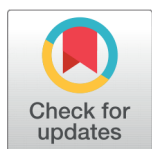


Tribological Analysis of Utilizing Halide Ionic Liquid as an Additive to the Bio-Based oils



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The widespread usage of petroleum-based lubricants has caused environmental and operator health problems; therefore, a substitute for these lubricants must be investigated. Bio-based lubricants give superior lubricity as compared to petroleum-based lubricants. Modifying a bio-based lubricant's tribological properties is crucial for improving its lubricating efficacy. The efficacy of an ecologically friendly halide ionic liquid as lubricant additive in a bio-based lubricant is investigated in this research. 2 hydroxyethyl trimethylammonium chloride [C₅H₁₄CINO] was chosen as an addition to improve the tribological performance of bio-lubricants based on coconut and cottonseed oils. Three different mass concentrations of the additive (0.5 wt.%, 1 wt.%, and 1.5 wt.%) were added to the bio-based lubricants. Pin-on-disc tribology studies on AISI 1030 steel/steel contact was performed. The steel sample was then examined further with various techniques such as scanning electron microscopy (SEM) and stylus to identify the lubricant's lubricating mechanism. The results showed that the IL blended into the bio-based lubricant had a positive synergistic impact on lubrication performance of both bio-based oils. Coconut oil + [C₅H₁₄CINO] 1 IL 1.5 wt.% decreases friction coefficient by 40%, wear volume by 45% and improves bio-based oil kinematics. [C₅H₁₄CINO]IL 1.5wt.% increases the friction and wear volume of steel surfaces. The results of the complete experiment demonstrate that ionic liquids can change the tribological behavior of bio-based lubricants, and that the formed IL-based samples are safe for the environment and the machine operator's health.

Keywords: Ionic Liquid, Coefficient of Friction, Lubrication, Conventional Mineral Based Lubricant, BioBased Oils

INTRODUCTION

In order to maintain the proper operation of machinery and equipment with moving parts, lubricants are used to reduce the friction and wear of contacting surfaces. Depending on the use, lubricants can be solids (such as graphene, graphite, molybdenum, Teflon, and tungsten disulfide) or semi-solids (such as

grease) and liquids (such as water or oil)^{8,32,37}. Fluid lubrication is utilized in mild working circumstances because of their long service life, less mechanical noise, and high heat conductivity. Mixtures with additives or pure mineral oils are the conventional commercial fluid lubricants. In addition to lowering wear and friction, additives are used to improve viscosity index, viscosity,

corrosion, and oxidation resistance^{15,19}. Our reliance on petroleum-based lubricants is increasingly under the limelight, as environmental awareness, and concerns about environmental has been grown in the recent years. Further reliance on these lubricants, which are poisonous, non-renewable, and damaging to the ecosystem may have unforeseeable effects^{5,25}. Furthermore, the health of machine operators and the environment are negatively impacted by the toxic emissions from mineral oils. Skin contact with petroleum-based oils caused 80% of all intermittent infections among machine workers^{12,26,28,36}.

The need to find a suitable replacement for petroleum-based lubricants without compromising tribological performance is therefore paramount. Bio-based lubricants provide superior lubricity, shear resistance, a high viscosity index and a high flash point as compared to mineral oils. Bio-based lubricants are created using organic materials and animal fats including cottonseed, soybean, mustard, sunflower and other edible and non-edible plants^{6,30,35}. Bio-based oils have a stronger polarity than mineral oils because they contain triglyceride molecules. The attraction to metallic surfaces increases with increasing polarity. Vegetable oils and their derivatives display remarkable tribological performance because of this distinctive property, making them suitable candidate for lubricating applications^{1,4,27}. All lubrication regimes, with the exception of elasto hydrodynamic lubrication, are appropriate for bio-based lubricants since they are amphiphilic²³. The reported results shows that bio-based lubricants are more appropriate for tribo-pairs that function in the boundary lubrication regime³⁸. To increase the anti-wear and anti-friction qualities of these bio-based lubricants, various additives are added with them. The literature indicates that the physicochemical properties of the lubricating base oils are

improved when ionic liquids are used as additives, in addition to their intrinsic tribological performance. Cations and anions make up molten salts known as ionic liquids (ILs) at room temperature^{14,39–41}.

Ionic liquids (ILs) have emerged as a viable solution for traditional additives in recent years due to their exceptional tribological capabilities, decreased hazardous emissions, ash free nature and environmentally favorable footprint^{43,44}. ILs adhere tightly to metal surfaces, preventing wear and protecting interacting surfaces from carbon deposits and soot⁴². Liu et al. firstly investigated the suitability of ILs for tribological applications and resulted that, the ILs exhibits excellent friction-reduction, antiwear properties, both in air and vacuum, as compared with phosphazene (X-1P) and perfluoropolyether (PFPE)⁴⁵. ILs have been examined as lubricants and lubricant additives in the past^{46,47,9}, and it is clear from the published data that ILs are capable of producing a reducing the effects of corrosion, strong anti-wear coating, and withstanding heavy loads³. Polyethylene glycol (PEG), Polyalphaolefin (PAO4), palm oil-based trimethylolpropane ester (TMP) and many more motor oils are among the most regularly used base lubricants employing ILs as additive²⁰. Lglesias eand Jiménez group conducted studies between 2004 and 2006, and resulted that ILs as a room-temperature lubricant additives performed well in reducing the wear and friction loss^{21,48}. However, in 2012, with ILs soluble synthesis in non-polar lubricant oils²⁰, the study of lubrication with IL additives became a popular research topic.

Many publications have been published since then, but the advances in research in this topic have only been covered in three review articles, in 2017 the first one was published⁴⁹ and in 2020 the other two was published^{7,31}. The first two references investigated the chemical structure cor-

relations and other physicochemical aspects of IL additives with their tribological performance and the role of tribo-films in lubrication, while the latter⁷ addressed the usage of ILs as additives in bio-lubricants. Imidazolium-based ionic liquids have most likely been the most widely used additions since the initial studies in 2004–2009²¹. The Liu group investigated the effect of adding imidazolium-based ionic liquids to poly urea grease (PUG) and polyethylene glycol (PEG) on the lubrication of steel-copper and steel/steel pairs at ambient and high temperatures at the beginning of the current decade. They discovered significant reductions in wear and friction, which they attributed to the creation of hydrogen bond⁵⁰.

ILs produces low COF values than bio-based and mineral-based oils, it was found when the tribological performance of (C₁₀mimTf₂N) was compared to that of other bio-based oils³⁴. Tribological investigation of different concentrations (0–100) wt.% of (C₁₀mimTf₂N) utilized as an addition in avocado oil. Investigation was performed via a pin-on-disk setup with an applied load of 10N, a velocity of 36mm/s and an ambient temperature. The COF value decreased significantly as ionic liquid concentration increased. In comparison to pure avocado oil, pure ionic liquid reduce friction by 67.19%. A similar pattern was observed in wear behavior. The greatest reduction in wear volume (59.25%) was seen when pure ionic liquid is used as lubricant¹¹. Gonzalez et al. found that ILs and mixtures containing zinc dialkyldithiophosphate (ZDDP) both significantly reduced COF when compared to plain PAO4. However, larger ZDDP concentrations resulted in higher COF values, but higher ILs concentrations show lower COF values. When compared to the PAO4, the mixtures in the ILs showed a significant reduction in wear. With neat PAO4, plastic deformation and adhesive wear scars were visible, while samples that had

been lubricated with ILs based lubricants showed signs of abrasive wear¹⁶. Blanco et al. also studied the tribological behavior of [P₆₆₆₁₄] [NTf₂] at concentrations of different concentration (0.5, 1, 1.5 wt.%) in Priolube oil. Tribological testing was conducted at room temperature for 30 minutes with an 80 N load. Confocal microscopy was used to locate wear scars. XPS/SEM and engineering data system were used to inspect the worn surface. "The results showed that the base oil and the mixtures performed similarly in terms of friction, but only the 1 wt.% sample had a somewhat lower wear volume than the base oil. SEM scans revealed that all work pieces examined had similar wear mechanisms and wear track width (707-796 μm)²².

Wang et al., used an oscillating reciprocating tribo-tester to evaluate the tribological behavior of glycerol, tributyl phosphate and IL-alkylimidazoliumdialkyl phosphates (PBE) at 25 °C and 100 °C. To investigate COF and wear volume, experiments were carried out utilizing Si₃N₄(Ball)-Ti₃SiC₂ (disc) contacts. The results demonstrated that alkylimidazoliumdialkyl phosphates ILs were effective in minimizing wear and friction for tribo contacts and their efficacy was superior to glycerol and tributyl phosphate. The findings also show that the exceptional tribological endurance of alkylimidazoliumdialkyl phosphates was mostly attributed to its superior load-carrying capability and the production of protective tribo-films comprised of TiO₂, titanium phosphate, amines and NO_x by tribo-chemical processes². Ferri²⁴ investigated the tribological properties of conventional lubricant and another coolant using ionic liquid (5%) as additive to both coolant and oil. It was discovered that both coolant and oil with ILs performed better in terms of wear reduction. Ionic liquid-based oil and coolant reduced wear by 28% and 8% respectively. However, the same conclusion could not be drawn for the COF. During the

ILs circumstances, the wear traces were similarly reduced, with much less surface degradation²⁴.

In a study conducted by Duan et al.,⁵¹ four oil soluble ionic liquids were utilized as additives in polyalphaolefin PAO (base oil) used for lubricating a titanium disc surface sliding against a steel ball (AISI 52100). The two phosphates ILs outperformed the ZDDP and were found to improve the tribological characteristics and frictional behavior between the contacting surfaces. Although zinc dialkyldithiophosphate produced more material loss but ILs had excellent wear resistance, indicating high chemical-material compatibility. Otero et al.¹⁸ combined two different cations [C₄C₁C₁Im]⁺ or [P₆₆₆₁₄]⁺ with the same anion [(C₂F₅)₃PF₃]⁻ to formulate a lubricant. In terms of coefficient of friction and wear rate, IL [P₆₆₆₁₄] (C₂F₅)₃PF₃] outperformed [C₄C₁C₁Im] (C₂F₅)₃PF₃]. IL [P₆₆₆₁₄] (C₂F₅)₃PF₃] also revealed smooth and fine wear tracks. However, perfluoropolyether had a higher COF and wear rate¹⁸. Sani et al.²⁹ investigated the tribological characteristics of phosphonium-based ionic liquids as an addition to modified palmolein trimethylolpropane ester utilizing a four-ball tribotester. The addition of a minute amount of PIL (1 wt.%) improves the MRPO's tribological properties²⁹. A tribological study using steel-to-steel contact was conducted on pure neem oil and two ionic liquids, such as tetra-butyl-ammonium bromide (C₁₆H₃₆BrN) and tetra-butyl-phosphonium bromide (C₁₆H₃₆BrN). Neem oil coupled with this IL has significantly reduces wear and friction at 0.5 wt. %¹⁰. ILs containing aliphatic acid with phosphate units, such as SAPN, PAPN, LAPN, and OAPN, were tested as lubricant additives in 5CST and rick simpson oil. PAPN surpassed SAPN, OAPN, and LAPN in four-ball tribological tests in terms of frictional and anti-wear properties⁵². Tri-[bis(2Hydroxyethylammonium)] citrate (Dci), an environmentally

friendly protonic ionic liquid, was investigated as an addition in bio-based oil utilizing reciprocating ball-on-flat disc tribo-testers which is used as lubricant for machining of titanium ceramic contact at three unique frequencies of 3Hz, 4Hz, and 5 Hz. At a frequency of 5 Hz, the results show a 23% reduction in wear and a 50% reduction in friction³³. Different ILs are employed as lubricants in manufacturing processes but some of these are unfriendly to the environment and could harm human cells, aquatic ecosystems, and terrestrial surroundings. The majority of research on harmful ILs and their interaction with synthetic oil is done in literature but there is little work on the tribological study of commercially available eco-friendly ionic liquid as an addition to the bio-based lubricants.

The main focus of this research is to use 2 hydroxyethyl trimethylammonium chloride as additive into the bio-based oils of coconut and cottonseed which not only improve the tribological character of bio-based oil but also safe for the health of machine operator. 2 hydroxyethyl trimethylammonium chloride which is an environment friendly ionic liquid is used as lubricant additive in coconut and cottonseed oil as majority of the ionic liquids used in previous study are damaging for environment. The result of this research shows that formulated oil samples have superior tribological character and can be easily used in open environment as compared to the conventional lubricants.

METHODOLOGY

This study involves three phases: pin material and oil selection, experimental phase, and surface characterization. Figure 1 provides a summary of this research study by displaying the sequential information of research operations as a flow chart.

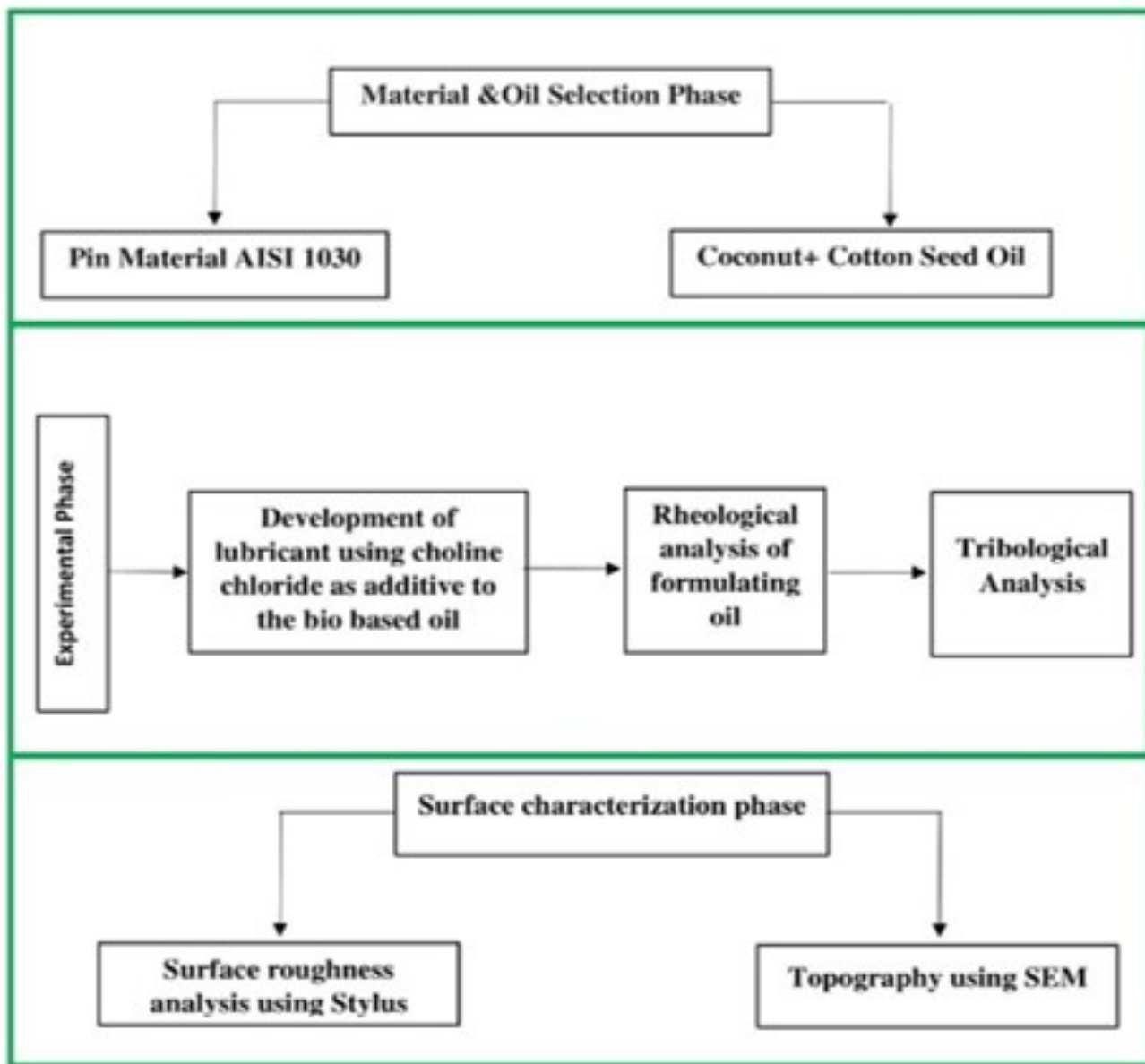


Figure 1. Workflow schematic of study with all the steps involved.

Lubricant

The current study aims to develop environmentally friendly bio-based lubricants. As base lubricants, coconut oil and cottonseed oil were employed. Coconut oil has a high viscosity, a high flash point superior lubrication and low vapor production. Coconut oil has much greater oxidative and thermal stability due to its saturated nature. Cottonseed oil, on the other

hand, can sustain high pressure due to its highly dense homogeneous strong lubricating film and dipolar character. 2-hydroxyethyl trimethylammonium chloride halide ionic liquid is used as an addition to increase the tribological and lubricating ability of bio-based oils by varying its concentration. For this experiment, three different concentrations of IL were chosen, namely 0.5 wt.%, 1 wt.%, and 1.5 wt.% as an addition in bio-based oils to inves-

tigate their tribological performances. Sigma-Aldrich private limited provides the ionic liquid, which is subsequently vacuum-dried.

Preparation of Bio based oil Containing IL

Both bio-based oils were preheated at 70°C before adding the IL and vigorously swirled for 1.5 h with a magnetic stirrer at 1050 RPM to achieve

lubricant homogeneity as shown in Figure 2. Eight lubricant samples are prepared, including two pure bio-based oil samples and six samples of coconut oil and cotton seed oil with varying concentrations of ionic liquid, namely 0.5 wt.%, 1 wt.%, and 1.5 wt.%. Rheological parameters such as the Kinematic viscosity of prepared oil samples at 40 °C were determined using a redwood viscometer as illustrated in Figure 3 in accordance with ASTM D445 standard. The kinematic viscosity of formulated oil samples is then calculated using Eq.1.

$$\text{Kinematic viscosity} = (A \times t) - B / (t) \quad (1)$$

A = Viscometer constant.

B = Kinetic Energy Coefficient

t = Flow of oil in Seconds

The values of these constants are A = 0.00247 and B = 0.5 for all the samples.

Tribo-Testing with a pin on disc

The anti-friction and anti-wear properties of the lubricant samples were examined using a pin-on-disc tribotesting machine (Koehler 93500). For testing each lubricant sample, an AISI 1030 cylindrical steel pin (99.13% Fe, 0.270-0.340% C, 0.60-0.90% Mn, 0.050% S, and 0.040% P) with a diameter of 8mm and a length of 10mm is utilized. Figure 3 and Table 1 illustrate the schematic diagram of the pin on the disc tribotester as well as the testing parameters.

After the tribological testing, acetone was used to clean the steel pins and then drying of pin is done by using industrial grade paper. After that, the wear scars of steel's surface were examined under a microscope. The tribo-testing machine automatically calculated the coefficient of friction (COF) using "data acquisition computer software" attached to it. To explore the effect of a specific load on the wear proportion of steel, the specific wear of the steel pin is measured in millimeter cube per newton meter. For that purpose, weigh the pin before and

after the test to determine the wear volume of steel and then use Eq. 2 to calculate specific wear.

$$\text{Specific Wear} = \frac{\text{change in volume}}{\text{load} \times \text{displacement}} \quad (2)$$

Surface Characterization Using SEM

To understand the lubricating mechanism of synthesizing ionic liquid-based bio-lubricants, the topography of the surface of the pin is investigated at a magnification of 500 μm using highly vacuum analytical TESCAN SEM VEGA3 microscope.

Direct Measurement of Surface Roughness Using Stylus Apparatus

Stylus apparatus depicted in Figure 4 is used to measure the surface roughness of steel pins directly. For each pin sample, two surface roughness readings are taken, and their average values are computed. For the measurement of surface roughness of steel pin move the stylus tip equipped with a detector across the surface of the sample.

RESULTS AND DISCUSSIONS

Rheological characteristics of lubricant samples

Table 2 displays the rheological characteristics of pure coconut oil, cottonseed oil, and combinations of IL (0.5 wt.%, 1 wt.%, 1.5 wt.%) into both bio-based oils. The kinematic viscosity of formulated samples increased when IL concentrations in both bio-based oils have been gradually raised. The improvement in kinematic viscosity of the formulated oil samples is caused by the development of a hydrogen bond between the IL and base oil, which in turn reduces molecular mobility.¹⁷

Pin on Disc Tribological Tests

Coefficient of friction of lubricated sample

Figure 5 and Figure 6 show the coefficient of friction (COF) from tribology testing for all formulated samples. When the concentration of IL is increased to 1.5 wt. %, the COF (0.09) of pure coconut oil reduce to 0.04. Strong tribo-chemical interactions between the surface of steel and formulated coconut oil's fatty acids create a lubricant layer of metal oxide that separated the contact surfaces. It reduces the negative consequences of friction between the contacting surfaces of pin and disc. COF trends are similar for cottonseed oil up to a 1% IL concentration.

Coefficient of friction posted by IL based coconut Oil

Further increases in ionic liquid Concentration result in the formation of residual film of chlorine which affects cottonseed oil's dipolar character and causes a sharp decline in the strength of the protective coating formed by the formulated sample. This causes the coefficient of friction to increase⁵³.

Specific wear of steel pin lubricated by formulated oil samples

Figure 7 depicts the specific wear rate of steel pin after tribology tests. Throughout the sliding period, the steel pin was subjected to continuous material removal and deformation processes when using pure coconut and cottonseed oil. In comparison to pure coconut oil, the specific wear of steel lubricated with coconut oil+1.5 wt.% IL is reduced by almost 80%. This enhancements in the anti-wear characteristic of formulated oil sample were caused by tribo-chemical interactions on the steel surfaces during the sliding period of the ionic liquid to produce stronger inter



Figure 2. Preparation of Lubrication Samples

Table 1. Test Parameters

Description	Values
Load	45N
Rotation Speed	400rpm
Track Radius	40mm
Time Duration	10 minutes

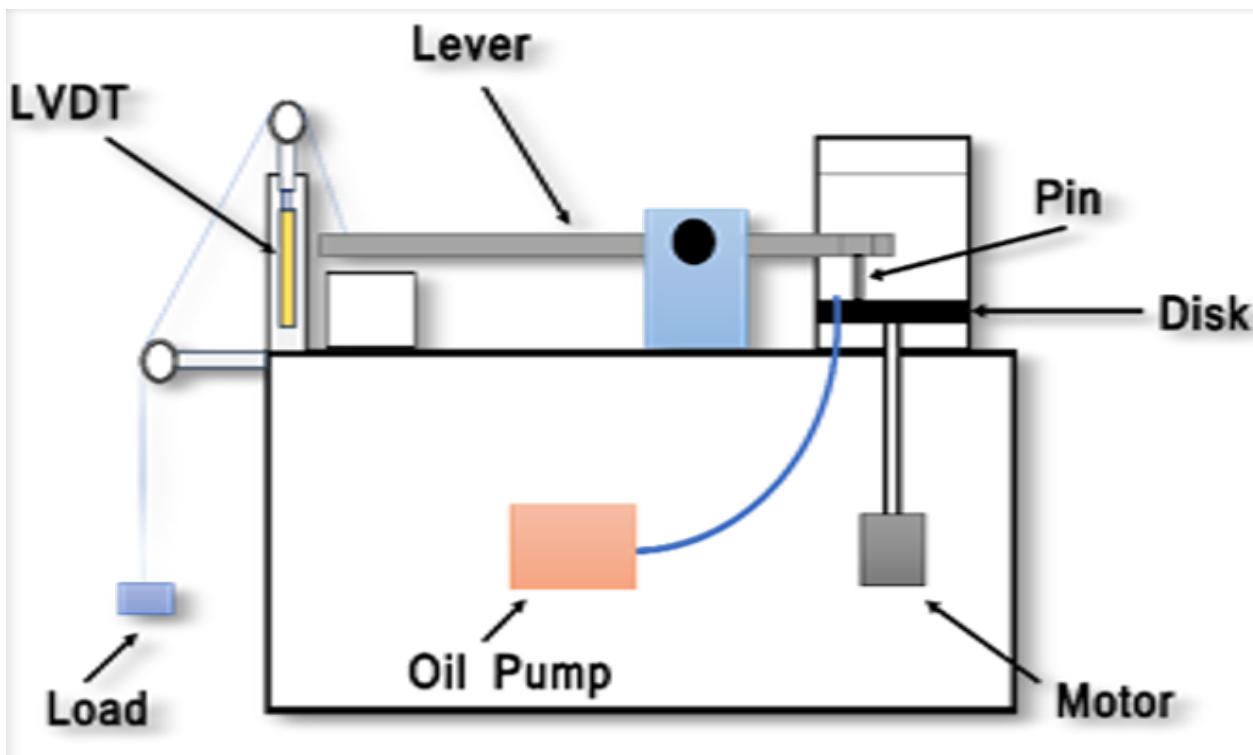


Figure 3. Schematic Diagram of Pin on Disc Tribo Machine



Figure 4. Stylus Surface Roughness Apparatus

Table 2. Rheological Characteristics of Lubricant Samples

Sample No.	Lubricants	Kinematic viscosity ($10^{-4} \text{m}^2/\text{s}$)
1	Pure Coconut oil	0.2519
2	Coconut oil+0.5wt.% IL	0.2602
3	Coconut oil+1wt.% IL	0.2702
4	Coconut oil+1.5wt.% IL	0.2830
5	Pure Cottonseed oil	0.3232
6	Cottonseed oil+0.5wt.% IL	0.3358
7	Cottonseed oil+1wt.% IL	0.3463
8	Cottonseed oil+1.5wt.% IL	0.3503

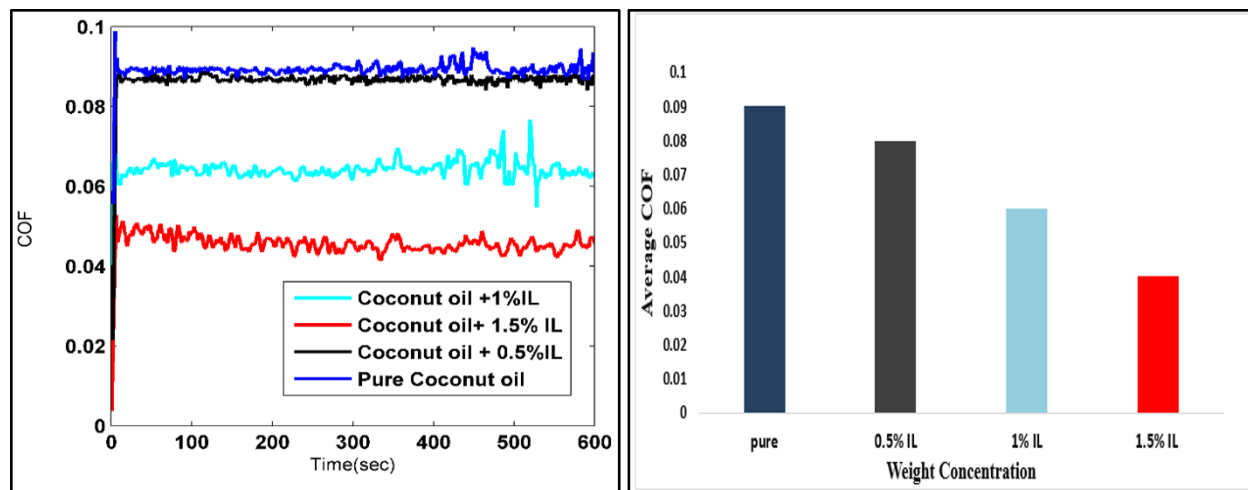


Figure 5. Coefficient of Friction Posted by IL based Coconut Oil.

and intramolecular contacts between the formulated oil sample and the metal surfaces results in the formation of tribo-film, which reduces the wear of steel pin. Thus, increasing the IL concentration in bio-based oils causes an increase in the physical proximity of IL molecules to metal surfaces, resulting in thicker and durable lubricant coatings that prevent metal's asperities direct contact¹³.

As the concentration of ionic liquid increases upto 1 weight percent, the specific wear rate of steel while employing cottonseed oil decreases. Figure 8 demonstrate that the wear rate of steel will increase as ionic liquid concentration increase further (1.5 wt.%) because residual chlorine films will form which act as a resistive barrier for the lubricating oil.

Surface morphology analysis

Figure 8 depicts SEM micrographs of pure coconut oil, cotton seed oil and Bio-based lubricants +IL lubricated samples. It can be noticed that the highly magnified images of steel pins at a magnification of 500m reveal less tendency of the shallow grooves and pits at high concentrations of ionic liquid due to production of oxide layer which acts as sacrificial layer and protects the steel surface from further dam-

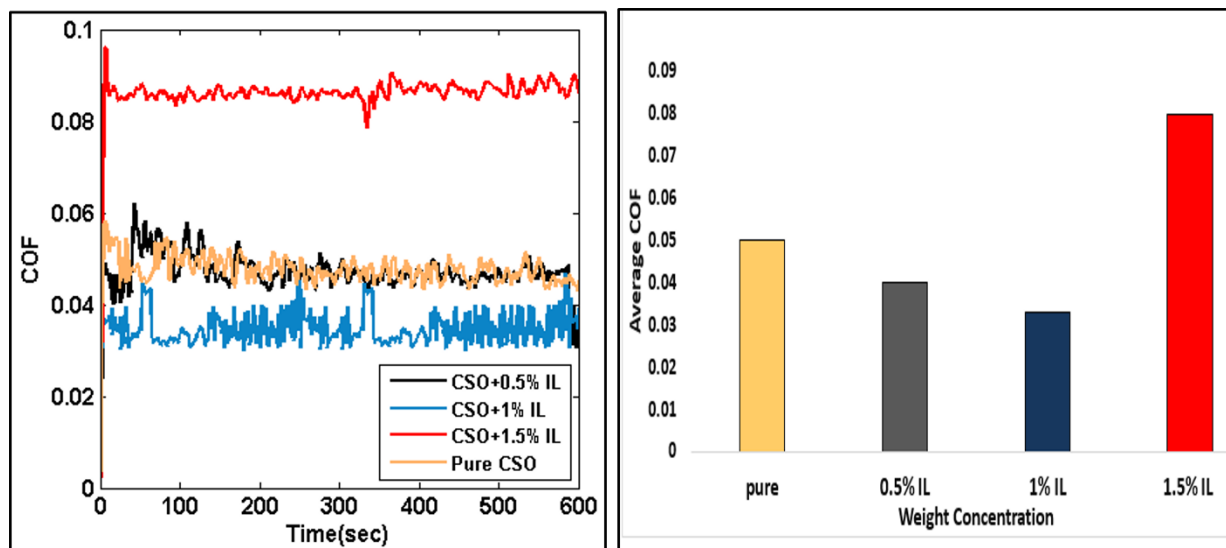


Figure 6. Coefficient of Friction Posted by IL based Cottonseed Oil.

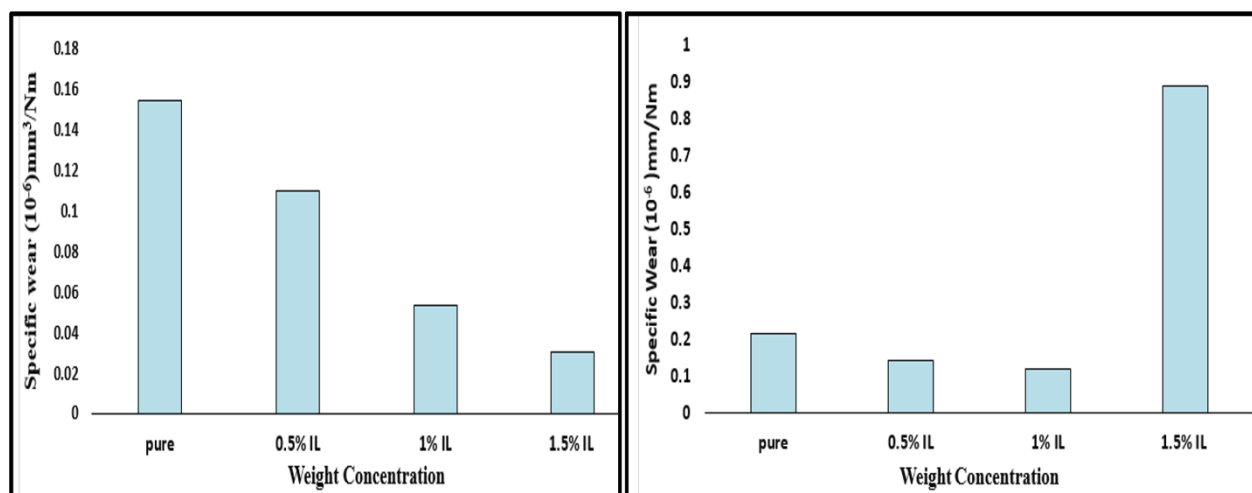


Figure 7. Specific Wear VS Weight Concentration of IL in Coconut Oil (left) and Cotton Seed (Right)

Table 3. Average Surface Roughness of Lubricated Steel pins

Sample No.	Lubricants	Avg. Surface Roughness μm
1	Pure coconut oil	0.893
2	Coconut oil+0.5wt.% IL	0.61
3	Coconut oil+1wt.% IL	0.436
4	Coconut oil+1.5wt.% IL	0.266
5	Pure cottonseed oil	0.861
6	Cottonseed oil+0.5wt.% IL	0.845
7	Cottonseed oil+1wt.% IL	0.562
8	Cottonseed oil+1.5wt.% IL	1.612

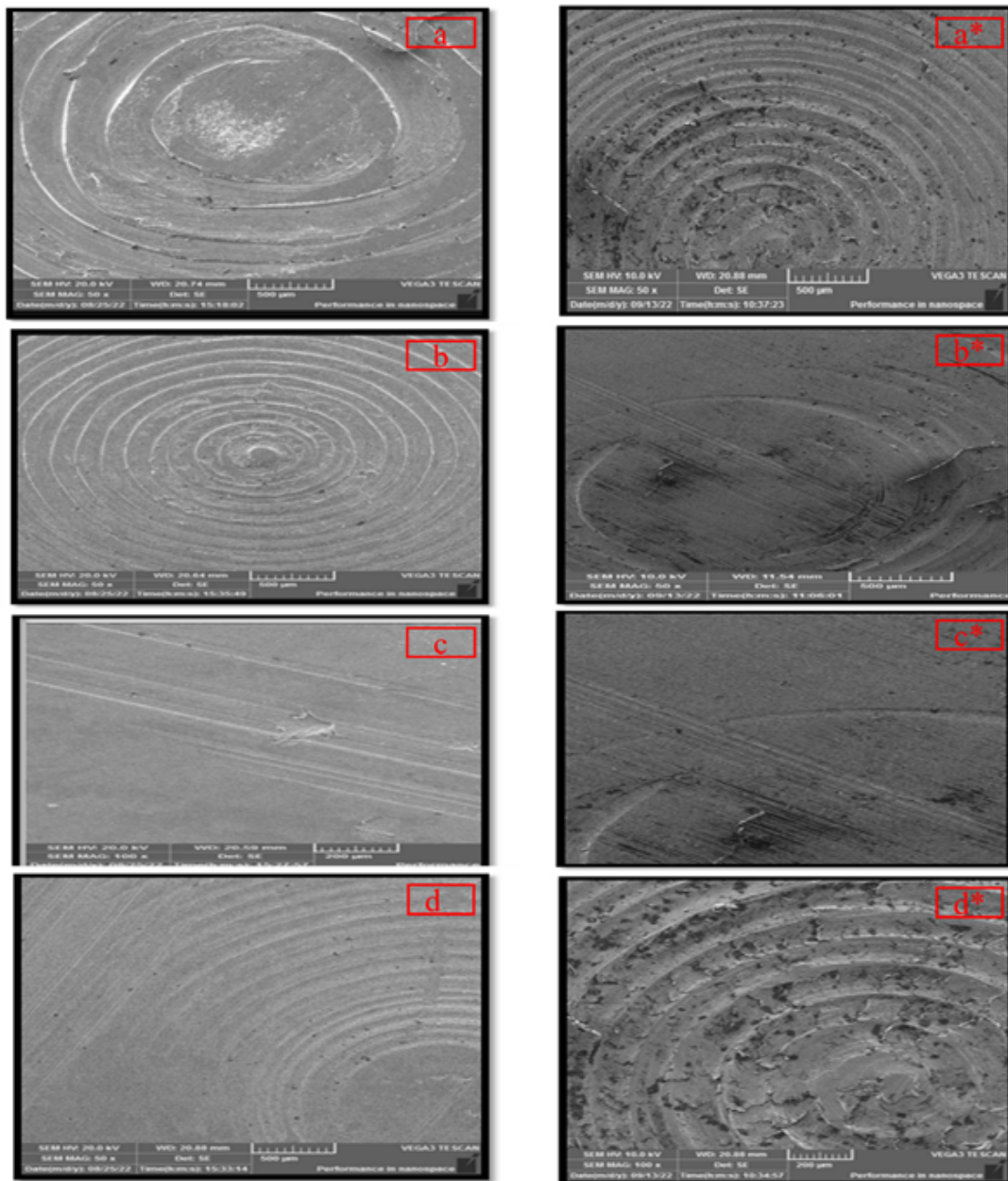


Figure 8. Comparison of SEM Morphologymicrographs of Steel Pin Lubricated by pure Formulated Oil Samplescoconut and cottonseed oil shown as (a & a*), (b & b*) represents Steel Pin Lubricated with addition of 0.5 wt.% of [C₅H₁₄CINO] with base oils, (c & c*) represents Steel Pin Lubricated with addition of 1 wt.% of [C₅H₁₄CINO] with base oils, (d & d*) represents Steel Pin Lubricated with addition of 1.5 wt.% of [C₅H₁₄CINO] with base oils

age. However, for a cottonseed oil containing 1.5% IL, the micrograph shows oxidative wear (black strains) and scuffing (abrasive wear) of the surface due to the production of residual layers of Chlorine.

Surface roughness of steel measured by Stylus apparatus

The maximum roughness of steel pin was posed by pure coconut oil and pure cottonseed oil. The surface roughness value of steel pin reduces as the IL concentration in the base oils increases, due to a reduction in COF by increasing the IL concentration. Cotton seed oil + IL1.5 % has an average roughness of about 1.612 μm , which is roughly 19% higher than pure cottonseed oil. The average roughness of all steel pin lubricated by the formulated oil samples and pure bio-based oils are shown in Table 3.

CONCLUSIONS

The addition of bio-compatible ionic liquid (IL) to both bio-based oils had a positive synergistic effect on their tribological performance. For coconut oil, 1.5 weight percent of IL addition was shown to be an appropriate treat rate, resulting in outstanding tribological properties, better rheological characteristics, and superior anti-wear and anti-friction performances. Superior friction reduction (56%) and reduced specific wear (80%) have been demonstrated using coconut oil + IL1.5%.

Similarly, tribological investigation of cottonseed oil revealed a reduction in friction up to 1% ionic liquid concentration, but at 1.5 wt.% IL concentration the coefficient of friction increased to 60% in comparison to pure cotton seed oil. Cottonseed oil's COF has increased due to the production of a chlorine residual film which causes insufficient ionic liquid dispersion in the bio-based oil.

Based on these findings, it can be inferred that all formulated samples, with the exception of cottonseed oil +

1.5 IL, have a great deal of potential for use as sustainable lubricants which help to further promote "greener" manufacturing processes that are more energy-efficient and have a positive environmental impact. Regarding environment friendly operations, all the developed samples are discovered to be a good substitute for the industrially common mineral oil-based lubricants.

Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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