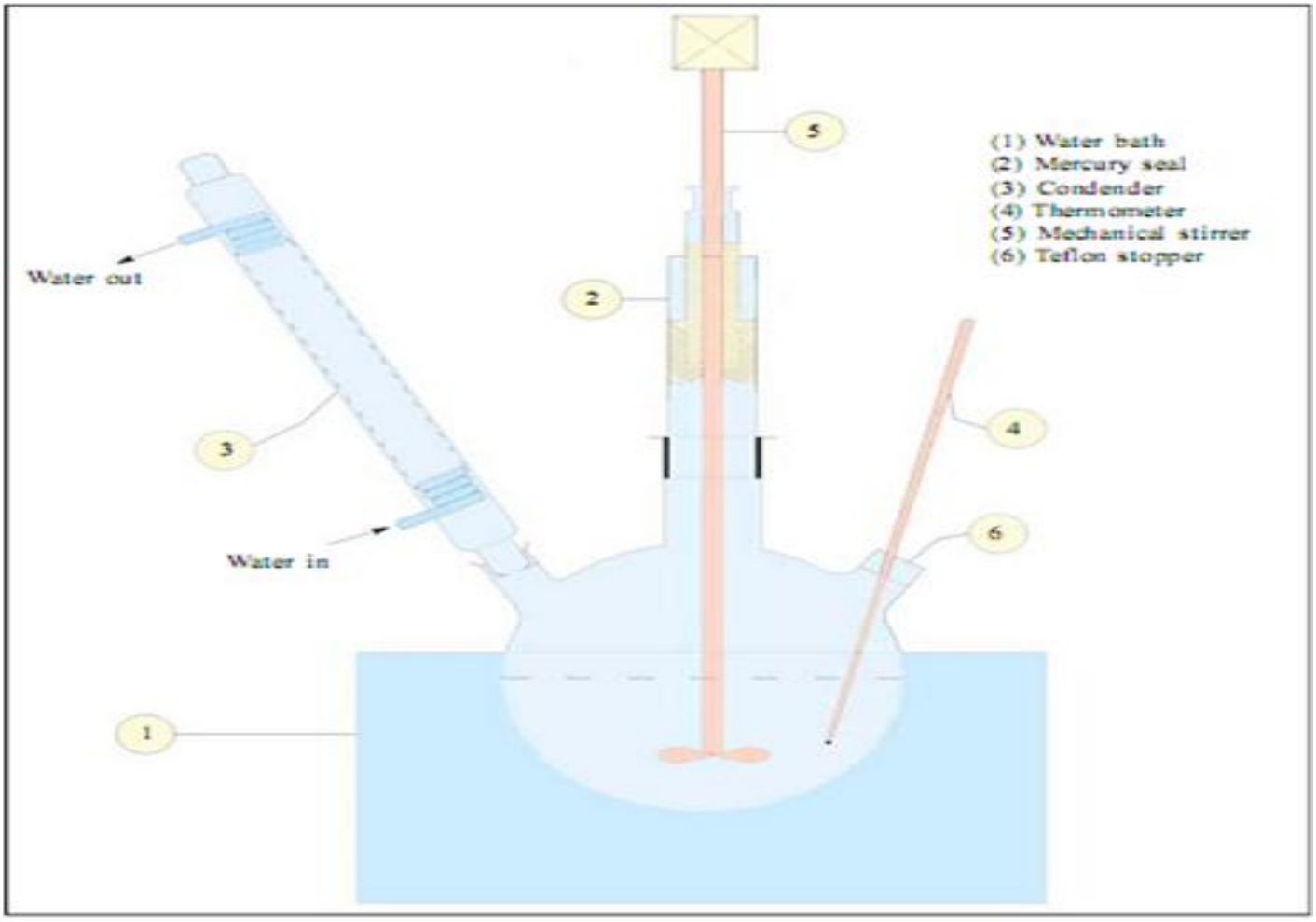

Figure 2. Esterification/Transesterification set-up.

Methyl ester synthesis

This was achieved by transesterification of the oil sample with methanol using potassium hydroxide as catalyst in the following way; 89.36 g (100 ml) of the oil was transesterified with methanol. The weight ratio of oil-to-methanol used was 3:1, the amount of catalyst used was 0.5% w/w of the oil and the reaction was conducted at a temperature of 60°C for two hours (2 h), (Ghazi et al., 2010).

Biolubricant synthesis

This is achieved by transesterification of the methyl ester with ethylene glycol in 50 ml batches using 0.5 M Sodium methoxide (prepared simply by dissolving fresh clean sodium in 30% methanol) as catalyst. The weight ratio of oil-to-methanol used was 3.5:1, the amount of catalyst used was 0.8% w/w of the total reactants and the reaction was conducted at a temperature of 120°C for two hours thirty minutes (2.5 h).

RESULTS AND DISCUSSION

The results obtained from experimental analysis of both the Jatropha (crude) oil sample and synthesized lubricants are presented in Tables 1, 2 and 3. These are explained under major three sub headings namely: characterization of Jatropha crude oil, FFA reduction by esterification and analysis of Jatropha biolubricant presented below.

Characterization of jatropha crude oil

The properties of the crude oil which are pertinent to lubricity are the viscosities at 40 and 100°C, viscosity index and the pour point. From Table 1 these properties have values of 66.74 cSt, 14.28 cSt, 220.70 and 5°C respectively. These properties make the sample (Jatropha

Table 1. Characteristics of Jatropha crude oil.

S/N	Property	Units	Values
1	Density	Kg/m^3	920.4
2	Acid value	Mg KOH/g	29.06
3	Free fatty acid(FFA)	%	14.6
4	Saponification value	-	198.76
5	Pour point	°C	5.0
6	Viscosity@40°C	cSt	66.74
7	Viscosity@100°C	cSt	14.28
8	Viscosity index (vi)	-	220.7

Table 2. The esterification result showing the decrease in FFA with time.

S/N	Time (h)	FFA (%)
1	0	14.6
2	1	12.87
3	2	7.79
4	3	4.99
5	4	2.76
6	5	0.44

Table 3. Properties of synthesized biolubricant alongside standard properties according to ISO VG-46 and those of petroleum based lubricant.

Property	Jatropha crude oil	Jatropha biolubricant	ISO VG-46	Petroleum based lubricant*
Density @ 25°C (Kg/m^3)	920.4	889.7	-	885.6
Viscosity@40°C (cSt)	66.74	55.17	>41.4	10.801
Viscosity@100°C (cSt)	14.28	10.96	>4.1	3.136
Viscosity index	220.7	195.22	>90	165.4
Pour point	5	-7	-10	-9

* Abdullahi (2012).

crude oil) a reliable feedstock for biolubricant synthesis. However, the FFA value (14.6%) is too high and if the oil is used for the production of lubricant, the product is likely to have high saponification value. High saponification value may lead to foam formation. Moreover, when this oil is used for transesterification to produce methyl ester, the yield will be very low as separation of the methyl ester from its byproduct- glycerol will be difficult. Muazu et al., (2013) indicated that according to several researchers; the oil or fat used in alkaline transesterification reactions should contain no more than 1% free fatty acids (FFAs), which is equivalent to 2 mg KOH/g triglyceride, (Freedman et al., 1984; Liu, 1994; Mitterlbach et al., 1994). If the FFA level exceeds this limit, saponification hinders separation of the ester from glycerine and reduces the yield and formation rate of fatty acid methyl ester (FAME) also known as biodiesel. However, it has been recommended that acidity below 1 mg KOH/g triglyceride (that is, a 0.5% FFA content) is suitable for biodiesel production (Canakci and Van Gerpen, 2001). Hence, there is need to reduce this value to below 1%, as indicated in Table 2, this was achieved by neutralizing the oil by esterification with methanol.

FFA Reduction by esterification

The reduction in FFA value with time (in 1 hour interval) is presented in Table 2. The FFA value obtained (14.6) was reduced by esterification with methanol. This is because El-Diwani et al. (2010) have discovered that high value of FFA > 1% poorly affect the transesterification

reaction in which the FFA in the oil reacts with the alkali catalyst to produce soap (saponification) hence, decreasing the catalyst amount needed for the transesterification reaction. The formation of emulsion (saponification) usually results in low biodiesel yield and difficulties in the downstream recovery and purification process of the biodiesel (Emin, 2008). The reduction was carried out in order to avoid saponification during methyl ester synthesis. At the end of the esterification reaction after 5 hours, the FFA value was found to be 0.44 % as can be seen from Table 2.

Analysis of produced Jatropha biolubricant

The product (Jatropha biolubricant) was subjected to certain property tests to ascertain its applicability as lubricating oil. These properties include the pour point, viscosities at 40 and 100^{o}C, and the viscosity index .The major lubricating properties of the biolubricant produced are presented in Table 3, alongside those of the Jatropha crude oil and standards for light gear application according to ISO VG-46 specification. It was also compared with the petroleum based lubricant as reported by Abdullahi (2012).

ISO Viscosity Grade (VG) 46 is one of many grades requirements based on the viscosity range of the lubricants (Ghazi et al., 2010) ISO VG46 is one of the three grades that represents over 80% of all lubricant consumed (Lauer, 1995).

The viscosities of the biolubricant at 40 and 100°C are very important lubricity properties; they are useful in determining the fluidity of the lubricant at low and high temperatures. They also show the thermal stability of the lubricant. The viscosities of the synthesized Jatropha biolubricant were found to be slightly lower than those of the Jatropha crude oil but could meet the requirement of the ISO VG 46 since its viscosities are within the standard ISO VG 46 range presented in Table 3.

The viscosity index obtained for Jatropha biolubricant is 195.22 and it is comparable to other plant based biolubricant. Viscosities index (VI) shows the characteristic of the lubricants viscosities when temperature changes are applied. The high viscosity index of the biolubricant is an indication that changes in viscosities at higher temperatures are going to be minimal. Viscosity index is also a very important lubricity property and the higher its value, the more preferable is the lubricant.

Pour point is the lowest temperature at which oil flows as its container is tilted for a prescribed period. It is crucial for oils that must flow at low temperatures. It is one of the most critical properties which determine the performance of lubricants. The pour point of the synthesized Jatropha biolubricant was significantly improved when compared to that of Jatropha crude oil. The pour point improved from originally 5 to -7°C. This value is also comparable to the pour point value of other plant based oil (Ghazi et al., 2010).

The density of the lubricant was also observed to decrease when compared with that of the crude oil sample. This may be attributed to the series of modifications it passed through; esterification and transesterification processes, thereby making the lubricant to be higher than the crude oil, hence improving its lubricity.

Conclusion

Synthetic esters namely Jatropha biolubricant was synthesized via transesterification method and the major lubricating properties of the synthesized biolubricant were analyzed. These properties when compared with standards as specified by the international standards organization (ISO) conform to those of viscosity grade 46 (VG-46). Hence, the synthesized biolubricant can favorably serve as substitute for petroleum based lubricants for light gear applications.

RECOMMENDATIONS

The following recommendations have been suggested to enable better understanding and improve the results obtained from such research:

(i) Additives especially pour point depressants should be incorporated in the synthesized biolubricant in order to improve the low temperature properties.
(ii) Standard equipment for testing the anti-wear and antifriction properties of lubricants such as High Frequency Reciprocating Rig (HFFR) should be employed for detail analysis of the lubricant properties.
(iii) Freshly extracted Jatropha *curcas* oil should be used for the production of biolubricant as the one used for this research has aged and could have possible effects on the properties of both the crude oil and the biolubricant.

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