Vol. 44, No. 4 (2022) 641-651, DOI: 10.24874/ti.1342.07.22.10

Tribology in Industry

www.tribology.rs

RESEARCH

A Preliminary Investigation of Physicochemical, Rheological and Tribological Properties of Bio-lubricant from Thevetia Peruviana Oil

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Keywords:

Thevetia Peruviana
Bio-lubricant
Four-ball tribo-tester
Physicochemical
Tribological

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Received: 29 July 2022
Revised: 3 September 2022
Accepted: 4 October 2022

A B S T R A C T

The present work is a comparative evaluation of physicochemical, rheological, thermal, and tribological properties of Thevetia Peruviana oil (TPO) as bio-lubricant. The tribological properties were investigated by a four-ball tribo-tester according to ASTM 4172-94 and ASTM D2783 standard. The corresponding results were compared with sunflower oil, jatropha oil, SAE 20W-40 and ISO VG 46. The acid value, pour point and flash point of TPO were found to be 1.383± 0.015 mg KOH, -4 ±0.231°C and 344±0.04°C respectively. The viscosity index of TPO is 235.59 with viscosities 31.76 mm²/s at 40°C and 7.89 mm²/s at 100°C. The oxidation onset temperature of TPO having a value of 282.20°C is comparable to SAE 20W-40 but higher than that of ISO VG 46. The results obtained from tribological test show that TPO has low coefficient of friction 0.0496±0.001 with wear scar diameter 0.462±0.001 mm. Despite the low pour point temperature, TPO are comparable to commercial lubricants, which prove its suitability as bio-lubricant base-stock based on physicochemical, thermal, tribological properties, and high viscosity index.

1. INTRODUCTION

Lubricants are substances utilized in all machines to allow a smooth movement between surfaces in contact with each other, minimizing friction and wear. Currently, approximately 90% of lubricants are petroleum-based or synthetic oil [1]. There is a global concern over the use of mineral oils as lubricants due to the depletion of fossil fuels, fluctuating crude oil prices and environmental hazards [2]. Global lubricant consumption in 2019 was predicted to be 45.5 million metric tonnes [3], necessitating the search for alternatives due to environmental toxicity and poor biodegradability concerns associated with mineral oils. Vegetable oils as bio-lubricants are biodegradable, non-toxic, and possess a higher flash point and provide better lubrication in comparison to mineral oils owing to the amphiphilic characteristic of the triglyceride of vegetable oils, which makes them a suitable option for petroleum-based lubricants [4]. Despite their advantages, bio-lubricants are still a long way from becoming fully developed industrial lubricants due to their high pour point temperature and limited thermo-oxidation stability [5].

Currently, different vegetable oil-based bio-lubricants including palm oil [6], canola oil [7], Cucurbita pepo L. [8], soybean oil [9], sunflower oil [10], coconut oil [11], olive oil [12], Pongamia pinnata oil [13], rapeseed oil [14], jatropha oil [15], rice bran oil [16], rubber seed oil [17], castor oil [18], sesame oil [19], Shorea Robusta (sal) oil [20], Moringa oleifera oil [21], etc. have been investigated. These studies revealed that the existence of polar groups and the long chains of fatty acids help in the formation of a monolayer film between the mating pairs, and therefore, vegetable oil provides effective boundary lubrication. The polarity of the fatty acid causes one end of its molecule (carboxyl group, -COOH) to be attracted to the metal surface, while the other end (alkyl group, -CH3) is repelled from the surface [22]. This process is known as physical adsorption (physisorption) and the van der Waals force or hydrogen bond plays a major role in the formation of a mono-molecular layer to minimise friction on the surface [23]. With regards to the coefficient of friction (CoF), the value of the CoF of vegetable oils for boundary lubrication is in the range of 0.001–0.2 [24]. These oils exhibit higher flash point temperatures, higher viscosity indices, and better anti-corrosion properties compared to mineral oils. Vegetable oils with a high degree of unsaturation have a lower pour point temperature but poor oxidation stability [5]. Reported studies revealed that vegetable oils such as olive oil [12], sunflower oil [10], rubber seed oil [14], soybean oil [9], jatropha oil [15], sesame oil [19], rapseed oil [14], etc., which contain oleic and linoleic acids as major fatty acid components, exhibit a pour point temperature between -3 and -29 °C. The main reason for the low pour point temperature is the bent structural configuration in oleic and linoleic acids, which prevents close packing of molecules during cooling [22].

Non-edible seed oils are preferred for bio-lubricant production to overcome corundum issues such as fuel versus food controversies, the environment, etc. [25]. India has many species of crops, both edible as well as non-edible. Thevetia Peruviana, more commonly known as karabi or yellow oleander, is a small evergreen plant belonging to the Apocynaceae family and is widespread in tropical and sub-tropical regions of America, the Asian and African continents. The plant does not require conventional agricultural lands and can be grown on barren lands. It is usually grown on roadsides. The plant fruits and seeds nearly throughout the year. The plant also produces seeds rich in oil (60–67%) [26]. The rate of seed yield per annum per hectare is 52.5 tonnes [27]. It has been reported that a hectare of wasteland can yield approximately 1750 l of oil [28].

Thevetia Peruviana oil (TPO) has potential for several applications. The high oil yield makes T. Peruviana suitable feedstock for biodiesel production. Deka and Basumatary [29] and Sut et al. [28] extracted oil and investigated the physicochemical properties, which have proven its potential as biodiesel feedstock. Recently, Yadav et al. [30] and Arun et al. [31] studied the engine performance and emission characteristics of TPO when compared to diesel. Yadav et al. [30] performed the engine tests of TPO blended with diesel at a ratio of 10, 20, and 30%. They reported a 40.6% reduction in hydrocarbon emissions and a 41.4% decrease in carbon monoxide (CO) emissions compared to diesel fuel in a 30% blend and a 20% blend, respectively. Jabar et al. [32] reported that TPO has good quality for cosmetics, soap, paint, and polyurethane production. Previous literature shows that TPO can be used as an alkyd resin, medicine, abortifacient, and astringent [33]. Rajbhar and Kumar [34] reported that TPO is used as a heart stimulant due to the presence of thevetin. In addition to that, it has been used in the treatment of congestive heart failure and malaria. TPO exhibits good anti-bacterial, anti-fungal and anti-termite properties, and hence it is used for surface coating [44]. Most of the previous literature is limited to the utilization of T. Peruviana as biofuel feedstock, raw material for industrial-scale resin, oleochemicals, and medicines.

The current work focuses on evaluating the physicochemical, rheological, and tribological properties of T. Peruviana oil (TPO) as well as comparing the results with an edible oil- sunflower oil (SO), a non-edible oil- jatropha oil (JO) and commercial lubricants (SAE 20W-40, ISO VG 46). No study has been found on the tribological characteristics of TPO. This work aims to utilize TPO as a sustainable alternative solution to petroleum-based lubricants as well as gear oil.

2. MATERIAL AND METHODS

2.1 Material

T. Peruviana seeds were collected from different locations in Guwahati, Assam. Sunflower oil (SO), Jatropha oil (JO) and commercial lubricants were purchased from local vendors in Guwahati, Assam.

Oil was extracted from the grinded kernels using n-hexane as a solvent by the Soxhlet extraction method. The solvent was removed with the help of a rotary vacuum evaporator (IKA, RV-3V, Germany). Anhydrous sodium sulphate was used to remove a trace amount of water (if any) from the oil. Oil sample was weighed to estimate the oil yield using Eq. 1:

$$\text{Yield of TPO} = \left(\frac{massofTPO}{massofthesample}\right) x\ 100\% \quad (1)$$

2.2 Fatty acid profile

GC–MS analysis of the oil samples was carried out to determine the fatty acid composition of oils using Perkin Elmer Clarus680 GC/600C MS along with the peak identification using a mass spectral library (NIST14 version 2.4).

2.3 Physicochemical and thermal properties

The acid value, flash point and pour point temperatures of oils were determined according to ASTM D974-14 [36], ASTM D92 [15] and ASTM D97-12 [36], respectively. Density was measured using a pycnometer (ASTM 1298). The tests were performed three times and the results were mentioned as the mean ± standard deviation.

2.4 Rheological characteristics

The viscosity of TPO was measured at temperatures of 40°C and 100°C with shear rate of 100-1000 s^{-1} using a rheometer (Make-Anton Paar, model MCR-102). The viscosity index was calculated according to ASTM D2270-10 [37].

2.5 Thermal-oxidation stability analysis

TPO and other oils were heated at 10˚C/min in a temperature range of 25–600˚C under both N_2 and O_2 atmospheres in a thermogravimetric analyzer (Make-Mettler Toledo, model TGA2) to evaluate thermal-oxidation stability.

2.6 Analysis of Tribological Properties

Figure 1 shows the four-ball tribo-tester. A four-ball tribo-tester was used to investigate the tribological properties of oils. Steel balls (AISI 52100) with a diameter of 12.7 mm and 64 HRc were used. The anti-wear test and extreme pressure test were carried out according to ASTM D 4172-94 and ASTM D2783 [38]. Test conditions are presented in Table 1. The data acquisition system measured the coefficient of friction (COF).

The last non-seizure load (LNSL) and the initial seizure load (ISL) were determined. Weld load (WL), at which all four balls were welded, was also determined. The hertz line and compensation line were drawn for the lubricant samples. The wear morphology was studied using an optical microscope (Make-Metatech, Mode-Metagraph A).

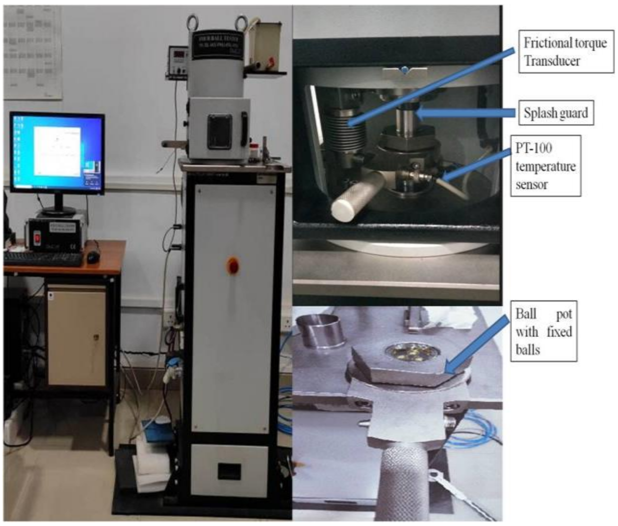


Fig. 1. The four-ball tribo-tester.

Table1. Test conditions of four-ball tester.

Parameters	ASTM D4172	ASTM D2783
Oil Temperature	75°C	Ambient temperature (28±4°C)
Test duration (s)	3600	10
Spindle speed (rpm)	1200	1760
Load (N)	392	Starting from load 392N to weld load

2.7 Uncertainty analysis

Uncertainty evaluation is carried out by repeating each experiment three times and calculating the standard deviation of the measured uncertainty values. Uncertainties of the measured physicochemical (density, acid value), thermal properties (flash point and pour point), coefficient of friction, and wear scar diameter were determined using Eq. 2 [36]:

Overall uncertainty of the experiment =

$$\sqrt{u(D)^2 + u(A)^2 + u(PP)^2 + u(FP)^2 + u(CoF)^2 + u(WSD)^2} \quad (2)$$

The overall uncertainty value was $\pm$ 3.871%, so the accuracy of the results is accepted to be within $\pm$ 5%. The uncertainty analysis is presented in Table 2.

Table 2. Uncertainty analysis of different properties.

Parameter measured/ Abbreviation	Equipment used	Uncertainty	% uncertainty
Density (g/cm³) [$\boldsymbol{u(D)}$]	Pycnometer with thermometer AS-R- Analytical Balance	$\pm$0.01ml and $\pm$0.2°C (thermometer) $\pm$0.0001g	0.876
Acid value (mg KOH/g) [$\boldsymbol{u(A)}$]	AS-R- Analytical Balance 1l Volumetric flask 10 ml burette	$\pm$0.0001g $\pm$0.1ml $\pm$0.05ml	1.493
Pour point (°C) [$\boldsymbol{u(PP)}$]	Cloud and Pour point apparatus	$\pm$0.1°C	2.833
Flash point (°C)[$\boldsymbol{u(FP)}$]	Cleveland open cup tester	$\pm$0.5°C	0.154
Coefficient of friction [*u (CoF)*]	Fourball tribo-tester	0.01	1.918
Wear scar diameter [*u(WSD)*]	An optical microscope	0.001mm	0.512

Table 3. Fatty acid composition of TPO and other vegetable oils.

Components	**Fraction (%)**		
	TPO	**Sunflower oil (SO)**	**Jatropha oil (JO)**
Palmitic acid (C16:0)	15.455	6.751	14.138
Stearic acid (C18:0)	6.751	3.671	6.401
Oleic acid (C18:1)	45.103	28.781	42.187
Linoleic acid (C18:2)	31.6	57.412	33.054
Linolenic acid (C18:3)	0.607	--	0.261
Arachidic acid (C20:0)	--	0.377	--
Saturated fatty acid (SFA)	22.206	10.799	20.539
Mono-unsaturated fatty acid (MUFA)	45.103	28.781	42.187
Poly-unsaturated fatty acid (PUFA)	32.607	57.412	33.315

3. RESULTS AND DISCUSSION

3.1 Fatty acid profile

Table 3 shows the fatty acid compositions of TPO, SO, and JO. TPO consists of 22.206% SFA, 45.103% MUFA, and 32.607% PUFA. Major portions of SFA, MUFA, and PUFA were contributed by palmitic acid (15.455%), oleic acid (45.103%), and linoleic acid (31.6%), respectively. The low oxidation stability of vegetable oils is mainly due to the double bond (C=C) in fatty acids [5]. In general, bio-lubricants having a high percentage of PUFA are susceptible to rapid oxidation due to the reaction of double bonds present in alkyl chains with oxygen to form free radicals which degrade to form peroxides and acids [5, 39]. However, SFA influences the low-temperature properties of vegetable oils due to the rapid self-stacking of SFA chains in comparison to the unsaturated fatty acid, which results in the formation of a macro-crystalline structure [5, 39]. Reeves et al. [40] concluded that the higher the oleic acid in vegetable oils, the better the oxidative stability and tribological properties but lower the pour point temperature [40]. The geometrically linear structure of stearic acid provides strong protective layers, reducing friction and wear [41]. Murakami and Sakamoto showed that stearic and oleic acids reduced friction up to a temperature of 110°C, but linoleic and linolenic acids maintained low friction behavior even at higher temperatures [42]. The presence of high unsaturated fatty acids especially PUFA in oil, improves low temperature properties since the presence of bent configuration in them prevents close packing of the molecules during cooling [43]. The stearic acid and oleic acid contents in TPO (6.751% and 45.103%) are higher than in SO (3.671% and 28.781%) and JO (6.401% and 42.187%). However, the linoleic acid content of TPO is lower than that of SO. It can be concluded that TPO will exhibit better thermal-oxidation stability and will form a stable lubricating film based on its fatty acid composition.

3.2 Physicochemical and thermal properties of TPO

The oil yield of Thevetia Peruviana seed was found to be 55.34%. Table 4 represents the physicochemical and thermal properties of TPO and other oils. The density of TPO is 0.913 ± 0.001g/cm^3, which is in the range of other vegetable oils (0.827-0.934 g/cm^3). The acid value of TPO was found to be 1.383 ± 0.015 mg KOH/g. Although the acid value of TPO is lower than SO (4.11 ± 0.003 mg KOH/g) and JO (5 ± 0.002 mg KOH/g) and higher than SAE 20W-40 (0.84 ± 0.07 mg KOH/g), it is lower than the recommended value of 10 mg KOH/g for good quality lubricant [44]. In comparison to SO, JO, SAE 20W-40, and ISO VG46, the pour point of TPO is low, which is due to the difference in oleic and linoleic acid composition in vegetable oils and the presence of additives in commercial oils. The flash point of TPO was found to be 344°C, higher than that of SO, JO, SAE 20W-40 and ISO VG46, which indicates its better resistance to fire hazards.

Table 4. Physicochemical and thermal properties of TPO, commercial oil and other vegetable oils.

Oils	Density (g/cm^3) at 20^{0}C	Acid value (mg KOH/g)	Pour point (°C)	Flash point (°C)
TPO	0.913±0.001	1.383±0.015	-4±0.231	344±0.04
SO	0.919± 0.002	4.11±0.003	-12±0.143	332±0.07
JO	0.920± 0.001	5±0.002	-1±0.119	264±0.05
SAE 20W-40	0.87± 0.001	0.84± 0.07	-20±0.07	204±0.08
ISO VG 46	0.865± 0.001	0.56± 0.02	-20±0.03	232±0.02

3.3 Rheological properties of TPO

Figure 2 shows the dynamic viscosity of TPO and other oils measured at temperatures of 40°C and 100°C and the variation of shear stress with respect to shear rate. From the figures, the Newtonian behaviour of TPO is ensured throughout the shear rates. TPO exhibits a viscosity of 31.76 mm^2/s at 40°C and 7.89 mm^2/s at 100°C, as presented in Table 5. The viscosity of TPO at 100°C is in the range of 5 -15 mm^2/s, as defined by an industrial lubricant standard [45]. The viscosity of TPO is relatively lower than SAE 20W-40 and ISO VG46 but comparable to SO and JO. The higher viscosity index (VI) of TPO in comparison to SO, JO, SAE 20W-40 and ISO VG46 indicates more stable lubricating films across a wider temperature range.

Table 5. Rheological properties of TPO, commercial oil and other vegetable oils.

Oil type	Kinematic viscosity (mm^2/s) at 40°C	Kinematic viscosity (mm^2/s) at 100°C	Viscosity Index
TPO	31.76	7.89	235.59
SO	27.22	6.08	181.22
JO	33.68	6.78	164.8
SAE 20W-40	121.88	14.2	116
ISO VG46	46.12	6.67	100.818

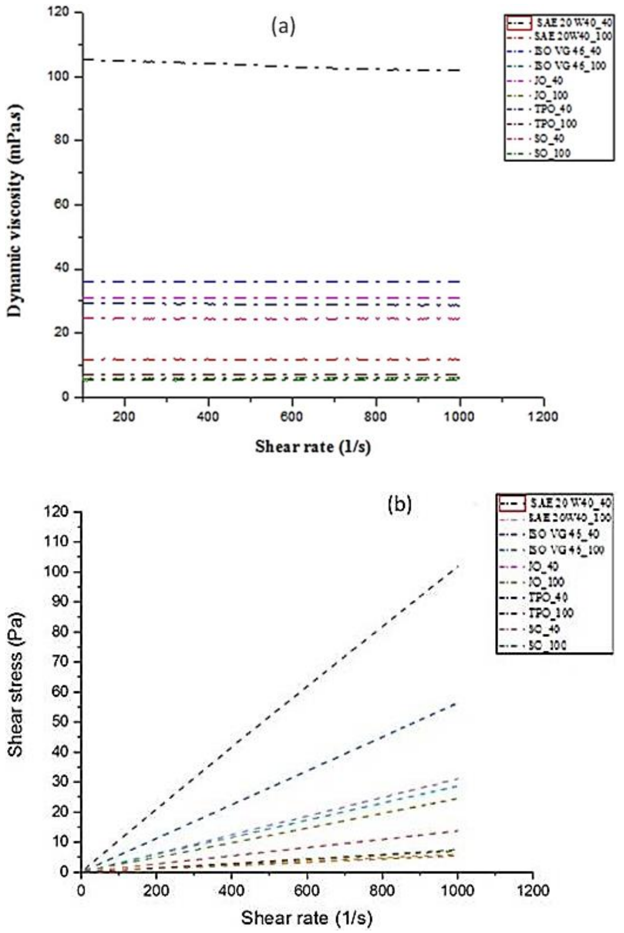


Fig. 2. (a) Dynamic viscosity vs. shear rate (b) Shear stress vs. Shear rate.

3.4 Thermal-oxidation stability analysis

The thermogravimetric curves of the oils in nitrogen and oxygen environments are shown in Figure 3. The thermal and oxidation onset temperatures of all oils are presented in Table 6. From the table, it can be found that the thermal and oxidation onset temperatures of TPO are 359.33°C and 277.37°C respectively, which is comparable to SAE 20W-40 and higher than ISO VG46. In an oxygen atmosphere, degradation occurs in three stages followed by the decomposition of PUFA, MUFA, and SFA. The difference in thermal behaviour of vegetable oils TPO, SO, and JO is mainly due to the presence of different percentages of MUFA and SFA. The oxidation rates of various fatty acids are as stearic : oleic : linoleic : linolenic = 1 : 10 : 100 : 200 [44]. Reeves et al. [40] reported that the higher the content of fatty acids having C18 atoms, the higher the oxidation onset temperature. In comparison to SO and JO, TPO exhibits better thermal-oxidation stability due to the presence of a higher amount of stearic and oleic acid.

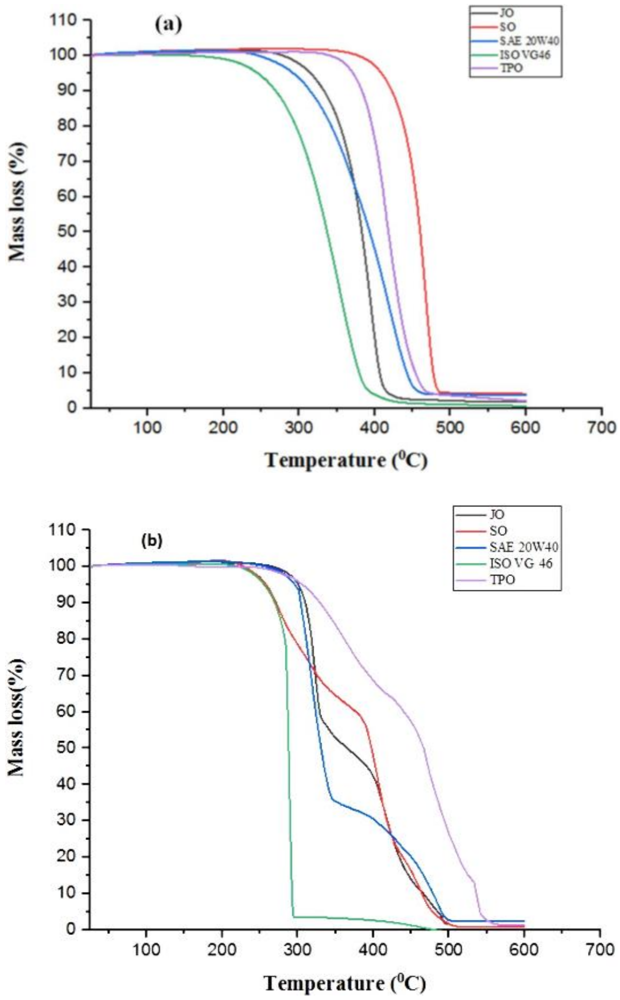


Fig. 3. TG profiles of TPO and other oils in (a) N_2 atmosphere and (b) O_2 atmosphere.

Table 6. Onset temperature in thermal degradation of TPO and other oils under nitrogen and oxygen atmosphere.

Oil type	Onset temperature (°C) under N_2	Oxidative onset temperature (°C)
TPO	359.33	282.20
SO	395.52	237.96
JO	298.27	282.16
SAE 20W-40	270.17	277.37
ISO VG46	219.33	241.33

3.5 Tribological studies

Figure 4 demonstrates the variation of the coefficient of friction (CoF) during the test. From Figure 4, it can be seen that the CoF was unsteady for the first few seconds due to the initial friction between the bottom three stationary balls and the top rotating ball. After 200 s, the variation of the CoF of TPO became stable and lower than other tested oils. The variation of the CoF of TPO and SAE 20W-40 is similar to that of SAE 20W-40 and JO.

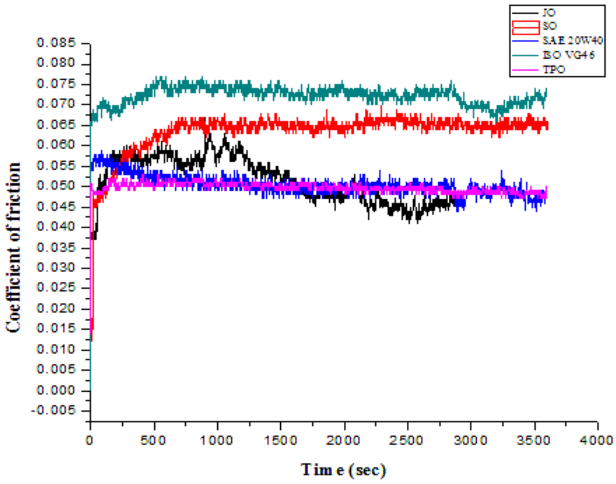


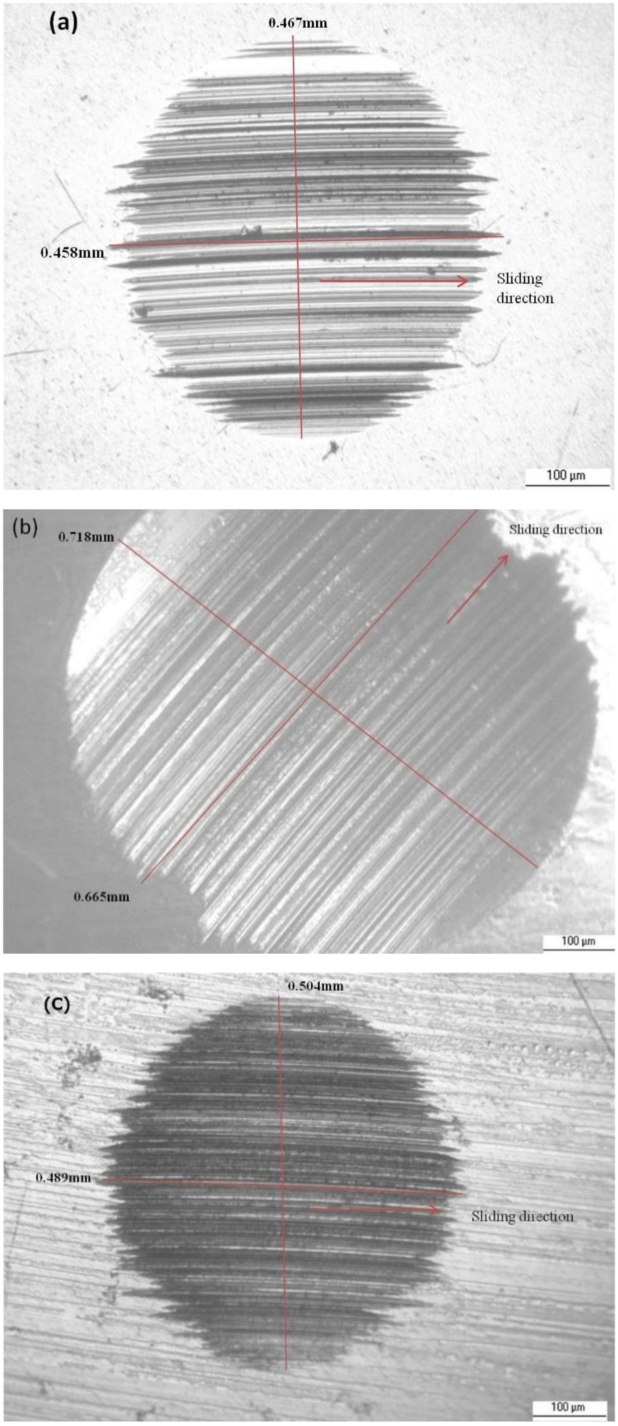
Fig. 4. Coefficient of friction of TPO and other oils.

Table 7 shows the CoF and wear scar diameter (WSD) of all tested oils. WSD of TPO and other oils are shown in figure 5. The WSD of TPO was less than that of SO and JO but 27.9% and 14.5% higher than SAE 20W-40 and ISO VG46, respectively. The presence of polar fatty acids in vegetable oils helps in the formation of a stable boundary layer as ester groups attach to the metal surface. Siniawski et al. [46] concluded that stearic acid plays a significant role in reducing the friction and wear rate. They also mentioned that the presence of unsaturated fatty acids, particularly linoleic acid, prevents the close packing of molecular chains due to the double bonds in its structure, which results in the formation of weaker protective layers. SO has a higher CoF and WSD value than TPO and JO due to its higher linoleic acid content of 57.412%. Due to the presence of stearic acid and oleic acid at a significantly good amount, TPO formed a relatively stable surface protection film. These results are in agreement with previous studies [46, 47] where vegetable oils with high stearic and oleic acid had low CoF and wear due to the development of an effective monolayer.

Table 7. Coefficient of friction and wear scar diameter of TPO and other oils.

Oil type	Coefficient of friction	Wear scar diameter (mm)
TPO	0.0496±0.001	0.462±0.001
SO	0.0634±0.003	0.692±0.003
JO	0.0509±0.002	0.496±0.001
SAE20W-40	0.0506 ±0.004	0.333±0.002
ISO VG46	0.0722±0.005	0.395±0.004

Figure 5 shows the wear morphology. As shown in figure 5, wear patterns in the case of TPO, SO, JO and ISO VG46 are in the form of regular grooves parallel to the sliding direction, whereas the wear patterns in the case of SAE 20W-40 are in the form of spall development owing to plastic deformation. Deeper grooves and shallow grooves in the case of TPO, SO, and JO can be visualized, which are the characteristics of abrasive wear, as concluded by Suresha et al. [47]. The wear scar of ISO VG46 is relatively smoother in comparison to other tested oils.



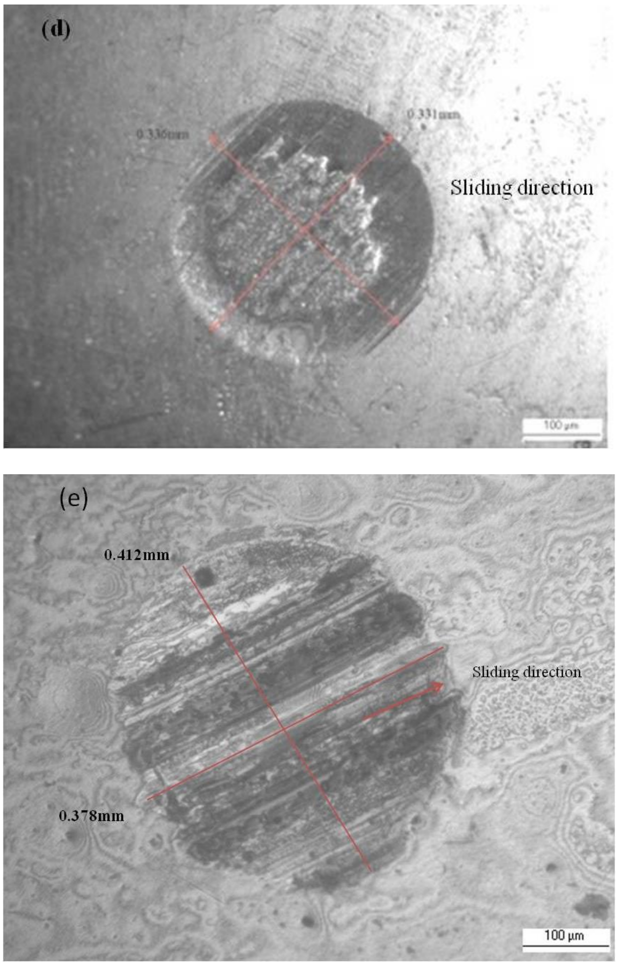
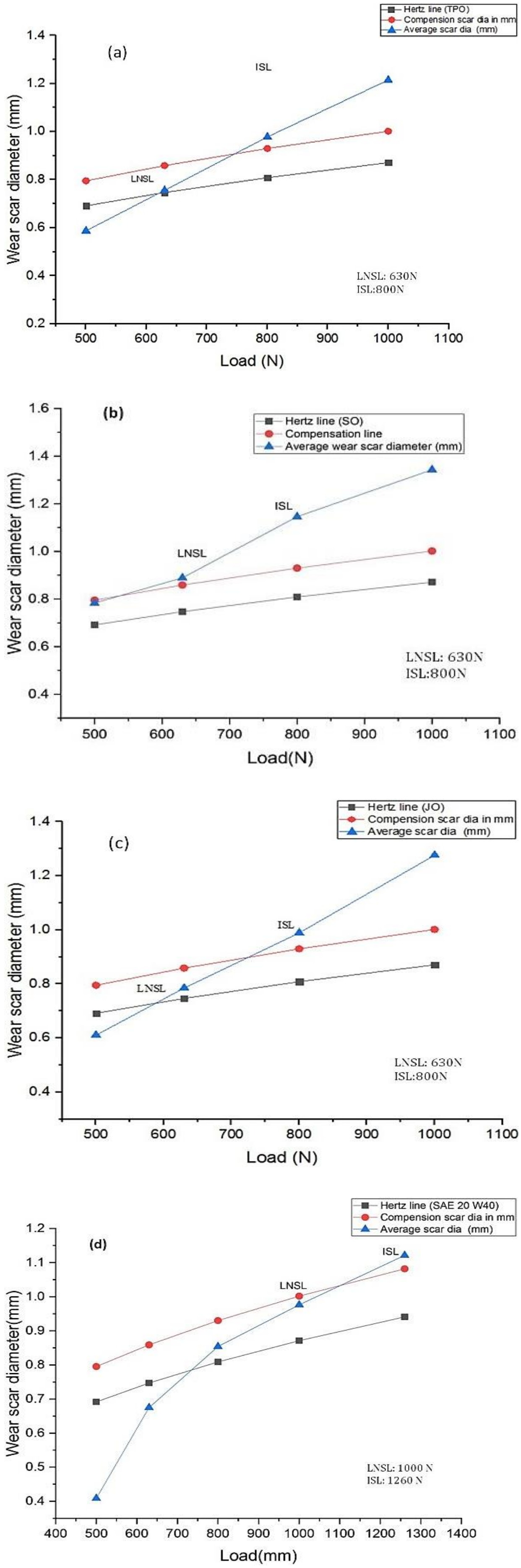


Fig. 5. Wear morphology of (a) TPO, (b) SO, (c) JO, (d) SAE 20W-40, and (e) ISO VG46 .

Figure 6 represents the results of extreme pressure tests of oils. LNSL, ISL, and WL of oils are presented in Table 8. The weld load of TPO is the same as SO, JO, and ISO VG46 but lower than that of SAE 20W-40. The region between LNSL and ISL is termed as the incipient seizure load region [48]. This region is characterized by a sudden increase in WSD and CoF due to the temporal break-down of the lubricating film [48]. The low WSD of TPO in this region in comparison to SO and JO indicates better resistance to wear. The presence of additives delayed the incipient seizure of SAE 20W-40 (1000N and 1260N) and ISO VG46 (800N and 1000N).

Table 8. EP test results of TPO and other oils.

Oil	LNSL (N)	ISL (N)	WL (N)
TPO	630	800	1260
SO	630	800	1260
JO	630	800	1260
SAE 20W-40	1000	1260	1600
ISO VG46	800	1000	1260



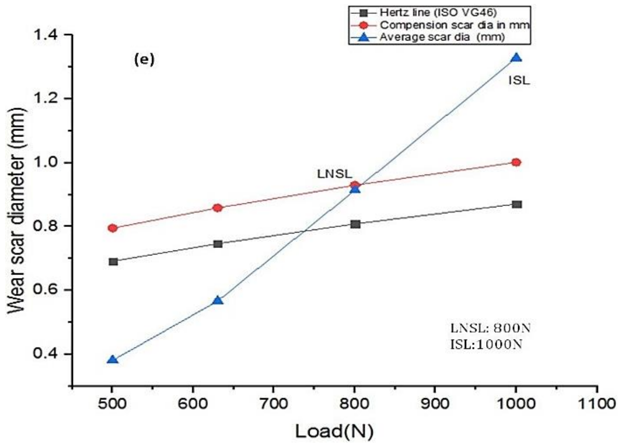


Fig. 6. Results of extreme pressure test of (a) TPO, (b) SO, (c) JO, (d) SAE 20W-40, and (e) ISO VG 46.

4. CONCLUSION

The following conclusions have been drawn from the present study:

1. The anti-wear properties of TPO are comparable to those of SAE 20W-40 and ISO VG46. The results of extreme pressure testing show that the LNSL of TPO is less than that of both ISO VG46 and SAE 20W-40. The weld load of TPO is the same as ISO VG46 but lower than that of SAE 20W-40.
2. TPO's thermal-oxidation stability is comparable to SAE 20W-40 and ISO VG46.
3. Although the viscosity of TPO is less than that of SAE 20W-40, it is better than ISO VG46. TPO has a higher viscosity index (235.59) which indicates the formation of a stable lubrication layer over a wide range of temperatures.
4. TPO has a higher flash point temperature but its pour point is lower than SAE 20W-40 and ISO VG46, which can be improved with the addition of additives, and pour point depressant.

From the results of the present study, it can be concluded that low coefficient of friction and wear scar diameter make TPO a suitable bio-lubricant base stock.

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