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ORIGINAL RESEARCH ARTICLE

BIOLUBRICANT PRODUCTION FROM PARINARI POLYANDRA AND AZADIRACHTA INDICA SEED OILS USING TRIMETHYLOLPROPANE POLYOL

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ARTICLE INFORMATION

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ABSTRACT

Environmental pollution over time has raised challenges that elicit concerns for the nearest future. Synthetic lubricants, especially those obtained from petroleum products, contribute to environmental pollution. Thus, there is a need for the production of environmentally friendly lubricant as an alternative to petroleum-based lubricant currently being used. This work explored the potential of Parinari and Neem oils for the production of biolubricant using double transesterification method. Parinari oil was extracted from parinari seed using Sohxlet extraction method, and neem oil was purchased from Akande market in Ogbomoso, Oyo state, Nigeria. Both oils were pre-analysed for physico-chemical properties based on American Society for Testing and Materials (ASTM) methods. Trimethylolpropane (TMP) was used to increase the lubricating property of the transesterified parinari seed oil. The results showed that, the kinematic viscosity for Parinari based lubricant produced ranged between 12.1 – 67.4 cP at 40 ℃ and 15.5-28.6 cP at 100 ℃, while the kinematic viscosity for Neem-based lubricant produced ranged between 55.7 – 647.3 cP at 40 ℃ and 122.9 – 171.6 cP at 100 ℃. Parinari based lubricant had a viscosity index ranging from 159.7-174.4 and neem-based lubricant ranged from 326-365. Viscosities were all compared against SAE standard. SAE 30, 40 and 50 had kinematic viscosities ranging between 9.3-12.5, 12.5-16.3 and 16.3-21.9 respectively. Furthermore, all the biolubricants showed relatively high viscosity indexes with potentials for application in heavy equipment. NBL showed higher kinematic viscosity with longer transesterification reaction time. NBL gave random outputs for each run for biolubricants produced. Therefore, Parinari and Neem oil-based lubricants are potential lubricants as an alternative to petroleum-based lubricant.

1.0 Introduction

Fossil-based lubricant is commonly available in Nigeria. Nigeria depends heavily on fossil fuels, it is a country with a large oil reserve (Owuna, 2020). Petroleum based lubricants have constituted environmental pollution, affecting land and aquatic life within the ecosystem (Zhang *et al*., 2020). Addressing this challenge of environmental pollution has led to the search for suitable replacement of petroleum based lubricant (Srinivas *et al.*, 2020). Environmental pollution has been on an increasing trend, where man's activities over time has caused negative impact to the environment, leading to global warming (Tippayawong *et al.,* 2020). The united states of America is one of the leading nations in the production and use of alternative oils, hence, Nigeria is also in the search for suitable bio-based lubricants (Owuna, 2020). Lubricating oils like biolubricants comprise of oil and other chemicals which improve lubricant properties (Bhan *et al.,* 2020). Oils for the production of biolubricants are obtained from plants or animals, and modified to get biolubricant (Tulashie, 2020). Different steps involved in modification include esterification reaction, transesterification reaction (to produce biodiesel as an intermediate) and double transesterification to produce biolubricant. Trimethylol propane (TMP) was the polyol used in the production of biolubricant from Parinari seed oil and Neem seed oil (Nagaraj *et al.,* 2020). Other works, on production of biolubricant, has been carried out on edible oil, as published by Dibal and Ibrahim (2017), using Castor oil (Bilal *et al.,* 2013), Jatropha curcas oil (Madankar *et al.,* 2013), and canola oil, amongst others. However, the demand for food and increasing world population, has necessitated the search for, suitable, nonedible oil for biolubricant production (Singh and Erween*,* 2020). In this work, two non-edible oils, namely, Parinari and neem oils were used for biolubricant production.

Parinari polyandra Benth plant is found in the Northern, North-Central and Eastern parts of Nigeria. The average height of the tree is about 8 m (Odetoye *et al*., 2011). Its leaves are army green with short tips. The branches are easily breakable (Motojesi *et al*., 2011). Parinari fruit gets fully developed bi-annually in the month of March and October. The ripe fruit can be identified by its dark colour when compared to the early stages of the fruit (Dalziel, 1937; Ogunkunle *et al.,* 2018). The variety and season of harvest of fresh seed kernel affects oil yield giving an approximate range between 31-60% (Odetoye *et al*., 2011). Variation in fatty acid compositions was observed in the oil extracted from the seed in the month of April and November, which suggested different application advantages either appropriate for biodiesel production or alkyd resin production (Ogunkunle *et al*., 2020; Odetoye *et al*., 2014, Motojesi et al., 2011).

Neem (*Azadirachta indica)* is a tree in the family *maliaceae*. It grows in different parts of Nigeria. The evergreen tree is large, reaching 12 to 18 meters in height with a girth of up to 1.8 to 2.4 meters (Odetoye *et al*., 2014). The seeds have 40% oil which has high potential for the production of biodiesel. It has a higher molecular weight, viscosity, density, and flash point than diesel fuel (Ogunkunle *et al*., 2020). Neem oil is generally light to dark brown, bitter and has a strong odor that is said to combine the odors of peanut and garlic (Fazal *et al.,* 2011; Shahin *et al.,* 2007). Neem comprises mainly of triglycerides and large amounts of triterpenoid compounds. It also contains polyunsaturated fatty acids such as oleic acid and linoleic acids (Muthu1 *et al.,* 2019).

Lubricants are substances employed for the reduction of friction in mechanical components of vehicles that could lead to wear, tears or heat emission (Bilal *et al.,* 2013). Lubricants exist in the three phases which could be solid, liquid and gas (Singh *et al*., 2021). Lubricants have enormous variety of applications in automobile engines, mechanical parts, refrigeration systems and compressor (Musa *et al.,* 2015). They are classified into two major groups (Shah, *et al.,*2021); mineral oil and biolubricant. Mineral oil lubricants are obtained from crude oil sources (Shah *et al.,* 2021). Mineral oils are harmful when disposed improperly or accidentally spilled into the environment (Monticelli 2018). This discharge of mineral based lubricant has led to environmental pollution and has shown a need for an alternative source of lubricant, that is environmentally friendly (Salimon 2010; Ahmad, 2021). Research is recently targeted towards alternative biolubricants in resolving the challenges faced from the use of mineral based lubricant (Ahmad 2021). Biolubricants have the ability to biodegrade easily and fast (Noreen *et al*., 2021). They are also nontoxic to humans, aquatic live and land (Salih *et al.,* 2011a). They may be based on oils extracted from plants, animals or esters manufactured from modified oils. Examples of oil used for the purpose of biolubricant production include jatropha seed oil, castor seed oil, soybean oil, sunflower oil, amongst other (Cavalcanti *et al.,* 2018; Odetoye *et al*., 2016; Kumar *et al.,* 2021).

2.0 Materials and Methods

2.1 Materials

Crude parinari oil was extracted from parinari fruits that were harvested in the month of October, 2019 at the University of Ilorin, Ilorin, Kwara State, Nigeria. Neem seed oil was purchased from Sabon-Gari market, Zaria, Kaduna State, Nigeria. All reagent used were analytical grade purchased from Integrated Research Laboratories Oke-Odo, Tanke, Ilorin, Kwara State.

2.2 Refining

The refining was done using degumming method. A clear sample was observed after washing and cooling to room temperature (Odetoye *et al.,* 2014), as shown in Figure 1. In carrying out the degumming, 100 g of Parinari oil and Neem oil were heated to 80℃, using a flat bottom flask. Heating magnetic stirrer was used, and stirred continuously to achieve even distribution of heat. 3% distilled water, was added at 90 ℃ to the continuously stirring oil. 0.2% Phosphoric acid was added to the oil mass and allowed to be stirred for another 1 hour. It was allowed to cool naturally. The refined clear oil was decanted from the phosphatides. The residual water in the oil was evaporated by heating the oil to 100 ℃ using the heating mantle.

Figure 1. Degummed clear sample

Figure 2. Separation of transesterification

2.3 Oil esterification

The refined oil sample of 100 g, was transferred into a two litre 3 necks spherical 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℃ (Chaurasia *et al*., 2020). The oils were then mixed in a three necks spherical bottom flask. 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 ℃. The timer was started simultaneously with the stirrer. After 1 hour, 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 1-hour intervals till the free fatty acid (FFA) was 0.5 or less (Dibal and Ibrahim, 2017). Having esterified the Parinari and Neem oil, the % free fatty acid was reduced to a value less than 0.5% (Amos et al., 2016). The sample was then washed to remove the catalyst as seen in Figure 2.

2.4 Methyl ester synthesis

Methyl ester was prepared using the esterified oil from samples, methanol, sodium hydroxide (NaOH), and further transesterification was carried out by pouring mass of Parinari oil and Neem oil respectively into conical flasks as recorded in Tables 1a and 1b. The reactor assembly as seen in Figure 3, was then heated to 60 ℃. The content of the set up as seen in Figure 3 are, the oil sample, methanol of 10.8% with respect to the mass of the oil (g) and NaOH, which was the catalyst (NaOH was 1.2% of the mass of the oil), heating mantle with a stirring rod, a thermometer and retort stand. The same procedure was repeated for every other sample, as shown in Table 1. (Ogunkunle *et al*., 2018).

Table1: Quantity of reagent used for transesterification of parinari and neem oil

	Mass of oil (g)		NaOH (W_1/W_2 1.2%)		Methanol (W_1/W_3 10.8%)	
Sample	Parinari	Neem	Parinari	Neem	Parinari	neem
1	200.10	200.00	2.40	2.40	21.60	21.60
2	205.00	201.15	2.46	2.41	22.14	21.72
3	199.95	200.25	2.39	2.40	21.49	21.63
4	202.23	200.10	2.42	2.40	21.82	21.61

Where: W_1 is mass of oil (g), W_2 is mass of NaOH (g), W_3 is mass of Methanol (g)

2.5 Biolubricant synthesis

After the completion of the transesterification, the catalyst and glycerol compound were separated from the methyl ester mixture, then the ester compound was washed using hot distilled water (Amos *et al.,* 2016; Bilal *et al.,* 2013; Dibal *et al.,* 2017). Thereafter, the unreacted methanol and trace moisture were removed by heating to a temperature of 105℃ for 1 hour. Trimethylolpropane was used in order to modify the property of the biolubricant. For this process, 15% TMP with respect to the mass of the ester produced was mixed with fatty methyl ester. After an hour 0.8% wt/wt catalyst with respect to the mass of the ester produced was added. The mixture blended until foam formation stabilized and the additives completely solubilised in the ester by continuous heating and stirring with a magnetic stirrer (Ahmad, 2021).

Table 2: Quantity of Trimethylolpropane (TMP) reacted with PFAME and NFAME

	Mass of oil(g)		TMP (15% of bio-diesel)		biolubricant produced (g)		Time (hrs)
	PFAME	NFAME	PFAME	NFAME	PFAME	NFAME	
Sample 1	21.54	20.05	3.23	3.01	24.77	23.06	3.0
Sample 2	22.28	20.10	3.34	3.02	25.62	23.12	3.5
Sample 3	21.44	20.05	3.22	3.01	24.66	23.06	3.0
Sample 4	20.20	20.15	3.03	3.02	23.23	23.17	3.5

Figure 3. Transesterification set up

3. Results and Discussion

3.1 Physico-chemical property

The kinematic viscosities at 40 °C and 100 °C for both parinari based lubricant and the neem based were compared with the SAE (Society of Automotive Engineers) standards at the said temperatures. Furthermore, the viscosity index, specific gravity and flash point were also compared as shown in Table 3, Figures 4 and 5.

Table 3. Physical and chemical properties of the sample

	Viscosity (cP) at 40 °C	Viscosity (cP) 100 °C	Viscosity index	Specific gravity	Flash point (°C)	Fire point (°C)
SAE 30	--	9.3-12.5	95.0	0.870	204	--
SAE 40	--	12.5-16.3	110.0	0.880	220	--
SAE 50	--	16.3-21.9	95.0	0.890	220	--
Parinari oil	195.2	87.8	--	1.00	--	--
PBL 1	40.7	15.5	174.1	1.02	82.0	93
PBL 2	66.0	24.2	161.6	1.07	86.0	94
PBL 3	38.4	12.1	174.4	1.02	78.0	92
PBL 4	67.4	28.6	159.7	1.01	89.0	97
Neem oil	78	64.8	--	0.39	156	--
NBL 1	647.3	171.6	641	0.44	175	--
NBL 2	522.4	122.9	363	0.41	170	--
NBL 3	480.2	136.4	326	0.49	173	--
NBL 4	356.6	130.7	365	0.40	163	--

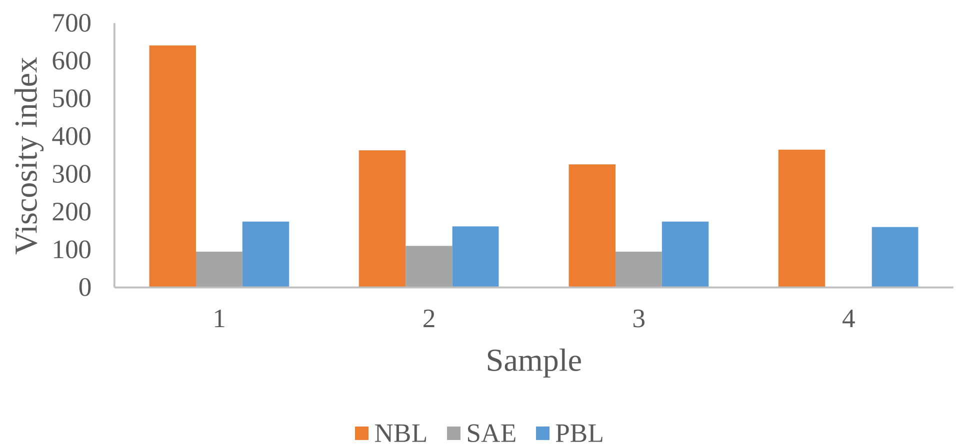

Figure 4: A chart of viscosity index for the biolubricant produced against SAE

The kinematic viscosities were measured at 40 °C and 100 °C because lubricants exhibit unique properties of lubricity at various temperatures. As seen in Figure 3, kinematic viscosity was compared with the SAE, the SAE (30, 40 and 50) standard as recorded in Table 3 shows a lesser kinematic viscosity.

The viscosity index for NBL was relatively higher when compared with that of PBL and SAE standards as shown in Figure 4, from the analysis carried out. The essence of viscosity is in the type of equipment it is required for application. Some equipment is heavy duty and require lubricant with higher viscosity. Lubricants with high viscosity index are mostly used in heavy duty

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equipment, of which the NBL produced fell in such category. The PBL also had a high viscosity when compared, with the SAE standards for SAE 30, SAE 40 and SAE 50. This also implies that the PBL is suitable, for heavy duty mechanical equipment. Lubricants with low viscosity index are more suitably used in mechanical equipment's that do lesser work when compared with heavy-duty equipment. PBL and NBL being biolubricant, also makes it environmentally friendly. Flash point is the lowest temperature at which the lubricants ignite. Comparing all samples, as seen in Figure 5, NBL 3 had the higher flash point for the biolubricants. However, when compared with petroleum based oil as stated by Dibal and Ibrahim(2017), petroleum based lubricant had higher flash point. Hence, petroleum-based lubricant is more suitable for application in heavy mechanical machines that generate high temperature due to friction. This challenge possesses fire hazards in such equipment, as the same lubricant can act as fuel in higher fire point.

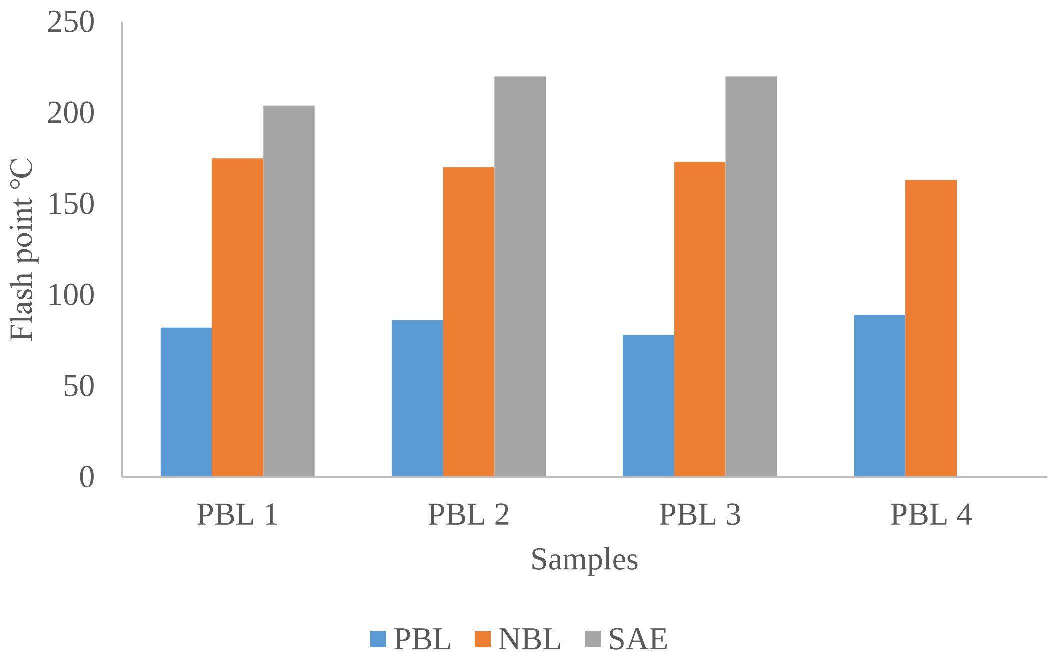

Figure 5: A chart of flash point for the biolubricant produced and SAE

3.2 GCMS analysis for Parinari based lubricant

The GC-MS analysis of the biolubricant shows the fatty acid methyl esters (FAMEs) composition. From the analysis, oleic was 37.33%, being the highest composition, and that is because parinari seed is rich with oleic acid. Other composition was, Methyl ester, which was the primary desired amongst, Tocopherol, which is an antioxidant found in plant and squalene is an organic compound that can be found in plant and animal. They all summed to 48% of the composition. Also 13.76% butanol was also present, butanol can be used as biofuel as reported by Mohamad *et al.,* 2022. Figure 6 shows the respective peaks at different retention time. Each peak shows the FAME present in the biolubricant sample.

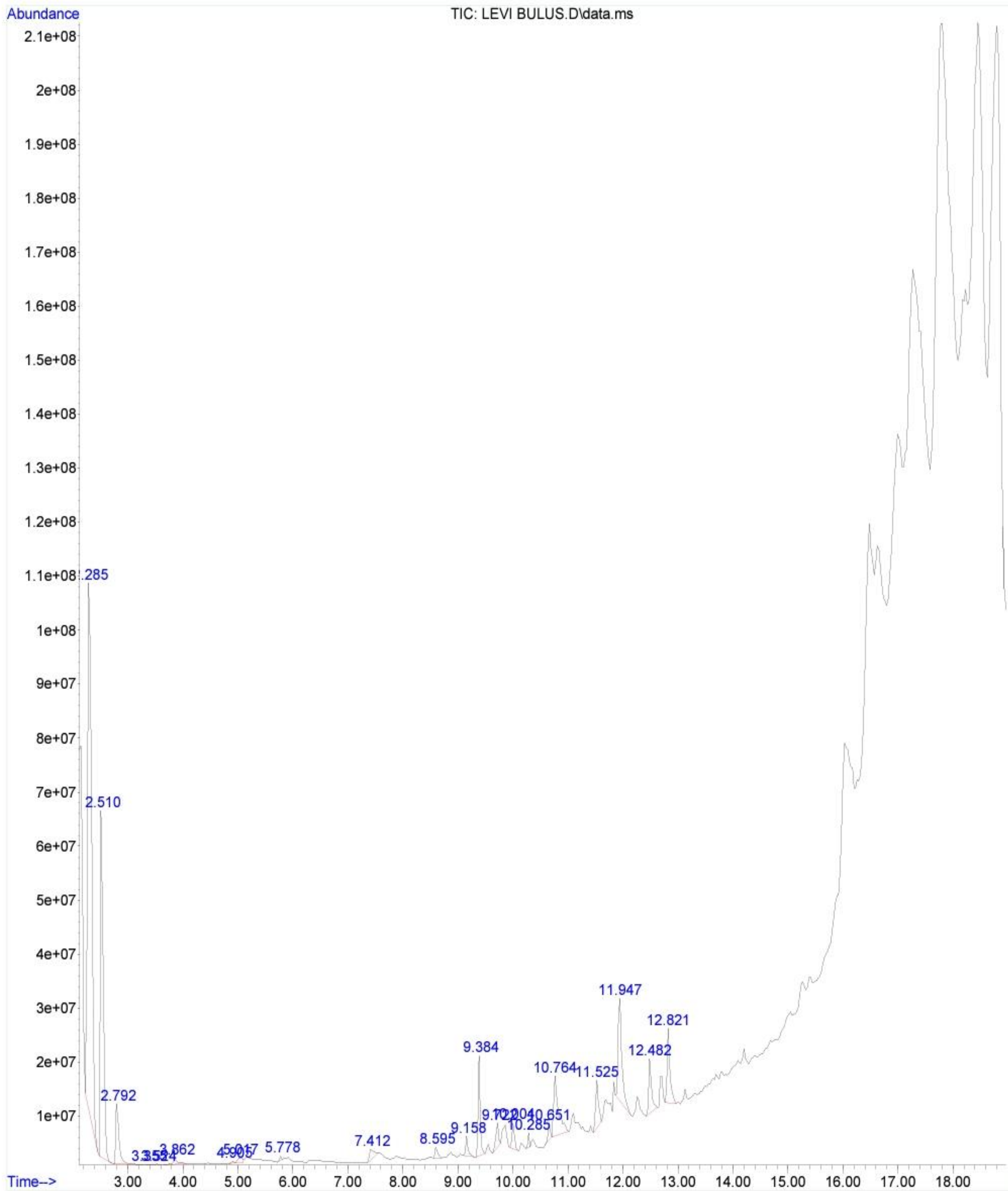

Figure 6: GCMS of fatty acid composition of parinari based lubricant

3.3 GC-MS analysis for neem biolubricant

Octadecenoic acid ester constituted 35.49% and methyl ester constituted 33.54%. Squalene being an organic compound commonly found in plant was 20.13% as seen in Figure 7. The methyl ester composition at their respective time and abundance are, 2.37 is the highest peak and has abundance of 3.4×10^7.

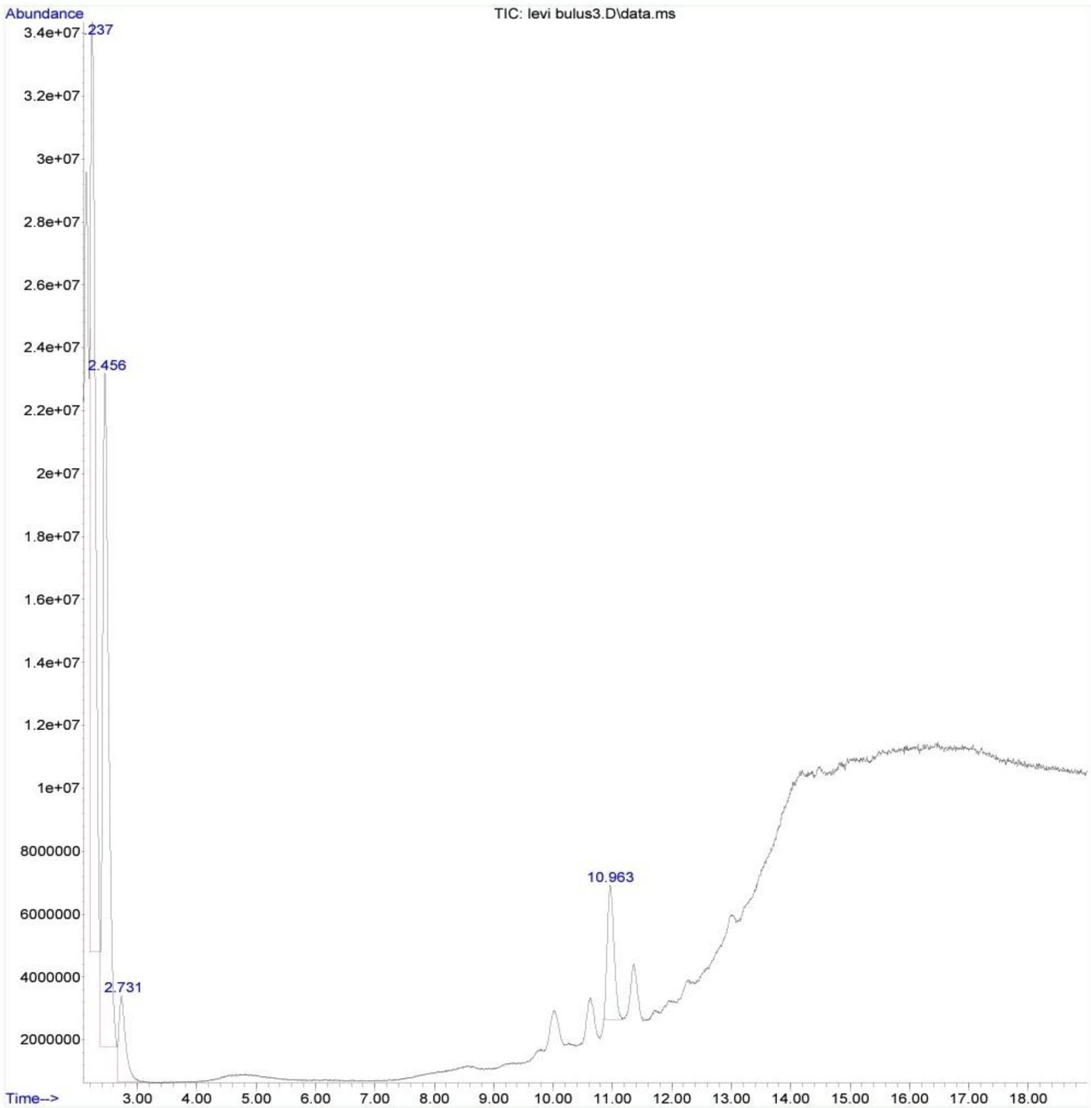

Figure 7: GC-MS fatty acid composition of neem biolubricant oil.

4.0 Conclusion

The production of bio-based lubricant via biodiesel intermediate step was carried out successfully for both parinari and neem oils. The physico-chemical properties were obtained. The kinematic viscosities of the biolubricant samples at 40 °C and 100 °C, range were higher, compared to the SAE 40. However, the flash points of the biolubricants produced were lower, compared to the SAE standard. Biolubricant is environmentally friendly and more researches can be done, to make biolubricant readily available and affordable to encourage the daily consumer. With that, the ecosystem will be less polluted. Thus, further work is recommended on to improve the flash point and other lubricating property of the biolubricants.

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