Chapter

Biolubricant from Pongamia Oil

Sabarinath Sankarannair, Avinash Ajith Nair, Benji Varghese Bijo, Hareesh Kuttuvelil Das and Harigovind Sureshkumar

Abstract

Recent researches focus on the development of lubricants from non-edible vegetable oil which are environment friendly and renewable. In the current work, an industrial lubricant is formulated from a non-edible vegetable oil viz. pongamia oil (PO) by blending it suitable additives. The additives such as silicon dioxide (SiO_2) nanoparticles, tert-butylhydroquinone (TBHQ) and styrene butadiene rubber (SBR) were selected as antiwear, antioxidant and viscosity improver additives respectively for the study. Various lubricant properties of the formulated oil (FO) are studied and comparisons were made against neat PO and popularly available mineral oil lubricant viz. SAE 20W40. It is found that the FO possesses superior viscosity index, and lower coefficient of friction than the commercial SAE 20W40. Moreover, the viscosity range, oxidative stability and the wear scar diameter of the FO is also in the range of SAE 20W40. This work is done with an aim of promoting Pongamia agriculture and reducing soil pollution.

Keywords: biolubricant, pongamia oil, agriculture, nanoparticle, viscosity improver, tribology

1. Introduction

The technique or process of using a material to reduce friction and wear between contact surfaces, which are in relative motion, is known as lubrication. It also helps in force transmission, foreign particle transportation and heat transfer. Lubricity is the property of a lubricant to reduce the friction. Lubricants are classified into solid lubricants (e.g.: graphite), semi-solid lubricants (e.g.: grease), liquid lubricants (e.g.: mineral oils based) and gaseous lubricants (e.g.: air). Liquid lubricants are further classified into fixed oil based, mineral oil based and synthetic oil based lubricants on basis of lubricant base stock [1].

Evidences for usage of lubricants for thousands of years have been found. Several methods were adopted by human race from time to time to solve the issues regarding friction and wear. Egyptians used animal fats in ball bearings for lubrication back in 1000 B.C. Oil-impregnated lumber were used to slide building stones in the time of the pyramids and on the axles of chariots dated to 1400 BC, calcium soaps have been found [2]. Romans used thrust bearing and lubricants having rapeseed and olive oil as well as animal fat as base back in 40 A.D. Mineral oil based lubricants were widely used since 1850, but they are non-biodegradable, fast depleting and also have adverse effects on the environment [3, 4]. Nowadays, synthetic oil is considered as a better alternative to these mineral oil based lubricants.

However, these synthetic oil based lubricants are much expensive than the mineral oil based lubricants [5]. The improper after-use disposals of the available lubricants are creating severe environmental issues by polluting the water bodies [6]. Developing an efficient lubricant from a non-edible plant oil base stock is an effective solution to the above issues, as they are biodegradable, renewable and environment friendly [7]. Vegetable oil base stocks also possess high thermal stability, low volatility, good biodegradability, non-toxicity and good lubrication properties in comparison to mineral oil base stocks [8].

The present article aims at developing a lubricant from a non-edible vegetable oil. In the current work, pongamia oil (PO) is selected as the base stock due to its high oleic acid content and non-edible nature [9–14]. Formulated oil (FO) is developed by blending suitable additives in PO. Rheological, oxidative and tribological properties of PO and FO are evaluated and compared against the properties of a commercially available lubricant SAE20W40. Hence the primary objectives of the current work are to:

- To develop a suitable bio-lubricant from a non-edible vegetable oil viz. PO.
- To add suitable additives to PO and enhance its various properties.
- To compare the FO with a commercially available lubricant viz. SAE20W40.

2. Methodology

Rheological properties, oxidative stability and tribological properties of the PO with and without the addition of suitable additives are studied. Various PO blends with the additives are prepared using a magnetic stirrer. Rheological properties (dynamic viscosity, kinematic viscosity and viscosity index) of the oil blends are evaluated by using a rheometer (Anton Par MCR 102) having parallel plate geometry and redwood viscometer. Oxidation stability of the sample is determined using hot oil oxidation test (HOOT) in a dark oven. Tribological properties viz. wear scar diameter (WSD) and coefficient of friction (COF) are acquired with the help of a four-ball tester apparatus. Worn-out portions of the ball specimens are examined initially using an optical microscope and later by a scanning electron microscope (SEM). Chemical properties of the PO such as total acid number, total base number, iodine value, saponification value are also analyzed as per ASTM Standards (**Table 1**).

2.1 Materials and methods

2.2 Experimental procedure

a. BLENDING OF POLYMERIC ADDITIVES TO PO: In the present study SBR and EVA were selected as the viscosity improver additives. The PO is blended with these polymer additives to enhance the rheological properties. The samples of 0.5, 1.5 and 2.5 SBR and EVA in PO are prepared separately with the help of a magnetic stirrer having hot plate.

b. BLENDING OF ANTIOXIDANT ADDITION TO PO: The PO is blended with TBHQ to enhance the oxidative stability. 0.5, 1.5 and 2.5 wt% of antioxidant TBHQ is blended with PO respectively. The blends are prepared using a magnetic stirrer.

Materials	
Category	**Item**
Base oil	Pongamia oil
Commercial oil	SAE20W40
Viscosity improver additive	Styrene butadiene rubber (SBR) & ethylene-vinyl acetate (EVA)
Antioxidant additive	Tert-butylhydroquinone (TBHQ)
Antiwear additive	SiO_2 nanoparticle
Instruments	
Device	**Description**
Magnetic Stirrer with hot plate	Used to blend additives in oil sample
Rheometer (Anton Par MCR 102)	It is a device used to measure the dynamic viscosity of oil samples
Redwood viscometer	It is a device used to measure the kinematic viscosity of oil samples
Hot air oven	Used for hot oil oxidation test of oil samples
Four ball tester	Device used to study the tribological properties of oil samples
Thermo gravimetric analyzer (TGA)	It is used to continuously measure mass while the temperature of a sample is changed over time
Differential scanning calorimetry (DSC)	It is used to measure the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature

Table 1.
Materials and instruments used for the analysis.

c. BLENDING OF ANTIWEAR ADDITIVES TO PO: The PO is blended with nanoparticles of SiO_2 (Particle Size: 15–40 nm) to improve its tribological properties viz. wear scar diameter and coefficient of friction. The blend is prepared by adding SiO_2 in 0.6, 0.8 and 1.0 wt% respectively to PO using a magnetic stirrer.

2.3 Tests conducted

a. *TOTAL ACID NUMBER (TAN):* The TAN of the PO is obtained by titration method. It is a measure of acidity of the oil that is done by dissolving the PO in toluene and then titrating it against potassium hydroxide (KOH) using phenolphthalein as indicator. ASTM D664 standard was used for calculations.

b. *TOTAL BASE NUMBER:* The TBN of the PO is obtained by titration method. It is a measure of basicity of the oil done by dissolving PO in chlorobenzene and then titrating it against hydrochloric acid (HCl) using phenolphthalein as indicator. ASTM D2896 standard was used for calculations.

c. *SAPONIFICATION VALUE:* Saponification value denotes the number of milligrams of potassium hydroxide needed to saponify 1 gof fat according to the conditions specified. It is a calculation of the average molecular weight (or chain length) of all the fatty acids present. ASTM D5558-95 standard was used for calculations.

d. *IODINE VALUE:* The iodine value (or iodine adsorption value or iodine number or iodine index) is the mass of iodine in grams that is consumed by 100 g of a chemical substance. Iodine numbers are used to find the quantity of unsaturation in fatty acids. ASTM D1959 standard was used for calculations.

e. *DYNAMIC VISCOSITY ANALYSIS:* The dynamic viscosity is measured using Anton Par Rheometer MCR 102 in rotation mode having parallel plate geometry. Dynamic (absolute) viscosity is the tangential force per unit area required to move one horizontal plane with respect to another plane, at a unit velocity, while maintaining a unit distance apart in the fluid.

f. *KINEMATIC VISCOSITY ANALYSIS:* Kinematic viscosity is the ratio of absolute (or dynamic) viscosity to density. Force is not involved in this quantity. Kinematic viscosity can be found out by dividing the absolute viscosity of a fluid with the fluid mass density. The kinematic viscosity is measured using Redwood viscometer for PO from 40–100°C.

g. *WEAR SCAR DIAMETER ANALYSIS:* Four ball testing of the oil is done in a four ball tester apparatus, with the sample, given a load of 40 kg at 75°C. The ball is then analyzed using a scanning electron microscope and wear scar diameter is recorded.

h. *COEFFICIENT OF FRICTION ANALYSIS:* Four ball testing of the oil is done in a four ball tester apparatus, with the sample, given a load of 40 kg at 75°C. Calculations were done as per ASTM D 5183-05 standard to test COF.

i. *HOT OIL OXIDATION TEST:* The quickened aging of vegetable oil and PO added with antioxidant are stimulated with HOOT. This is done to find the oxidation stability.

j. *THERMOGRAVIMETRIC ANALYSIS:* The thermal stability of antioxidant selected and that of the formulated oil (FO) are evaluated using the thermo-analytical curves obtained from TGA, Q50 equipment, TA-Instruments.

3. Results

3.1 Preliminary analysis and enhancement of properties of PO

A series of experiments have been conducted as per standards and results are taken as an average of 3 readings having standard deviation of the sample as the error bar. The experimental data are given in the graphs and tables. **Table 2** represents the physicochemical properties of PO and compared with two widely studied bases stock viz. sesame oil and coconut oil.

The density of PO is found to be 0.92 g/cm^3 using a pycnometer, which is lower than that of water. It is found that the acid number of PO is slightly higher than that of the sesame oil and coconut oil. However, for lubricant TAN value should be low as possible [15]. PO has the least saponification value among the three. Low SV indicates long fatty acid chain, which helps in the formation of thick tribolayer [16]. Iodine value of a triglyceride indirectly shows the amount of unsaturation present in it [17]. From the iodine value, it is clear that unsaturation in PO lies between sesame oil and coconut oil. Thus, it is clear from the evaluation of physicochemical

Sl. no	Analysis	Pongamia oil	Sesame oil [9]	Coconut oil [9]
1	Iodine value (g I2/100 g)	88.18 ± 0.19	105.1	9
2	Saponification value (mg KOH/g)	168.75 ± 3.72	191	261
3	Total acid number (mg KOH/g)	3.94 ± 0.14	3.18	0.56
4	Total base number (mg KOH/g)	0.37 ± 0.01	0.41	0.16
5	Density (g/cm^3)	0.92	0.9216	0.92429

Table 2.
Physiochemical properties of PO, sesame oil, and coconut oil.

properties of PO that most of the chemical properties are well suited to the desirable properties of a base stock, which can lead to the development of an eco-friendly lubricant.

The temperature below which the liquid loses its flow characteristics is known as pour point of a liquid [18]. **Figure 1** represents the pour point of PO, which is evaluated from differential scanning calorimetry (DSC). The pour point of PO was found as 6.29°C. The pour point of PO is found to be lower than that of coconut oil [9] due to the presence of more unsaturated fatty acids. Pour point of the lubricants can be reduced using a suitable pour point depressant.

The thermal stability of PO is studied using TGA [19]. From the TGA results (**Figure 2**), the onset temperature of PO for thermal degradation of 98% is found out as 197.6°C. The weight percentage reduction 2% of PO was done by assuming

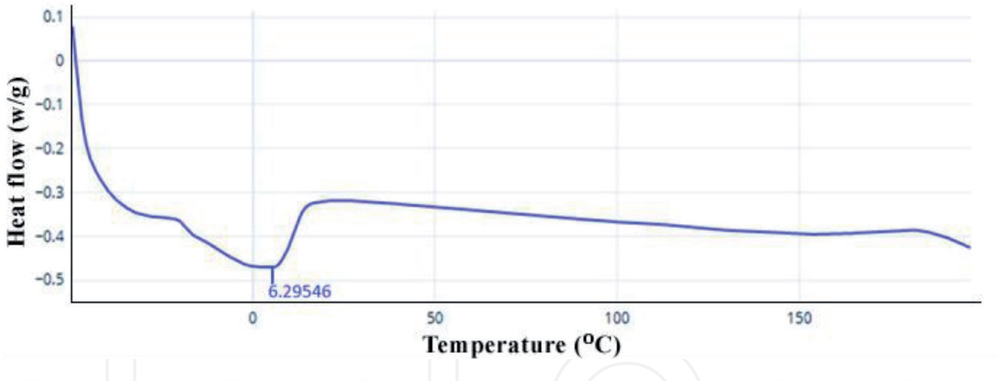

Figure 1.
Differential scanning calorimetry (DSC) curve of PO.

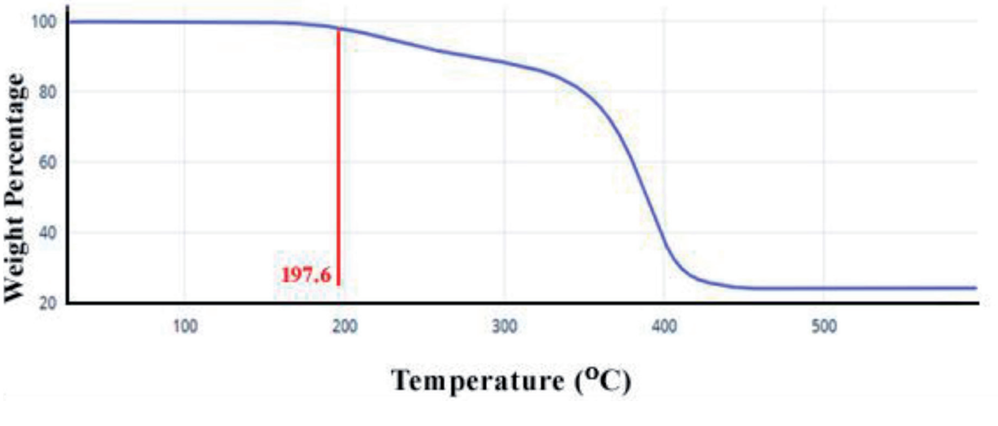

Figure 2.
TGA of PO.

the loss of moisture content and volatile components from it [19]. Thus, by observing the TGA, it is evident that PO is a well suited environment-friendly base stock for a wide range of temperatures. The thermal compared to SAE20W40 which have thermal degradation of at 204.39°C [9].

Table 3 shows the fatty acid profile of PO, compared with that of sesame oil and coconut oil. High amount of oleic acid present in the PO can improve the tribological properties [10]. A larger proportion of saturated fatty acids can adversely affect the pour point of the lubricants.

3.1.1 Analysis of rheological properties

It is clear from **Figures 3** and **4** that the dynamic viscosities of PO at various temperature range is inferior w.r.t commercially available SAE 20W40 (**Figures 3** and **4**). Hence, the dynamic viscosity of PO is enhanced using different weight percentages of EVA and SBR polymers. The variation of viscosity is studied from 25–100°C [20–22].

From **Figure 3**, it is observed that the plain PO is having the lowest viscosity both at lower as well as higher temperatures. The viscosity is found to be improving on addition of EVA in different weight percentages to PO. PO with 2.5% w.t. EVA has shown the highest change in viscosity both at lower as well as higher temperatures.

From **Figure 4**, it is found that SBR tends to show better improvements in viscosity than EVA at different weight percentages. The curve of SBR at 2.5% w.t. in PO is found to be close enough to reference oil SAE20W40. However, from the results obtained from **Figures 3** and **4**, at equal weight percentages of the additives PO + SBR combinations have shown higher viscosity enhancements. Hence, SBR has been selected as the viscosity enhancer in the current study.

Constituent	Pongamia oil [10]	Sesame oil [9]	Coconut oil [9]
Oleic acid	62.98%	42%	5%
Linoleic acid	16.84%	38%	1%
Palmetic acid	9.1%	13%	7.5%

Table 3.
Fatty acid profile of PO and sesame oil.

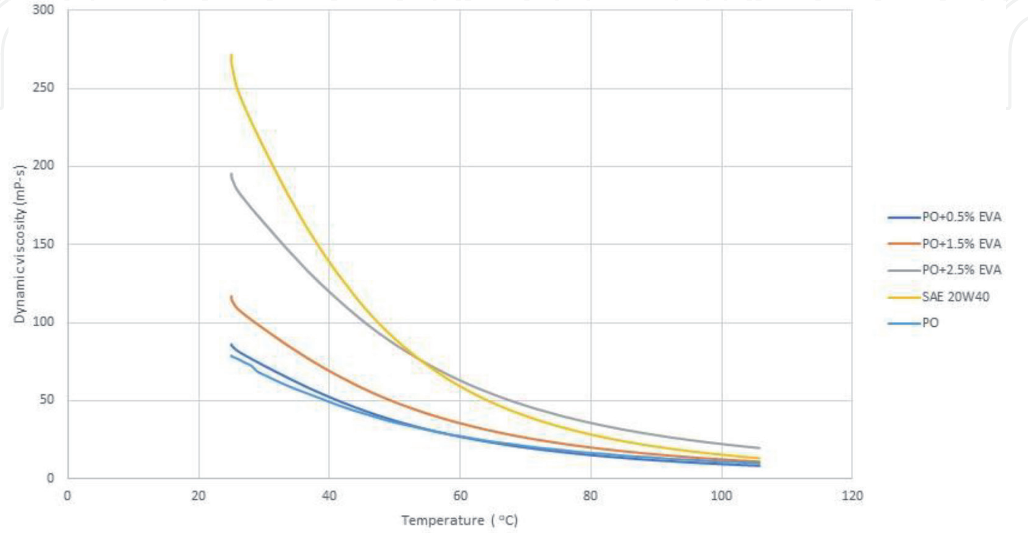

Figure 3.
Dynamic viscosity v/s temperature curve for PO with EVA compared against SAE20W40.

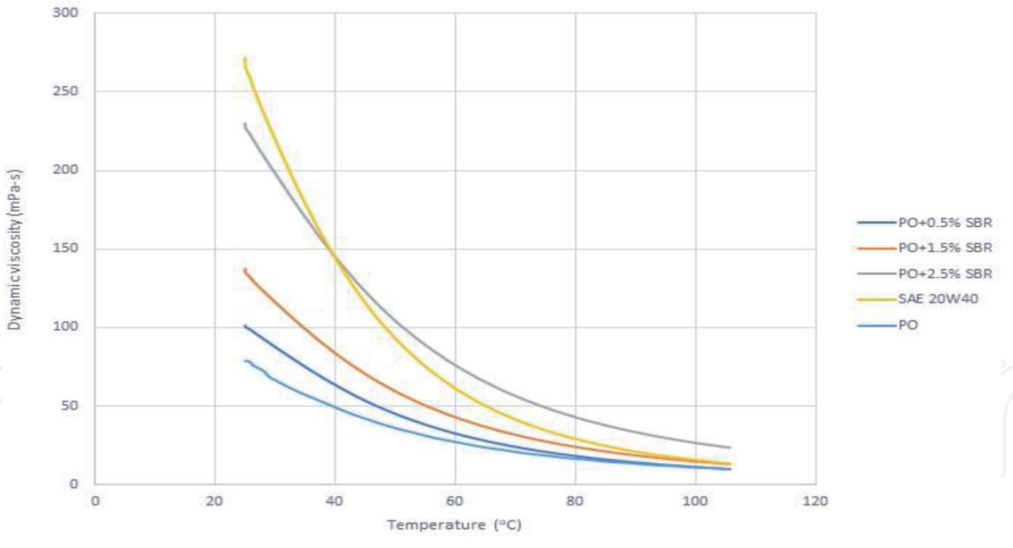

Figure 4.
Dynamic viscosity v/s temperature curve for PO with SBR compared against SAE20W40.

3.1.2 Analysis of oxidation stability

High oxidation stability of TBHQ is because of the longer alkyl chains present in it [23]. Oxidation stability of the oil samples are estimated by measuring the change in viscosity at 40°C after hot oil oxidation test (HOOT) from the fresh sample without subjected to HOOT [24]. Change in dynamic viscosity of neat PO, PO oil samples blended with different weight percentages of TBHQ and SAE20W40 at 40°C are calculated and plotted as shown in **Figure 5**. From the analysis of different oil samples blended with different weight percentages of TBHQ, PO + 2.5% TBHQ showed the least change in viscosity. It is observed that for the weight percentage of TBHQ beyond 2.5%, it is difficult to dissolve in PO. The change in viscosity at 40°C of PO + 2.5% TBHQ is found to be very lesser than neat PO comparable with that of SAE20W40. Thus PO + 2.5% TBHQ have excellent oxidation stability.

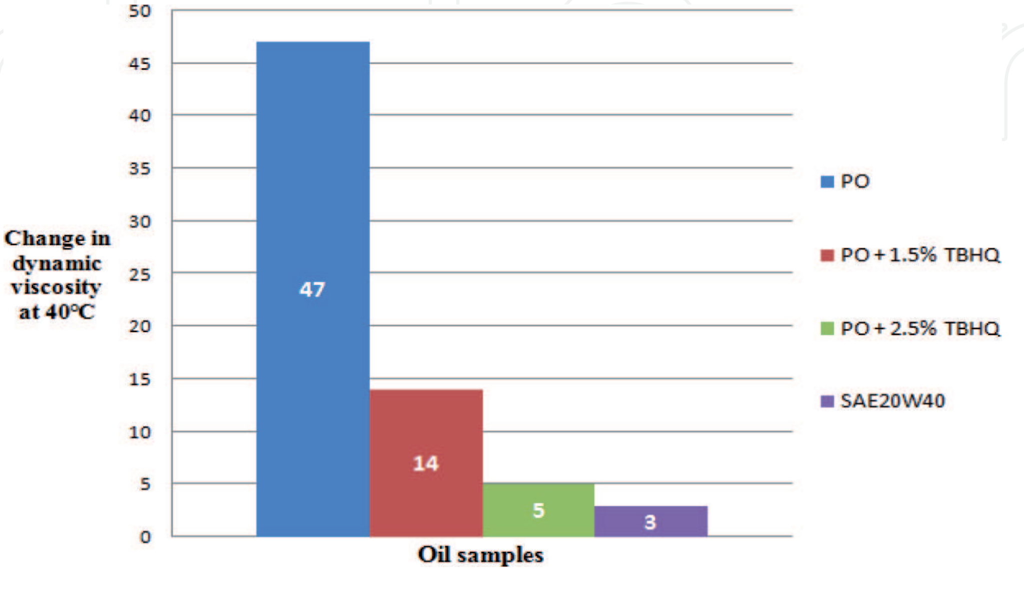

Figure 5.
Change in dynamic viscosities of oil samples at 40°C after HOOT.

3.1.3 Analysis of tribological properties

Tribological properties were evaluated with the help of a four ball tester equipment [25]. Coefficient of friction (COF) and wear scar diameter (WSD) are the parameters used to evaluate tribological properties. From **Figure 6**, it is noted that with the addition of nanoparticles, the WSD is reduced [26–29]. The WSD is found to be decreased by 18.31% with the blending of 0.85% weight percentage of SiO_2. The small size of SiO_2 makes improvement in the tribological properties by reducing friction and wear by rolling, mending, and protective film formation [30].

From **Figure 7**, it is noted that with the addition of nanoparticles, the COF is increasing for PO blends with SiO_2. However, in comparison with SAE20W40, the COF value of PO blends is considerably low.

3.2 Formulated oil results

Based on the tests conducted, the formulated oil (FO) is PO + 2.5 wt% SBR+ 2.5 wt% TBHQ +0.85 wt% SiO_2.

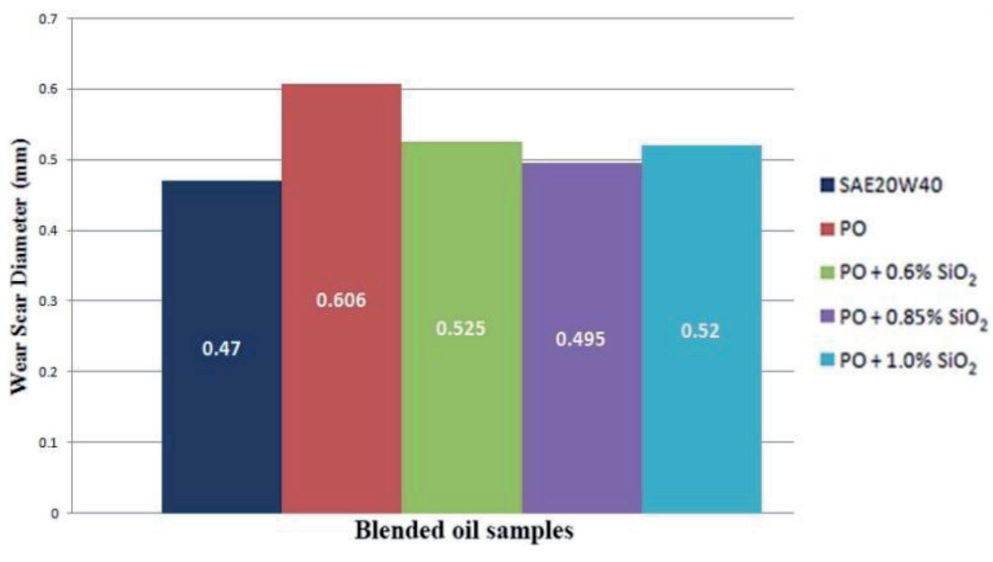

Figure 6.
Variation in WSD with the blending of different weight percentages of SiO_2 with PO.

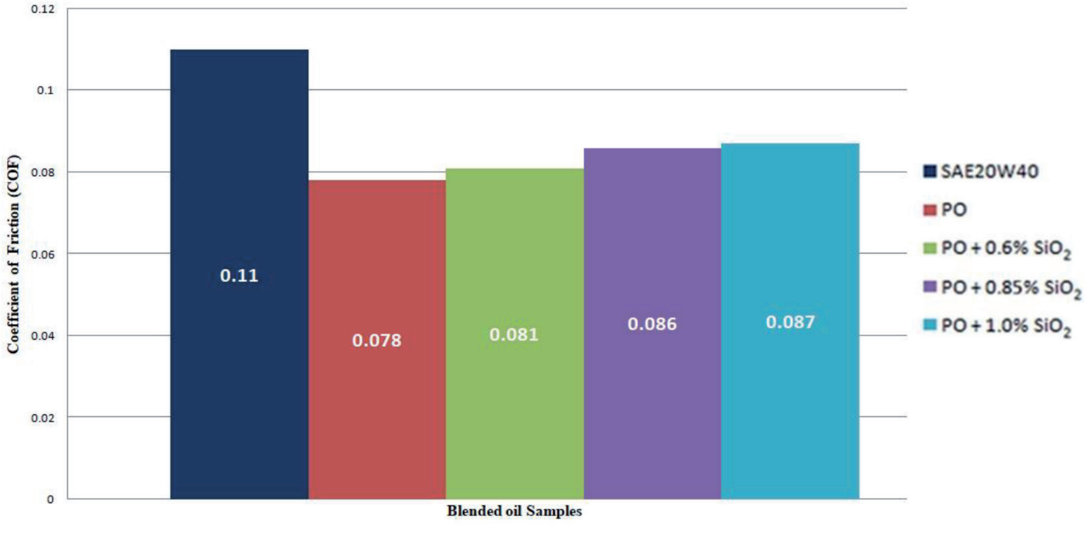

Figure 7.
Variation in COF with the blending of different weight percentages of SiO_2 with PO.

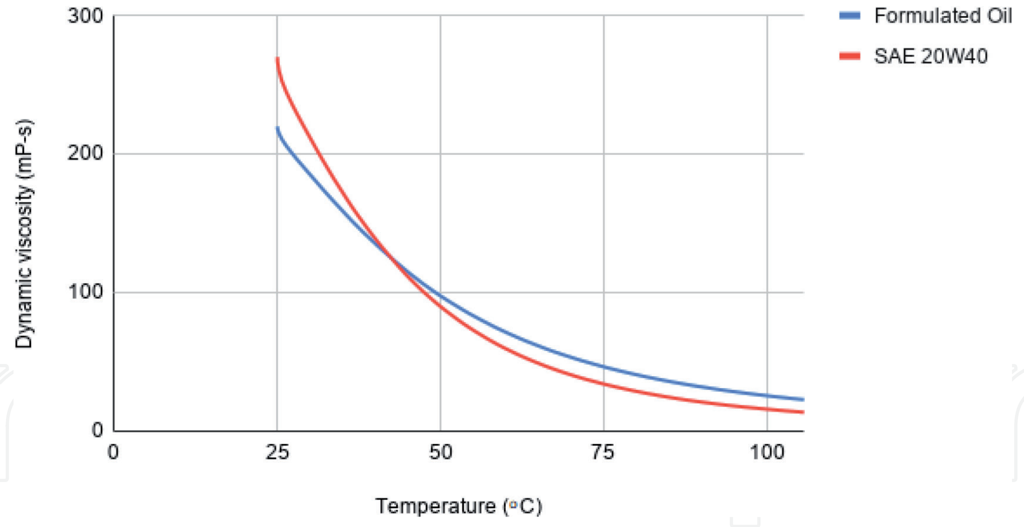

Figure 8.
Dynamic viscosity v/s temperature curve comparison of formulated oil and SAE 20W40.

Properties	FO	SAE20W40 [9]
COF	0.092	0.107
Wear scar diameter	0.48 mm	0.47 mm
Viscosity index	227.4	135.57
Increase in dynamic viscosity after HOOT at 40°C	5.4 cP	3 cP

Table 4.
Evaluated properties and results of the FO compared with SAE20W40.

Figure 8 represents the dynamic viscosity v/s temperature curve of the formulated oil and was found to be comparable with that of reference oil SAE 20W40.

The evaluated properties and results of FO compared with SAE20W40 is as shown in **Table 4**. The COF of FO is found to be comparatively lower than that of SAE20W40. This implies that the frictional effect produced by FO will be much lower than the latter. The viscosity index of the FO shows much improved results than that of SAE20W40. This ensures the use of FO at wide range of temperatures. The WSD of the FO is much closer to that of SAE20W40. Therefore, the FO has almost similar anti-wear effects as that of SAE20W40. Increase in dynamic viscosity after HOOT at 40°C indicates that the oxidation stability of the FO is superior that PO and is comparable with that of SAE20W40.

4. Conclusion

The project was aimed on formulating a bio-lubricant from a non-edible vegetable oil as base stock. In addition to non-edible nature, pongamia oil (PO) was chosen as the base oil due to its high oleic acid content. SBR, TBHQ and SiO_2 nanoparticle additives are added to PO for improving its viscosity, oxidation stability and antiwear properties respectively. The final base oil and additive combination of the formulated oil (FO) is PO + 2.5 wt% SBR + 2.5 wt% TBHQ +0.85 wt% SiO_2. Rheological studies show that the performance of the FO is identical to the dynamic viscosity trend of SAE20W40 and possess a superior viscosity index than SAE 20 W40. HOOT indicates that the oxidation stability of FO is much closer to that of SAE20W40. Tribological studies indicate that FO possess identical WSD and lower COF than SAE20W40.

Pongamia (milletia) is a genus of legume plant family. It is distributed in tropical and subtropical regions of the world. It is easily cultivable [31] and can grow in semi-arid conditions [32]. The cultivation of these trees has increased due to its recent interests in the field of bio-fuels and lubricants. This project is based on the enhancement of pongamia oil (PO) to a bio-lubricant, the cultivation of these trees will be promoted to a great extend thereby improving the agricultural sector.

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