

Formulation of Biogreases derived from Mahua and Karanja Oil

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Abstract

The present study reports the parametric investigation conducted on the formulation of biofuels, biolubricant/ biogrease from Mahua (*Madhuca Indica*) oil and Karanja (*Pongamia Pinnata*) oil. Biogrease complex has been formulated in 90:10, 80:20 and 70:30 oil/soap ratios for thickness from the oils, their methyl esters and the tri-esters. The experimental results show that the biogrease prepared from Karanja TMP triester showed a drop point of 176 °C which was higher as compared to biogrease prepared from Mahua TMP triester which showed a drop point of 172 °C. The water wash out resistance for both the biogreases was found to be 7%, which is comparable with conventional greases. Based on the cone penetration index, both the biogrease samples were found to fall in NLGI Grade 2 of greases, which is the most common grade of conventionally used greases. Such Biogreases can be effectively used in applications where these greases are directly released into the open atmosphere after their use like agriculture or marine machines and vehicles.

1. Introduction

Renewable resources are always gaining importance due to its availability and positive impact on environment. Vegetable oils are employed as a starting material for developing ecofriendly consumer products due to government regulations as well as increasing the societal concern. A lubricant is applied between two moving surfaces of a machine which reduces friction, thus increasing its efficiency and reducing the wear. It is generally present in any of the three forms viz. liquid, semi solid or solid between two surfaces which undergo relative motion. Liquid lubricants possess certain shortcomings like low viscosity and lower dropping point in machines operating at high pressure and temperatures and are not capable to perform where an exponential rise in these operating conditions was observed. This has triggered the idea behind the development and application of greases (Adhvaryu et al., 2004). Solid or semi-solid lubricants are termed as grease which is nothing but a uniform mixture of a thickener (soap) in a liquid lubricant (Mang & Gosalia, 2017).

Greases are employed in several machines where lubrication is required along with the reduction in friction between two moving parts of the machine, thus maintaining proper mobility under the application conditions (Mendoza et al., 2011). Greases also restrict the entrance of other foreign materials and contaminants in the system. The viscosity of grease allows them to act as a sealant to prevent the leakage of lubricant, its dripping and throw-off during the operation (R. Kozdrach, 2020). It is necessary for a grease to maintain its consistency during service and to act as an effective sealant (Schneider, 2006).

A typical grease composition contains 60–95% base oil (generally mineral oil), 5–25% thickener (which is generally an organic or inorganic soap) and 0–10% additives. Additives improve the performance of grease and increase its life by improving properties like oxidation stability, resistance to wear & corrosion, antifoaming characteristics, extreme pressure handling characteristics etc. (García-Zapateiro et al., 2014; Panchal et al., 2015).

Traditionally mineral oils were utilized as liquid lubricant for formulation of greases (Pilusa et al., 2013; Sánchez et al., 2008), but in recent years vegetable oils are gaining importance due to rising pollution problems and the need towards the sustainable development of renewable products. In this sense, it has been supposed that every year millions of tonnes of oils are disposed of in the environment, which can contaminate groundwater for up to next hundreds of years. Beside this, heavy taxation is paid to clean up the oil spills, which will otherwise be dangerous to aquatic life. Thus there has been constantly increasing demands for the green biodegradable materials that can be used as lubricants.

Vegetable oils are becoming more popular nowadays to substitute the age old petroleum oils for the synthesis of lubricants and greases because they have similar long chain structure as that of long chain hydrocarbons present in petroleum oils with the additional advantages of their renewable, non toxic nature and the low cost of production along with no harms to the environment (Gul et al., 2020; Mahadi et al., 2019). Recent raise in the environmental standards and awareness is the primary cause for development of such renewable products. Vegetable oil-based products are also safe to dispose of after their use, due to their environment-friendly and non toxic nature. Biofuels are already gaining popularity throughout the world because of their advantages and advantages over traditional petroleum fuels and also a chance to reduce the dependency on conventional fuels (Casas-Godoy et al., 2020). Now, a parallel and comparable growth is expected for biogreases, which are simply produced from non-edible vegetable oils to serve as specialty products for applications of lubrication (Rafał Kozdrach, 2021). The idea of manufacturing biolubricants based on vegetable oils and converting them into biogreases has attracted many researchers to develop progression tools for commercial production of such novel products with similar physicochemical properties and tribological characteristics (McGuire, 2014; Vafaei et al., 2021). Such biolubricants can be used as base stock, in mixtures or as additives. Biolubricant blends with selected additives can also show excellent lubricant properties. Reducing the dependence on the petroleum based lubricants and enhanced properties propose it as a valuable product in industrial applications.

A review on properties, manufacturing processes, and applications of biolubricants has been made by Maleque et al (Maleque et al., 2000, 2003; Masjuki et al., 1999). Porfiryev et al (Porfiryev et al., 2020) prepared low temperature grease (LTG) with lithium soap of stearic acid with base oil such as mineral oil, synthetic poly- α -olefin oils and the oil obtained from hydro processes. LTG obtained from these combinations is used for multipurpose due to its properties such as dropping point that was 190°C, colloidal stability 20%, wear scar diameter 0.5mm, effective viscosity at -60°C was 1200 Pa-s. Silica lubricating greases are also used for multipurpose because they can operate at high temperatures (Skibińska, 2020). Silica lubricating grease prepared from waste transformer oil (WTO) and also from fresh transformer oil (FTO) and after then compared the properties of both biogrease from FTO and WTO there was no large difference between the properties of both (A. Japar et al., 2019). Yunus et al reported mini pilot reactor for converting palm oil to biolubricants and explored effects of various parameters on the overall conversion (Sulaiman et al., 2007).

Biogrease was also synthesized from residual jatropha oil with lithium stearate/oleate soaps and the additive zinc dialkyldithiophosphate. The dropping point of grease is between 185°C – 195°C and the NLGI number is 2, which means high potential grease. The biodegradability of these oil are tested by converting into CO₂ and which was fully converted into CO₂ before the period of incubation, the toxicity also checked by growth of bacterial cell NS/8 and there was no change in the curve which shows that there will be no toxic effect of this type of biogrease (Nagendramma & Kumar, 2015).

Another two types of grease, lithium complex grease and polyurea grease were synthesized by earlier workers using mineral and polyalphaolefin oil. The dropping point of both greases at different ratios 80:20, 85:15, 90:10 are determined. The highest dropping point for LCG was 315.33°C and for PCG was 345°C. Also penetration was determined; the maximum values for both the grease were 28.64 and 42.64. These both greases also had other good properties such as colloidal stability, corrosion resistance, and thermal stability and these properties gave good results when both greases with 80% wt of mineral oil were used(Ren et al., 2021).

Biogreases have a wide range of applications in various fields such as forestry machinery, construction vehicles, rail curve, rail flange and marine, hydroelectric powered dams, refuse, mining, dredging, agriculture, plant operations(Japar et al., 2018).Biogrease has huge market demand, when biogrease prepared from edible oil and esters then the production cost of the biogrease was increased but when non edible oil is used then the price of biogrease decreases. Also due to its biodegradable property, it is both economical and environmentally friendly(Sharma & Singh, 2017). The biogrease prepared from karanja and neem oil has application of driers(Mahale et al., 2017).The weather conditions of India, mainly Vidarbha region, are suitable for Mahua (MadhucaIndica) and Karanja (Pongamia Pinnata) plantation and growth(Banka & Parikh, 2019). Also the non-toxicity of these plants and oils is less as compared to the other non-edible oils. Thus, Mahua and Karanja oils were selected for the present work.

2. Material And Methods

2.1 Materials

Purified Mahua oil and Karanja oil were purchased from the local market. The properties of both the oils were determined in the laboratory and are given in Table 1. Trimethylolpropane (TMP) 98%(Aldrich), potassium hydroxide, calcium hydroxide and sodium hydroxide pellets (Sigma-Aldrich), sodium methoxide 95%powder (Sigma–Aldrich), methanol 99.8% (Sigma–Aldrich)were used in the experiments for product synthesis.

2.2 Fatty Acid Methyl Esters (FAME) and Triesters of Oil:

The free fatty acids (FFA) content was found to be higher. Hence the oils were converted to their respective fatty acid methyl esters (FAME) using two step esterification and transesterification process(Baskar et al., 2017; Patel & Sankhavara, 2017). Methanol was utilized for the conversion and sodium methoxide was used as a catalyst. The upper layer obtained after the complete process was FAME which was further purified to remove traces of methanol(Sastry & Murthy, 2015).

Synthesis of triesters (biolubricants) was carried out by the reaction of Mahua oil methyl ester and Karanja oil methyl ester separately with Trimethylolpropane (TMP) in 500 ml batches using sodium methoxide (in 30% methanol) as catalyst. The molar ratio of respective methyl esters to Trimethylolpropane was kept as 4:1 and the amount of catalyst used was 1% w/w. The reaction was carried out at a temperature of 120°C for four hours (4 hours) in an oil bath along with the magnetic stirring arrangement. The reaction was carried out under vacuum conditions to promote a forward reaction(Heikal et al., 2017). The entire methanol produced was removed from the reaction mixture. Oleochemical esters such as Trimethylolpropane (TMP), pentaerythritol (PE), and neopentylpolyol (NPG) imparts good low temperature fluidity at low temperature and hence considered to be suitable for further applications.

2.3 Biogrease:

This step is again divided in two parts viz. soap synthesis and blending of soap with base oil. Three different ratios of oil to soap were studied for formulating grease from Mahua oil and Karanja oil. Further, three different base oils namely original refined oil, FAME and Triester were also used individually to prepare multiple types of grease.

2.3.1 Soap:

Approximately 50 g of Mahua oil was weighed into a 250 ml beaker and 40 ml of ethanol was added to it. Finally, 25% (m/v) sodium hydroxide based was added in the oil. The mixture was then heated indirectly for about 20 min, providing it constant and vigorous stirring. Disappearance of the odor of alcohol indicated the completion of reaction giving a viscous soap like mixture. The beaker was then placed in an ice-water bath. The soap was precipitated by adding 300 ml of a saturated sodium chloride solution to the soap mixture along with vigorous stirring. This increased the density of the aqueous solution by solidifying the soap and removed the excess sodium hydroxide from solution. The soap was then filtered using filter paper and washed with ice-cold water. Similarly, Mahua oil was replaced by Karanja oil to prepare Karanja oil based metallic soap. By similar process two other types of soap were also prepared from potassium hydroxide and calcium hydroxide.

2.3.2 Soap Blended with Base Oil:

First grease sample was formulated using the Mahua oil as base oil. Metallic soap was used as a thickener to formulate Mahua oil based grease. Thickener was thermo-mechanically dispersed in the oil matrix, using a high speed stirrer. Initially 80% of the total volume of the oil was charged in the reactor and heated up to 120°C. Then 20% of the soap was added and stirred vigorously at 200 rpm for intimate mixing. Temperature was gradually raised to 200°C with constant stirring. The mixing was continued till the desired consistency of the product was obtained, and then the temperature was lowered gradually. Stirring was continued while decreasing the temperature to avoid settling of soap, with decrease in temperature it was observed that the viscosity of oil increased gradually. Till reaching the room temperature the viscous oil turned into semisolid grease. No additives were incorporated in the formulation of grease at this stage (Buhlak et al., 2014; Fan et al., 2014; Nagendramma & Kumar, 2015).

Similarly, grease samples with oil to soap ratio of 70:30 and 90:10 were also prepared and compared. Furthermore, Mahua oil was replaced by its FAME and then triester for biogrease synthesis. Similar process steps were followed for Karanja biogrease synthesis. The formulated greases were then compared for

various ASTM standards and grease testing parameters viz. penetration index (worked), NLGI grade, dropping point and water wash out resistance (ASTM International, 2002). All the ASTM tests of the prepared grease samples were done using the standard equipment from Maharana Instruments Mfg. Co., Ajmer, India.

The consistency or hardness of grease is defined as its ability to resist deformation under the application of force. The consistency of the prepared samples of lubricating greases was measured by cone penetration test according to ASTM D217. The test was performed by dropping a cone of fixed dimensions and mass, in a standard cup containing worked grease for five seconds at 25 °C. The depth of penetration of the cone in the grease sample was measured in tenths of a millimeter and further it was used to determine the NLGI grade of the grease.

Dropping point is an important property of the grease and it is nothing but an indicator of its heat resistance. Dropping point was performed according to ASTM D2265. It was measured by taking a grease sample in a standard orifice and increasing its temperature at which grease became fluid enough and fell through the orifice of the test apparatus under test conditions. The capability of lubricating grease to resist the water effects without significant losses in its lubricity is called water washout resistance. Water washout resistance of the prepared samples was measured in ASTM D1264. The grease samples were applied on a ball bearing and then the bearing was impinged with a jet of water at the specified flow rate and temperature. The percentage of test grease washed out in one hour was determined on a weight basis.

3. Results And Discussion

3.1 Triesters from Mahua and Karanja Oil:

Both Mahua oil and Karanja oils are non-edible oils. However, Mahua oil has some uses in oleochemicals like soap manufacturing but both the oils can prove to be the cheapest raw material for methyl ester (biodiesel) synthesis and triester (biolubricant) synthesis. Hence, these oils can be considered as an effective substitute feedstock for manufacturing eco-friendly lubricants.

Table 1 shows the detailed analysis of Mahua oil and Karanja oil which were procured and then analyzed according to the ASTM standards. It was observed that the FFA content (Acid value) of both the oils was on the higher side, hence a two-step procedure of esterification and transesterification process was followed for FAME production (Patel & Sankhavara, 2017). Respective FAMES of Mahua and Karanja oils were then reacted separately with TMP for individual triester (biolubricant) synthesis.

There are many types of grease prepared from synthetic esters such as lithium grease with different oils such as paraffinic oil, naphthenic oil, poly- α -olefin (PAO) and polyol ester (PE) (Zhang et al., 2021). The various properties of lithium biogrease with different oils are studied and found that lithium biogrease with PE has a flat structure in SEM analysis which is stable for friction-reducing. Lithium - naphthenic oil based grease is more suitable for low noise bearing lubrication. But the different oil used in the preparation of lithium biogrease has increased the cost of biogrease. The formulation of biodegradable grease prepared from waste cooking oil, spent bleaching earth and fumed silica as an additive current research topic these days. The dropping point and NLGI number both increased by the addition additive. This type of grease is developing these days due to environmental problems. The biogrease prepared from waste material is both economical and environment friendly (Abdulbari et al., 2011; Abdulbari & Zuhan, 2018).

Transesterification of oils is a reaction that yields FAMES along with glycerol as a byproduct. Further transesterification of FAME with commercial polyols like TMP yields triesters of polyols. These triesters have higher resistance to temperature and oxidative degradation as compared to the original vegetable oils (Heikal et al., 2017). Thus, the greases formulated from these triesters will also have higher resistance to temperature and oxidative degradation in contrast to those formulated from original raw oils. Analysis of methyl esters and triesters of Mahua oil and Karanja oil are described in the following Table 1. From Table 1, it is clear that viscosity index (VI), which is the major property for any lubricant, is maximum for Mahua oil triesters i.e. 103.53. Though it is lower than the VI of Mahua methyl ester, the required viscosity of methyl ester is also very low.

In order to investigate the chemical composition of those triesters, FTIR tests were performed. The FTIR spectra of Mahua oil based TMP triester and Karanja oil based TMP triester are shown in the following Figs. 1 and 2 respectively. The FTIR spectrum of Mahua TMP triester shows absorption peak at 1739 cm^{-1} and for Karanja TMP triester the peak is obtained at 1743 cm^{-1} that peak is due to C=O and C-O stretching vibrations of carbonyl group in esters (Pavia et al., 2001). These peaks confirmed the presence of oxygen and proved that TMP triesters are formed in both the systems of Mahua oil and Karanja oil. The 2922 cm^{-1} band is most likely to group stretching vibrations -C=O in mahua oil and the 2921 cm^{-1} band is due to overlapping bands characteristic for symmetrical and asymmetric vibrations of the methyl and methylene groups (-CH₃, -CH₂-) in karanja oil (R. Kozdrach & Skowronski, 2019).

Bashiri et al. (Bashiri et al., 2021) explained the formation of biolubricants from sunflower waste cooking oil in which formation of triester in FTIR showed the peak at 1738 cm^{-1} that is due to carbonyl vibration. The formation TMP triester showed the peak around 1744.19 cm^{-1} of C=O group in ester during esterification of rubber seed oil with TMP (Ishak & Salimon, 2012). According to the lubricating oil spectrum, it is observed that the intense band at 1738 cm^{-1} corresponds to the carbonyl group of the ester formed after the transesterification reaction (Guimarães et al., 2021). The absorption peak of Jatropha oil based TMP esters showed at 1740 cm^{-1} (Heikal et al., 2017). The absence of -OH peak in the region of 3300 cm^{-1} showed that the -OH groups present in TMP were successfully converted to ester groups of biolubricants.

3.2 Biogrease from Raw Oil

Non edible oils can be directly used as base oils for grease formulation without any treatment, but they have certain disadvantages like poor thermo-oxidative and hydrolytic stability, low temperature of performance and high pour points. These drawbacks restrict the direct use of these non-edible oils as base oils for grease formulation. Hence the oils can be chemically modified by transesterification, epoxidation, enzyme catalyzed esterification, etc. for their use as

biolubricants and biogreases (Cecilia et al., 2020; Sharma & Singh, 2017). Table 2 and Table 3 show the analysis of grease samples prepared using Mahua oil directly as base oil with different ratios of soap prepared in the laboratory as mentioned above. Three samples were prepared with oil to soap ratio as mentioned in the table. The important properties of grease viz. cone penetration index, dropping point and water wash out characteristics were determined according to ASTM procedures (ASTM International, 2002). Oil to soap ratio of 70:30 resulted in highest values of all the standards, but the grease was a dry and rough lump which was inconsistent. This nature is unfavorable for greases. On the other hand, oil to soap ratio of 80:20 resulted in grease which was consistent and smooth in texture. Whereas, the ratio of 90:10 resulted in very wet, oily and very soft grease. Sukirno et al (Sukirno et al., 2009) synthesized the biogrease from refined bleached deodorized palm oil with lithium soap as a thickener. The process for the preparation of bio grease used was saponification → soap dilution → re-crystallization → homogenization. The cone penetration index with oil to soap ratio 80:20 was 190 and NLGI grade by using this value comes out to be 4. These both value showed that grease prepared by this method the consistency of grease was like hard ice cream (Ross, 2013). This type of grease has better antiwear property than the mineral oil grease and has application of protecting from metal to metal surface. In the present study according to the value of cone penetration index and NLGI grade the consistency of grease was like peanut butter (Ross, 2013). Similar results were obtained, when Karanja oil was directly used as base oil for grease formulation as shown in Table 2 and Table 3. The appearance of Karanja oil grease was more consistent and fibrous as compared to Mahua oil grease. Also, the dropping point for Karanja oil biogrease was more as compared to Mahua oil biogrease. But on the other hand its cone penetration index was also a bit higher (Fig. 3), though it does not affect the NLGI (National Lubricating Grease Institute) grade of the grease. Rahman et al (Abdu Rahman et al., 2019) produced the biogrease from waste engine oil with sodium soap. Various properties are determined with different concentrations and found that when the base oil was 80%, stearic acid 10% and sodium hydroxide 10% then the NLGI number was found to be 2 which shows that in this ratio biogrease has good consistency. Various types of vegetable biogreases are prepared such as biogrease from coconut, palm based oil (Skibińska, 2020), olive oil (Sterpu et al., 2016), corn oil and palm oil with calcium stearate soap (Vodounon et al., 2019). The optimum conditions for the preparation of biogrease from these oils are maintained at 100°C with 20–25% wt of soap content. The NLGI number comes out to be 3–6 at optimum conditions. According to rheological study, the palm oil based biogrease was superior to the corn oil based biogrease. Keeping in mind the consistency of the grease, the oil to soap ratio of 80:20 was found to be suitable.

3.3 Biogrease from FAME and Triesters

After selecting the optimum oil to soap ratio, methyl esters of Mahua oil (FAME) and then TMP triesters were used as base oil for formulation of different greases. Analysis of both the samples is tabulated in Table 2. From the analysis it is clear that the grease sample with TMP triester used as base oil gave the best results. Its dropping point was found to be maximum among all other samples i.e. 172 °C. Dropping point is a key test parameter for any grease specification as it determines how well the test grease will perform under applied load at high temperature under actual operating conditions (Suetsugu et al., 2013). Figure 4 shows the trend of increase in dropping point of Mahua biogrease with different base oils.

In recent studies, when biogrease prepared from karanja and neem oil with the oil to soap ratio 70:30 then the result showed that the dropping point of grease from karanja oil was 72°C and from neem oil was 55°C (Mahale et al., 2017) but in this study biogrease prepared from metallic soap using calcium and magnesium and the best property in that was low moisture content. Adly et al (El-Adly et al., 2015) prepared the biogrease from waste cooking and biochar (obtained by pyrolysis of rice straw) and other substances (Stearic acid, calcium hydroxide, colophony, natural rubber and octyl amine) with different proportions. The dropping point of this biogrease is 190°C which is very good as compared to commercial grease whose dropping point is 100°C. The benefit of grease prepared from waste cooking oil and biochar is that it preserves the environment.

Similar samples of grease were prepared and analyzed for FAME and TMP triesters of Karanja oil. It has been observed that Grease prepared from TMP triester of Karanja oil gave even better results in terms of dropping point. Its dropping point was found to be 176 °C. Figure 4 shows the trend of increase in dropping point of Karanja biogrease with different base oils. From the values, it is clear that the higher viscosity of triesters resulted in higher dropping point of the formulated greases. Also the oxidation stability and thermal resistance of the triesters is better as compared to their respective oils, which is advantageous for the greases.

Similar result shows that in Figs. 5 and 6 that was from potassium and calcium based soap and calcium based biogrease showed better result as compared to sodium and potassium based biogrease. However, mahua oil is more preferred as compared to karanja oil, as karanja oil is poisonous to marine life.

4. Conclusion

The formulated biogreases obtained from the triesters of Mahua and Karanja oil are biodegradable and non toxic. These biogreases can be used in all applications and can easily replace mineral oil based greases. Due to its property of water wash out resistance which is found to be 7% which is comparable to commercial grease, therefore it can prove superior particularly in first hand applications like agriculture and forest machinery, rail road and marine applications where the grease is lost to soil or water after use. Biogrease prepared from karanja and mahua oil with the oil to soap ratio 80:20 showed better results. The value of other property such as dropping point for karanja oil is 176 °C and for mahua oil is 172°C and the NLGI grade is 2 for both karanja and mahua oil, both the properties dropping point and the NLGI grade is comparable to the commercial grease prepared from mineral oil. The major drawback of biogrease prepared from raw karanja and mahua oil has poor oxidation stability and thermal resistance as compared to fatty acid methyl ester and triester but biogrease prepared from non edible sources is both economical and environment friendly. A thoughtful selection of base oil and additives is therefore essential for an efficient synthesis of biolubricating oil. Such proposal for the production of biolubricants could be important in improving the economic competitiveness in an industrial process. It is believed that such type of research study could be of assistance to researchers to meet the challenges ahead based on renewable and environmentally friendly products.

Declarations

Ethical Approval - This study is not involving humans or animals. We do the experimental work on the basis previous study of grease .Therefore, the Institutional Review Board Statement is Not Applicable and can be exclude this statement.

Consent to Participate - All authors have read and agreed to the published version of the manuscript.

Consent to Publish – This work is not published anywhere

Authors Contributions –Research & Methodology Amit Agrawal and Sakshi Bawa, Richa Tiwari, Vijay karadbhaje and Pratibha Agrawal .; Writing—original draft, Sakshi Bawa Writing—review & editing, Srinivasan Murthy.

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Tables

Table 1: Analysis of Oils, Methyl Esters and Triesters of Mahua oil and Karanja oil

Sr. No.	Properties	Analytical Method	Mahua Oil	Karanja Oil	Mahua-Methyl Ester	Karanja-Methyl Ester	Mahua Triester (Biolubricant)	Karanja Triester (Biolubricant)
1	Density @ 20°C	ASTM-D1298	0.907 gm/ ml	0.936 gm/ ml	0.8528 gm/ ml	0.88 gm/ ml	0.879 gm/ ml	0.88 gm/ ml
2	Viscosity @ 40°C	ASTM-D445	37.18 cst	54.54 cst	4.45 cst	4.604 cst	69.3 cst	43 cst
3	Viscosity @ 100°C	ASTM-D445	11.66 cst	15.18 cst	3.025 cst	1.94 cst	8.53 cst	6.5 cst
4	Viscosity Index	ASTM-D2270	325.6	304.9	447.5	205.66	103.53	101
5	Pour Point	ASTM-D97	18°C	-1°C	0 °C	-4 °C	0°C	-2°C
6	Flash Point	ASTM-D92	210 °C	220°C	150 °C	140 °C	205 °C	210 °C
7	Fire Point	ASTM-D92	220°C	240°C	165 °C	148 °C	218 °C	219 °C
8	Saponification Value	ASTM-D94	185.13 mg KOH/gm	188.5 mg KOH/gm	–	–	–	–
9	Acid Value	ASTM-D664	26.23 mg KOH/gm	18.40 mg KOH/gm	–	–	–	–
10	Applications				Replaces diesel fuel in the form of blending, has low emission of CO, HC and smoke due to excessive molecular oxygen(Shrivastava et al., 2012; Vijay Kumar et al., 2019)		Thermal stability, High biodegradability Lower toxicity - Good lubricating Cost-effective due to reduced costs of maintenance, disposal, and storage(Salih & Salimon, 2021)	

Table 2: Characterization of Biogrease of Mahua and Karanja oil with different ratios

Sr. No.	Properties	Analytical Method	Biogrease of Mahua oil (with sodium based soap)					Biogrease of Karanja oil (with sodium based soap)				
			oil : Soap Ratio			FAME:	Trieseter:	oil : Soap Ratio			FAME:	Trieseter:
						Soap Ratio	Soap Ratio				Soap Ratio	Soap Ratio
			70:30	80:20	90:10	80:20	80:20	70:30	80:20	90:10	80:20	80:20
1	Appearance	—	Dry and Rough Lumps	Smooth and Consistent	Very Soft and Oily	Smooth and Consistent	Smooth and Consistent	Dry and Rough Lumps	Smooth and Fibrous	Very Soft and Oily	Smooth and Fibrous	Smooth and Fibrous
2	Cone Penetration Index (worked) @25 °C	ASTM – D217	263	280	315	274	270	267	293	325	287	287
3	NLGI Grade	—	2	2	1	2	2	2	2	1	2	2
4	Dropping Point	ASTM-D2265	148 °C	135 °C	120 °C	139 °C	172 °C	171 °C	146 °C	132 °C	155 °C	176 °C
5	Water Wash Out Resistance @ 38 °C	ASTM-D1264	7%	10%	<15%	8%	7%	5%	8%	12%	8%	7%

Table 3: Characterization of Biogrease of Mahua oil with different ratios

Sr. No.	Properties	Analytical Method	Biogrease of Mahua oil (with potassium based soap)					Biogrease of Mahua oil (with calcium based soap)				
			oil : Soap Ratio			FAME:	Trieseter:	oil : Soap Ratio			FAME:	Trieseter:
						Soap Ratio	Soap Ratio				Soap Ratio	Soap Ratio
			70:30	80:20	90:10	80:20	80:20	70:30	80:20	90:10	80:20	80:20
1	Appearance	—	Dry and Rough Lumps	Smooth and Consistent	Very Soft and Oily	Smooth and Consistent	Smooth and Consistent	Dry and Rough Lumps	Smooth and Fibrous	Very Soft and Oily	Smooth and Fibrous	Smooth and Fibrous
2	Cone Penetration Index (worked) @25 °C	ASTM – D217	262	278	317	280	284	268	287	310	287	285
3	NLGI Grade	—	2	2	1	2	2	2	2	1	2	2
4	Dropping Point	ASTM-D2265	142 °C	135 °C	117 °C	147 °C	175 °C	210 °C	185 °C	173 °C	185 °C	192 °C
5	Water Wash Out Resistance @ 38 °C	ASTM-D1264	7%	10%	<15%	8%	7%	<5%	6%	7%	5%	5%

Figures

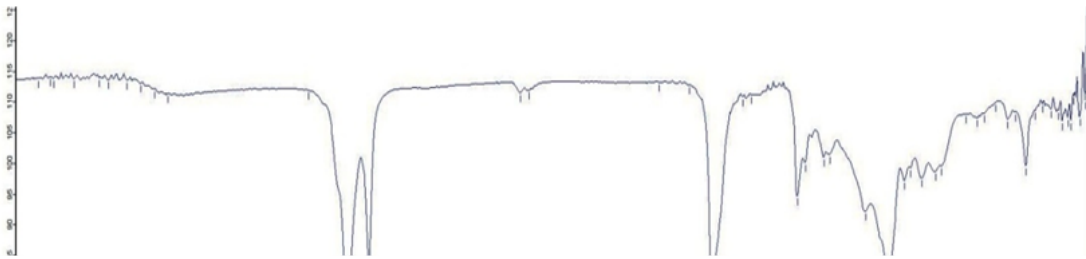


Figure 1

FTIR analysis of Mahua TMP triester



Figure 2

FTIR analysis of Karanja TMP triester

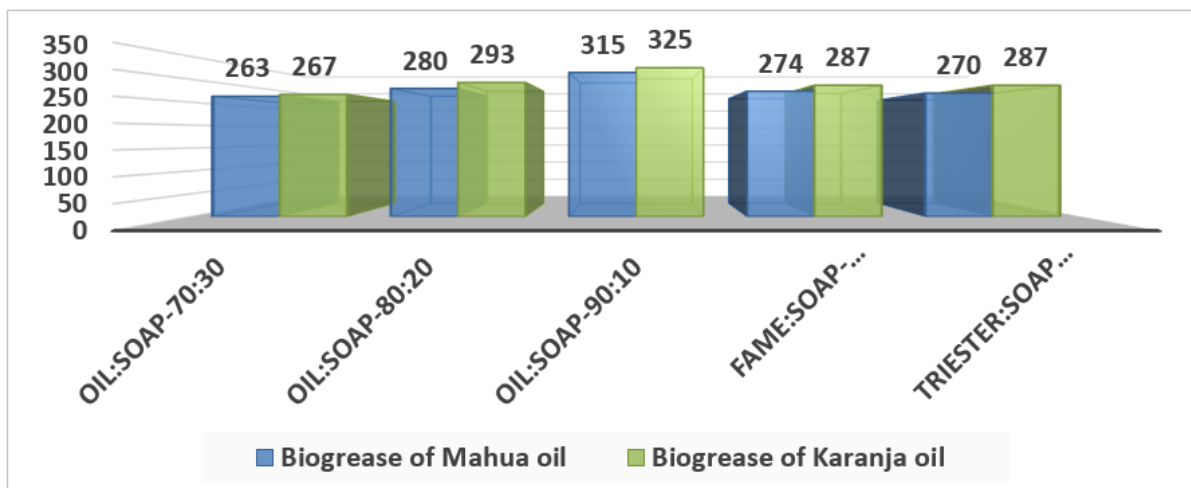


Figure 3

Cone Penetration Index (worked) @25 °C for formulated biogrease of Mahua and Karanja oil

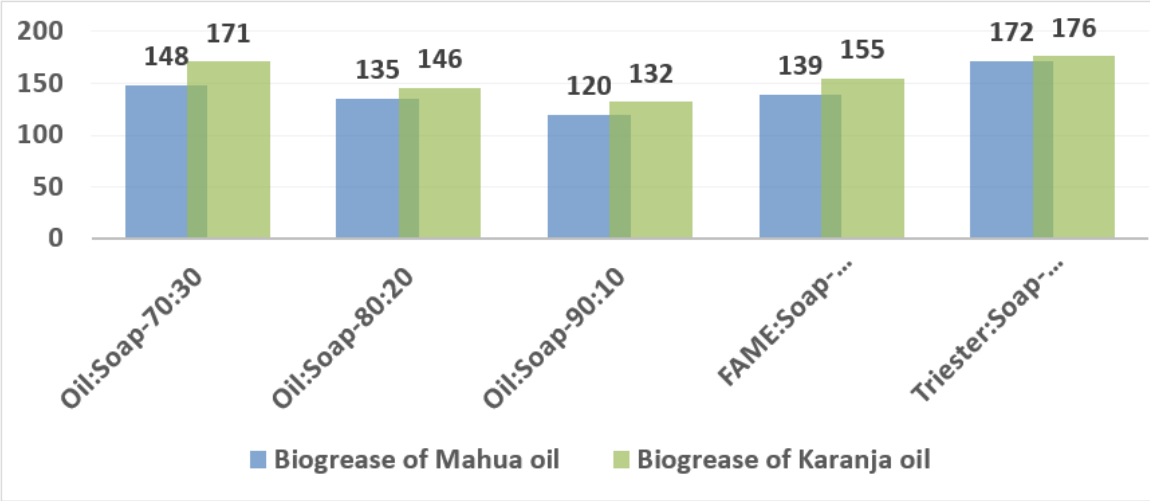


Figure 4

Dropping point for formulated biogrease of Mahua and Karanja oil

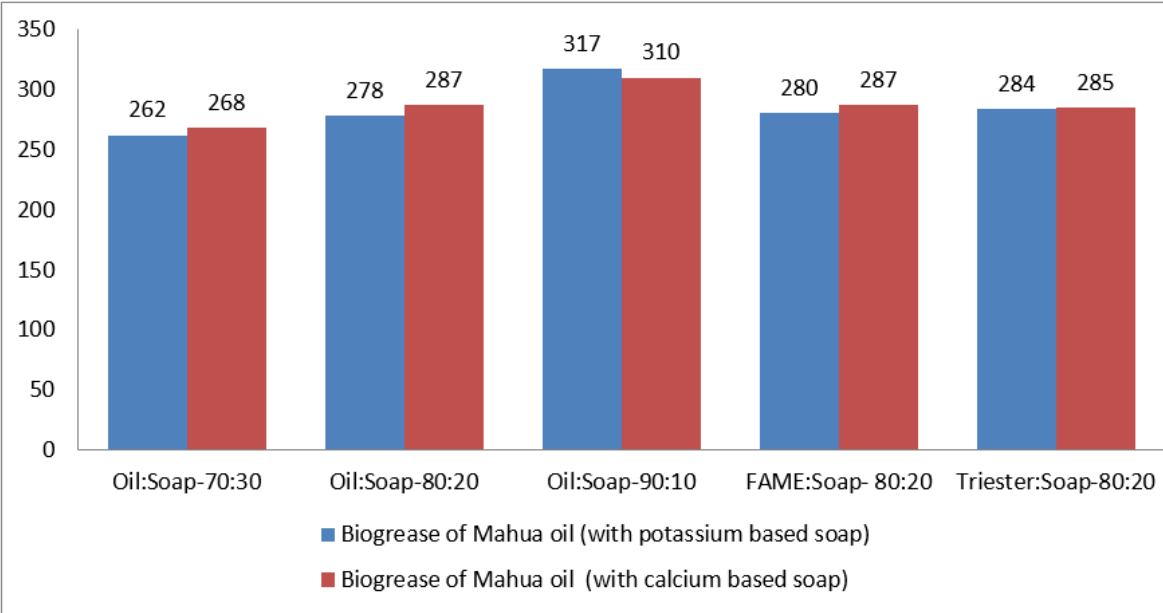


Figure 5

Cone Penetration Index (worked) @25 °C for formulated biogrease of Mahua oil

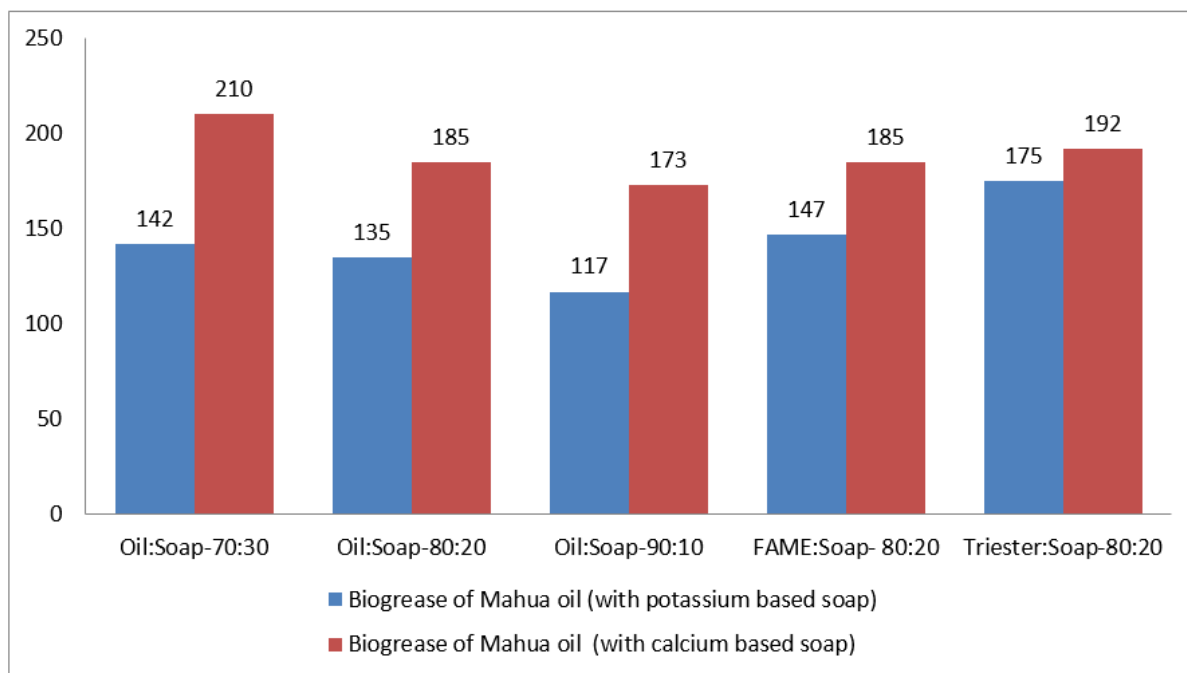


Figure 6

Dropping point for formulated biogrease of Mahua oil