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RESEARCH

Friction and Wear Properties of Phosphonium Based Ionic Liquid Used as Additive in Synthetic and Bio Based Lubricants

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A B S T R A C T

This research investigated the tribological performance of phosphonium-based ionic liquid Tri hexyl tetra decyl-phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate (IL) when used as an additive at a concentration of 1wt% to Cotton seed oil (CSO), Rattan Jot oil (RJO), Waste Cooking oil (WCO) and Polyalphaolefin (PAO) at different operating conditions. The results were compared with conventional PAO base oil. The tribological performance of all lubricants was studied using a Vertical Universal Testing Machine (MMW1A) and wear scar diameter were measured using an electronic microscope OLYMPUS DX-1000. The results indicated that the addition of ionic liquid further enhanced the tribological performance in terms of wear for all bio-lubricants except for waste cooking oil. However, the addition of ionic liquid does not have much effect on the tribological performance of PAO. Rattan Jot oil was found to have the best tribological properties with the addition of ionic liquid.

1. INTRODUCTION

In machines, the major cause of mechanical losses is friction which can be decreased by the use of lubricants. Lubricants introduce a thin film between two interacting bodies that helps reduce friction and wear between them. Apart from the reduction of friction and wear, lubrication also has other applications such as releasing heat produced during the sliding of two bodies, removing wear debris, transmitting forces between two bodies, decreasing operating temperature, and preventing materials from corrosion [1]. In passenger cars use of lubrication is important as almost one-third of the total energy produced from the burning of fuel is used by friction [2]. In an IC engine, almost 17% of the total energy produced by burning fuel is utilized in terms of friction losses [3]. Lubricants find their use in many fields such as automobiles, machine components, the pharmaceuticals industry, bearings, gears, and hydraulic systems.

Lubricants are generally classified into three categories: synthetic, mineral, and bio-lubricants. Synthetic lubricants are mostly generated from organic raw material by tailoring the reactions to get the required product. Examples of synthetic lubricants are synthetic esters, silicon, and polyalphaolefin. Mineral lubricants are derived directly from crude or petroleum reserves such as aromatic oils. Contrary to these, bio-lubricants are derived from animals and plants and are more environmentally friendly [4]. In the automobile industry mineral oils which are obtained from chemical modification of crude oil are mostly used as lubricants. Due to limited resources of petroleum reserves, toxic emissions which include noxious metals, problems in the disposal and overly increasing prices of petroleum products the conventional oils are being replaced by biodegradable and nontoxic vegetable oil [5,6].

Bio lubricants are derived from animal and plant feedstock. They have good chemical properties and can be used for lubrication purposes. Bio lubricants especially plant oils possess most of the properties that are required for lubrication which include a high viscosity index (VI), low volatility, higher molecular weight, low vapor pressure, and high flash point [7,8]. They consist of ester bonds which enable the molecules to attach themselves to metal surfaces and provide good lubricity as compared to nonpolar petroleum-based mineral oils [9]. In general bio lubricants are renewable, nontoxic to aquatic life, and biodegradable due to which it would be more favorable to use them as an alternate fuel. Plants oil when compared with mineral oils has better chemical properties but due to the fact that they oxidize when exposed to high temperature, they are not tribologically favorable to use under high-temperature conditions [10]. Vegetable oils are mainly composed of triglycerides (TAG) which can easily undergo elimination reactions and hence these vegetable oils get unstable at higher temperatures [8]. In order to solve this problem the lubricant is chemically modified by replacing glycerol with more suitable alcohol or by the addition of additives [11,12]. Bio-lubricants work really well under boundary lubrication regime but adding a certain quantity of ionic liquids can further enhance their tribological properties [13].

Ionic liquids are organic salts that exist in liquid form and have low melting temperatures as compared to water. Ionic liquid is a subject that is being investigated by researchers nowadays for better tribological performances due to the properties like low melting points, non-flammability, environmental friendliness, and low volatility [14]. Ionic liquids can be classified based on the cations present in them. Generally, ionic liquids have phosphonium, imidazolium, ammonium, or pyrrolidinium cations in them [15]. Studies have been done on using ionic liquid as a lubricant [16,17]. Ionic liquids, when analyzed from a tribological point of view outperformed most of the conventional lubricants. Ionic liquids although having great tribological properties cannot be used as a lubricant because of their high cost. However, they can be mixed with other base oils to improve their tribological properties [18,19]. To date, a lot of ionic liquids are used as an additive to different lubricants and their effect on the tribological behavior of those lubricants has been investigated [11,20]. There are variety of ionic liquids which can be used as an effective lubricant additive. In some research, mixtures of ionic liquid and other particles have been investigated as effective additives to lubricants [21,22]. These additives were investigated at different concentrations, different operating conditions, and under different applications as possible additives to lubricants. When the friction and wear behavior of these lubricants were studied they were found to be a lot more effective as compared to lubricants without these additives [23].

The effect of ionic liquids on the tribological properties of lubricants depends a lot on their bonding with the lubricant molecules. The solubility of ionic liquids in nonpolar oils is negligible due to which it forms ionic-oil emulsions and does not perform well. However, they are miscible in polar base oils [24,25]. A study has shown that ionic liquids are more soluble in solvents having more polarity and hence the ionic liquids show the best performance with base oils that have ester groups in them [26]. Also among ionic liquids, those having cations of phosphonium are found to have outstanding friction and wear characteristics especially in systems under boundary and mixed lubrication regimes [15]. [27] studied the effect of two ionic liquids tri hexyl tetra decyl phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate (IL1) and tri hexyl tetra decyl phosphonium bis (2-ethylhexyl) phosphate (IL2) as an additive at 1wt% to Polyalphaolefin (PAO) at different operating temperatures and slide to roll ratios. The results indicated that base oils with IL1 in them outperformed the other samples in terms of tribological performance under all operating conditions. Similarly [28] used tri hexyl tetra decyl phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate as an additive to TMP ester and found a huge enhancement in its frictional properties.

In this study Polyalphaolefin (PAO) and three bio-lubricants Cotton seed oil (CSO), Rattan Jot oil (RJO) and Waste Cooking Oil (WCO) were used as base oils and 1wt% of IL was used as an additive in these oils. Each bio-lubricant was tested for their tribological compatibility both in its pure state and after the addition of an ionic liquid.

2. EXPERIMENTAL PROCEDURE

2.1 Lubricants Used

Feedstock for bio-lubricants, including RJO, CSO and WCO were selected for the study. RJO and CSO were procured from the local market and were filtered to remove any impurity and WCO was collected from home. The chemicals used during productions were Methanol, TMP, KOH, NaOCH3, and propanol. All the chemicals required were bought from a manufacturer (Sigma Aldrich). These oils were then converted into bio-lubricants using transesterification reaction. A conventional lubricant PAO brought from (Sigma Aldrich), was also used for this comparative study. The ionic liquid based lubricants were produced by the addition of 1wt% ionic liquid and the mixtures were constantly stirred for 30 minutes at a temperature of 60°C.

2.2 Physiochemical Properties

Physiochemical properties of all the lubricants used were measured according to ASTM standards and are listed in Table 1.

Table 1. Physiochemical properties of lubricants.

Properties	Standard	Units	PAO	PAO+IL	CSO	CSO+IL	RJO	RJO+IL	WCO	WCO+IL
Density@ 20°C	ASTM D1480	kg/m^3	827.6	850.8	904.6	932.8	906.4	927.2	907.8	930.8
Viscosity@ 40 °C	ASTM D445	cSt	65.1	59	27.7	23.27	29.9	35.85	36.8	25.94
Viscosity@ 100 °C	ASTM D445	cSt	9.8	15.68	5.94	7.22	6	8.9	7.9	8.17
Viscosity Index	ASTM D2270	-	133	282	167	308	137	243	194	321
Pour Point	ASTM D97	°C	-41		-10.2	-10	-13.2	-12.4	-12.8	-12.2
Flash Point	ASTM D92	°C	230	218	175	155	150	205	155	187
Total Acid Number	ASTM D664	mmKOH/g	0.21	0.3	0.25	0.38	0.29	0.43	0.3	0.38
Total Base Number	ASTM D4739	mmHCL/g	0	0.42	0.14	0.62	0.15	0.44	0.1	0.78

2.3 Experimental setup

Four ball tests were conducted on the selected lubricants using the Vertical Universal Friction Testing Machine (MMW-1A) shown in Fig. 1. This four-ball configuration consists of four AISI 52100 steel balls with a diameter of 12.7mm and average surface roughness R_a=0.08μm, out of which three balls were fixed in a fixture containing cavity where the test lubricant was filled and one ball was held in a rotating spindle shown in Fig. 2. The tests were conducted following the standard ASTM D4172, with an additional parameter of 100°C along with 75 °C temperature, which represents the operating temperature for many mechanical systems. All lubricants were tested at two different loads 147N and 392N, two operating temperatures 75°C and 100 °C and the spindle rotating at a speed of 1200 rpm. For the purpose of repeatability, each test was conducted twice and average values were noted.

The conditions of the experiments are listed in Table 2. Before the start of each experiment the fixture and balls were cleaned with acetone. After that, 9mL of a sample of oil was taken and added to the cavity of the fixture. Three balls were taken and fixed in the fixture having oil inside it and one ball was fixed inside the spindle as shown in Fig. 2. Using the PID controller the temperature of the oil was raised at intervals of 5 °C to the required temperature. After reaching the required temperature the setup was allowed to maintain the required temperature for a time of about 20 minutes. After that load was applied and the test was allowed to run for 60 minutes and data for COF was stored. After completion of the test the setup was unloaded, and the balls were taken out of the fixture and cleaned.

The wear scars on the balls were marked to be further studied using a microscope. A digital optical microscope OLYMPUS DX-1000 shown in Fig. 3 was used for the study of wear and average surface roughness (R_a). The surface roughness was measured at cutoff filter of 80μm. Flow chart representing four ball test procedure is shown in Fig. 4.

Table 2. Four ball test conditions.

Operating Condition	Value
Speed of Spindle	1200 rpm
Temperature	75°C and 100°C
Load	147N and 392N
Soaking Time	20 Minutes
Run Time	60 Minutes

Fig. 1. Vertical universal friction testing machine (MMW1A).

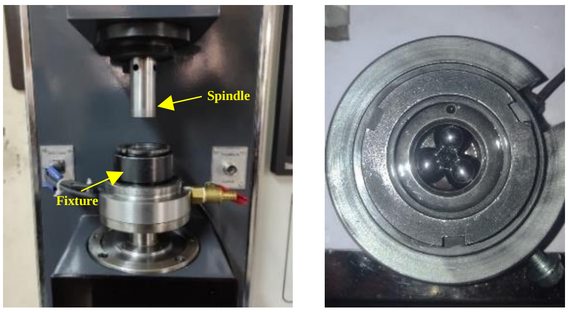


Fig. 2. Four ball fixture and spindle configuration.

Fig. 3. Olympus DX-1000.

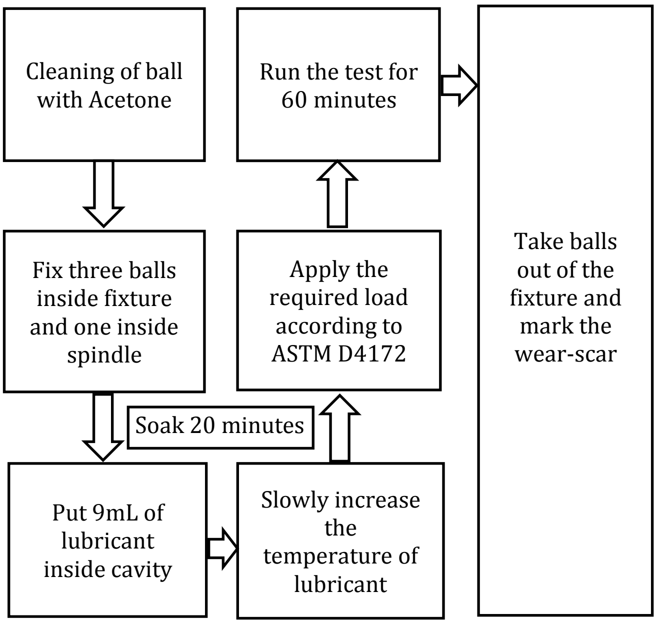


Fig. 4. Flow chart of four ball test.

3. RESULTS AND DISCUSSIONS

During the four ball tests the Vertical Universal Friction Testing Machine (MMW-1A) generated real-time COF graphs with respect to the time showing the complete behavior of the lubricant over the test period of 60 minutes. Using the COF plots, the average value of COF was obtained for each lubricant. Fig. 5 shows the average COF. For bare lubricants, CSO has the least value of COF (0.042) whereas WCO has the highest value of COF (0.067) at normal load of 147N. The reason for increased value of COF of WCO can correspond to the fatty acid composites present in WCO [29]. These WCO contain saturated fatty acids which can increase friction as compared to monosaturated fatty acids like oleic acid which is present in higher concentration in CSO [30]. Saturated fatty acids have single bonds between carbon atoms resulting in straighter more rigid chains. This enables tighter packing of molecules and creates densely packed layer on surface that offers more resistance to shearing force which leads to higher friction. Monounsaturated fatty acids, on other hand have one double bond which introduces a kink in the chain. This kink disrupts tight packing and reduces resistance to shearing forces leading to lower friction [31]. At 392N and 75°C, among the bare lubricants, PAO has the highest COF value (0.069), whereas RJO shows the lowest COF value (0.039). This greater value of COF for PAO as compared to bio lubricants can be explained by its non-polar nature [32,33]. The non-polar molecules does not form strong bonds with metal surfaces as compare to polar molecules present in bio lubricants. This can lead to a less effective lubricating film, resulting in higher friction.

At 75°C, addition of ionic liquid to PAO has caused an increase in the value of COF by 20.27% and 18.82% at normal load of 147N and 392N when tested for a time period of 60 minutes as shown in Fig. 5. This is attributed to the incompatibility of ionic liquid in non-polar base oils [34]. Ionic liquids form oil-ionic liquid emulsions in non-polar oils due to their negligible solubility. This can result in uneven film formation on metal surfaces resulting in increased friction [35]. In Fig. 5, for 147N load and 75°C, CSO encountered 46.15% increment in COF after adding ionic liquid then RJO and WCO. RJO and WCO showed decrement of 31.57% and 38.8% respectively in COF. The decrease in COF values of RJO and WCO when added with ionic liquid can be explained by the rapid formation of tribolayer at this operating condition which effectively separate the asperities from each other. At the contact points between rubbing surfaces, friction along with shear caused localized high temperatures and pressures. These extreme conditions triggered reactions between the lubricant base oil, ionic liquid and contacting surface which lead to the formation of tribo-layer for these oils. At 75°C and 392N, a decrease of 8.92% can be seen for CSO when added with ionic liquid whereas for RJO, addition of ionic liquid caused an increase in its COF value. This increase in the value of COF when combined with an ionic liquid shows that the addition of the ionic liquid to RJO negatively affects its frictional behavior at higher loads. This is due to the incompatibility of the oil with the ionic liquid at higher loads, which may lead to unstable tribofilm formation that breaks down readily under high loads. For WCO, addition of ionic liquid didn't show any effect on COF value at 392N.

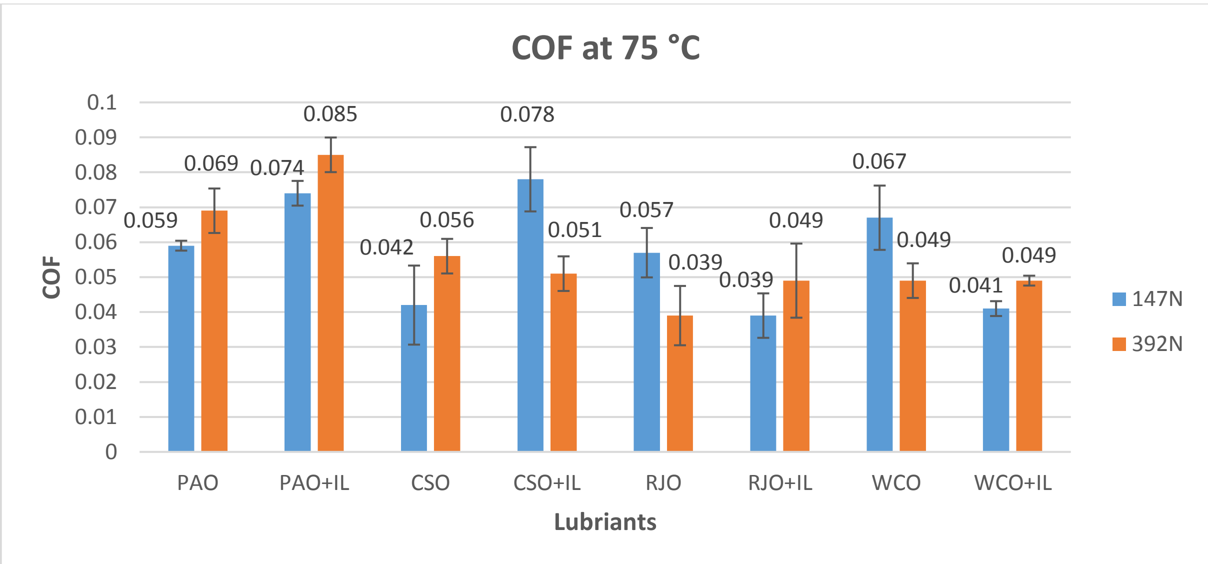


Fig. 5. Average COF of lubricants under different loads at 75°C temperature.

Fig. 6 shows the average COF of all lubricants. As shown in Fig. 6 that in the case of bare lubricants, PAO shows the highest values of COF at both normal loads 147N and 392N. This is because PAO has lower viscosity index as compared to bio-lubricants as shown in Table 1. At 100°C and 147N, among all bio-lubricants, WCO showed the least COF while at 100°C, 392N load RJO showed the least value of COF. This behavior of RJO can be explained by the fact that due to higher viscosity it offers a thicker lubricant film. This helps withstand higher pressures, thus reducing the direct metal to metal contact and friction. RJO also have better thermal stability thus maintaining its lubricant properties even at extreme conditions such as higher loads and temperatures. The value of COF at 147N load is in accordance to the viscosity index of lubricants i.e., lubricants with greater viscosity index have less value of COF. Lubricants with a higher viscosity index remain more stable at higher temperatures compared to those with a lower viscosity index. This stability allows them to maintain effective lubrication and minimize the COF under high-temperature conditions. The lesser COF value of RJO at 392N corresponds to its better load-carrying capacity.

From Fig. 6, at 100°C and 147N, there is decrement of 4.41% in COF of PAO+IL, while the COF values for CSO and WCO remains almost the same after addition of IL. Whereas a decrease of 17.3% can be seen for RJO values after the addition of ionic liquid to it. At 100°C and 392N, the addition of ionic liquid causes a decrease of 40.38% and 1.78% in COF values for CSO and WCO whereas in the case of PAO and RJO an increase of 6.5% and 5.13% can be seen after the addition of ionic liquid as show in Fig. 6.

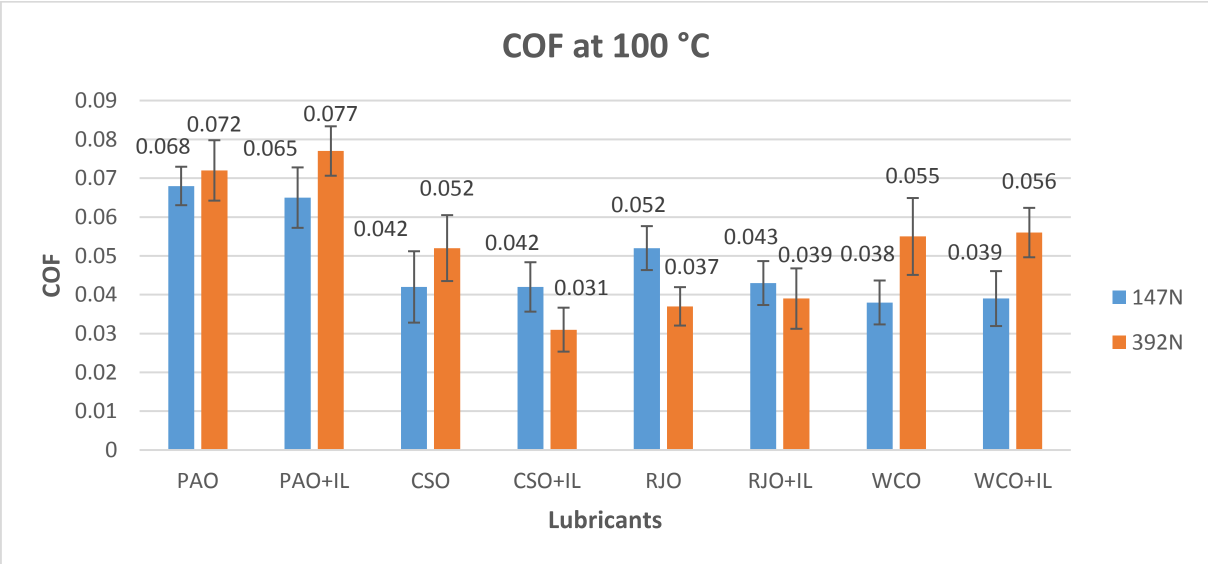
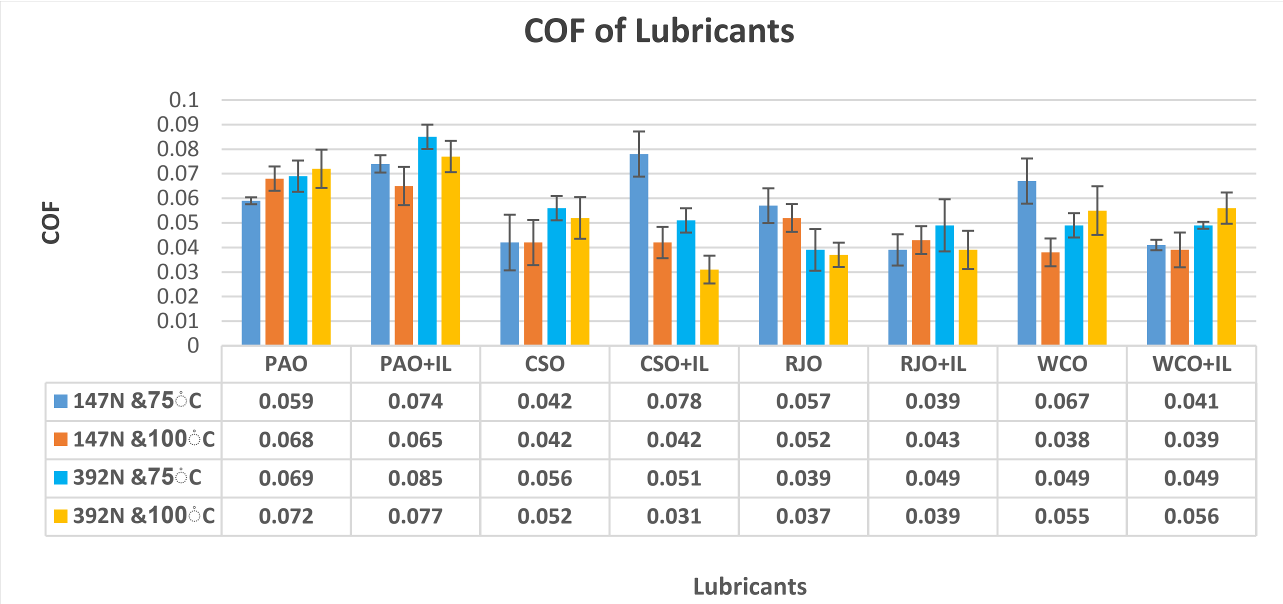


Fig. 6. Average COF of lubricants under different loads at 100°C temperature.

Fig. 7 shows the COF values for lubricants at all operating conditions. As shown in Fig. 7 at 75°C, with increase in load from 147N to 392N, a decrease in COF can be seen for CSO+IL, RJO and WCO. This decrease in COF values is due to the increase in surface roughness and increased amount of wear debris removal [36]. These wear debris act as a filler between the surfaces which reduces the direct contact of asperities and as a result decrease in the value of COF can be seen [37,38]. This effect will be further described in more detail in Figures 11 and 12. Similarly at 100°C same effect of increase in load can be seen for CSO+IL, RJO, RJO+IL. The increase in temperature has caused a decrease in COF values for all lubricants with additives of ionic liquids [39] except for RJO+IL sample at 147N and WCO+IL at 392N as shown in Fig. 7.



	PAO	PAO+IL	CSO	CSO+IL	RJO	RJO+IL	WCO	WCO+IL
147N &75°C	0.059	0.074	0.042	0.078	0.057	0.039	0.067	0.041
147N &100°C	0.068	0.065	0.042	0.042	0.052	0.043	0.038	0.039
392N &75°C	0.069	0.085	0.056	0.051	0.039	0.049	0.049	0.049
392N &100°C	0.072	0.077	0.052	0.031	0.037	0.039	0.055	0.056

Fig. 7. Average COF of lubricants at all operating conditions.

Fig. 8 shows the average Wear Scar Diameter (WSD) of all lubricants. For bare lubricants, PAO has the highest value of WSD as compared to bio-lubricants at both normal loads. The better performance of bio-lubricants as compared to PAO can be attributed to the presence of fatty acid molecules in bio-lubricants which react with metal surfaces and form a low shear strength layer over the metallic surfaces [40]. Among the bio-lubricants without ionic liquid additive, the WCO samples exhibited the least WSD at both normal loads as shown in Fig. 8.

At 75°C, the addition of ionic liquid has no significant effect on WSD value of PAO at 147N whereas at 392N load the value of WSD increases by 3.95% for PAO after the addition of ionic liquid. These values represent the ineffectiveness of ionic liquid with PAO because of its non-compatibility with it. At 75°C temperature and 147N load, the addition of ionic liquid has caused a decrease of 9.56% and 1.93% in WSD for RJO and WCO whereas for CSO 6.92% increase in WSD can be seen with the addition of ionic liquid. At 75°C temperature and 392N load, the addition of ionic liquid has caused a decrease of 7.8%, 17.7%, and 8.44% in WSD for CSO, RJO, and WCO respectively. This decrease in WSD represents the enhancement in load-carrying capacity of bio-lubricants with the addition of ionic liquid.

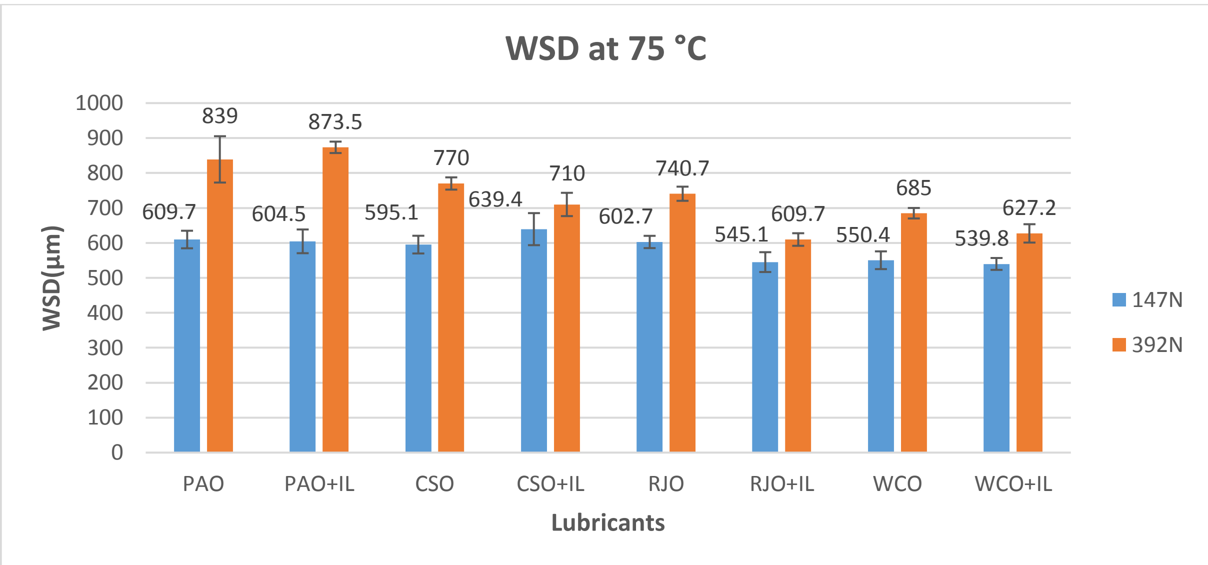


Fig. 8. Average WSD for lubricants under different loads at 75°C temperature.

Fig. 9 shows the average WSD of all lubricants. For bare lubricants, it can be seen that RJO has the greatest value of WSD at both normal loads. This high value of wear scar diameter shows the lower thermal stability of RJO as compared to other lubricants. For bare lubricants at 100°C temperature, WCO has the lowest value of WSD at 147N load whereas PAO has the lowest value of WSD at 392N load.

At 100°C temperature, the addition of ionic liquid has shown an increase of 28.17% and 21.75% for the WCO sample at a normal load of 147N and 392N respectively. This increase in WSD values represents the ineffectiveness of ionic liquid in WCO at high temperatures. Fig. 9 shows that CSO with the addition of ionic liquid has caused a decrease of 27.68% and 14% in WSD at 147N and 392N load respectively. Similarly, for RJO with the addition of ionic liquid a decrease of 63.93% and 22.92% can be seen in WSD at a normal load of 147N and 392N respectively. This decrease in WSD values for RJO and CSO, when added to ionic liquid, is because of the higher thermal stability of ionic liquid samples.

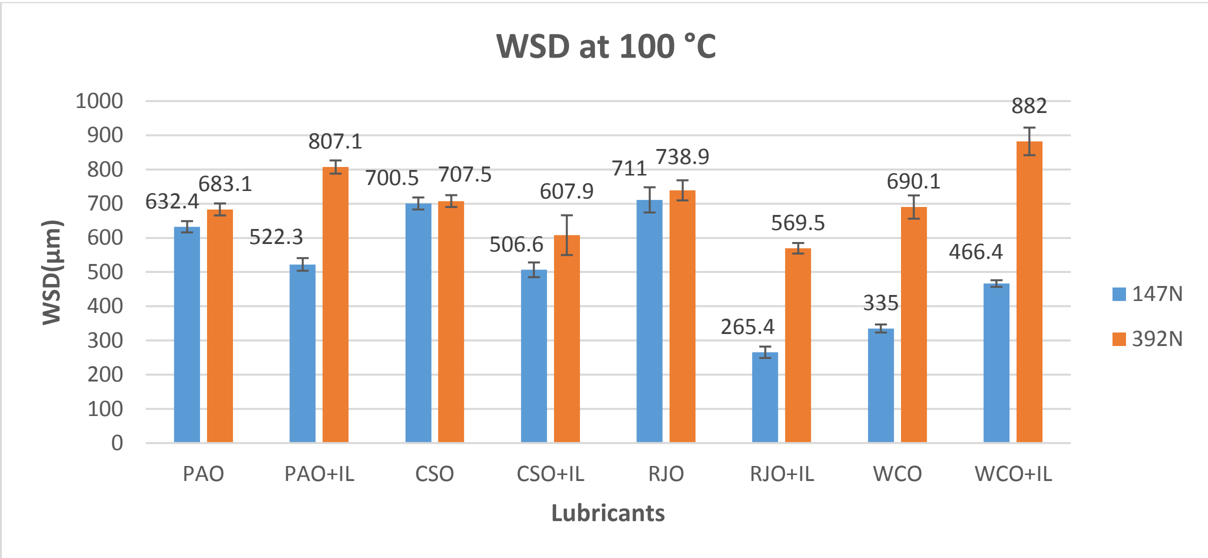


Fig. 9. Average WSD for lubricants under different loads at 100°C temperature.

Fig. 10 represents the average WSD values for lubricants under all operating conditions. From Fig. 10 RJO+IL samples have the least values of WSD at all operating conditions. With the increase in load from 147N to 392N the WSD of all lubricants increased as shown in Fig. 10 [41].

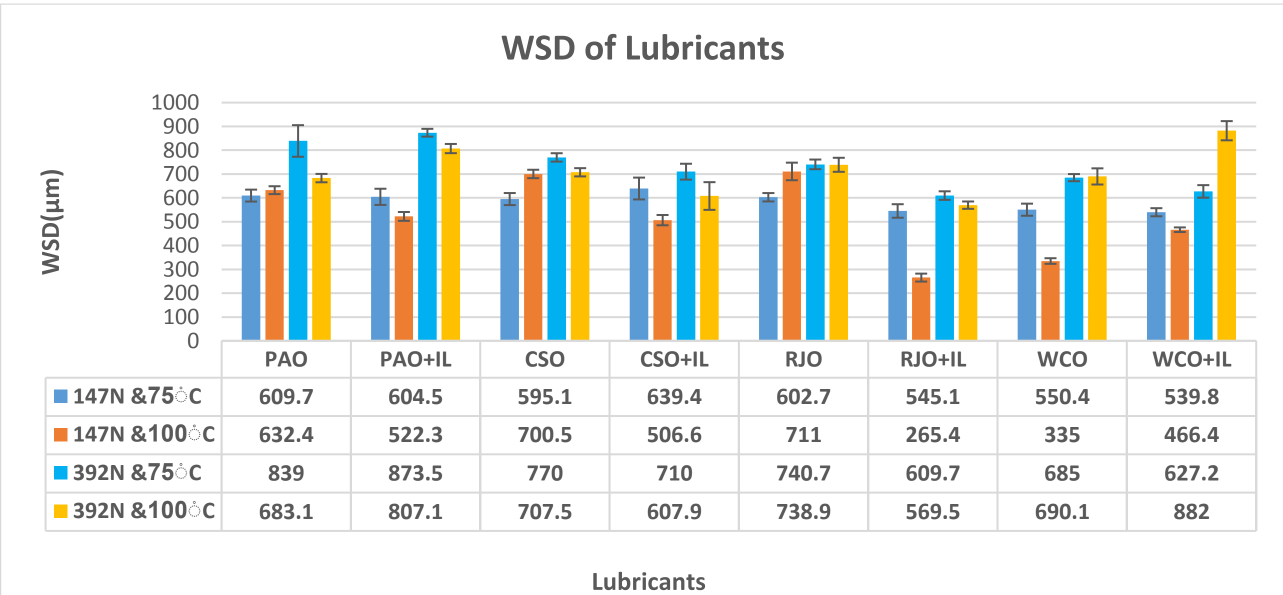


Fig. 10. Average WSD of all lubricants at all operating conditions.

This increase in WSD can be explained by the decrease in the strength of materials with an increased amount of heat generation due to an increase in load which can cause plastic deformation in the material [36,42]. Also Fig. 10 shows that with an increase in temperature from 75°C to 100°C, the WSD value decreases for all samples containing additives of ionic liquids except for WCO+IL at 392N. This decrease in WSD values can be explained by the fact that by increasing the temperature, viscosity decreases and ionic liquid additives inside the lubricant took the dominant role by forming a protective film over the surface, which leads to a decrease in WSD values [39].

Fig. 11 shows the values of average surface roughness (R_a). From Fig. 11 with the increase in load from 147N to 392N, surface roughness values for CSO+IL, RJO, and WCO increases. On the other hand, as Fig. 7 shows, that at 75 °C these lubricant's coefficient of friction (COF) values drops as the load increases. A decrease in surface roughness values can be seen with an increase in load for all other lubricants where the COF increases. These findings show that in contrast to COF, surface roughness, and load variation have an inverse connection. In particular, an increase in surface roughness values was noted for some lubricants with an increase in load loads while COF values drop [36].

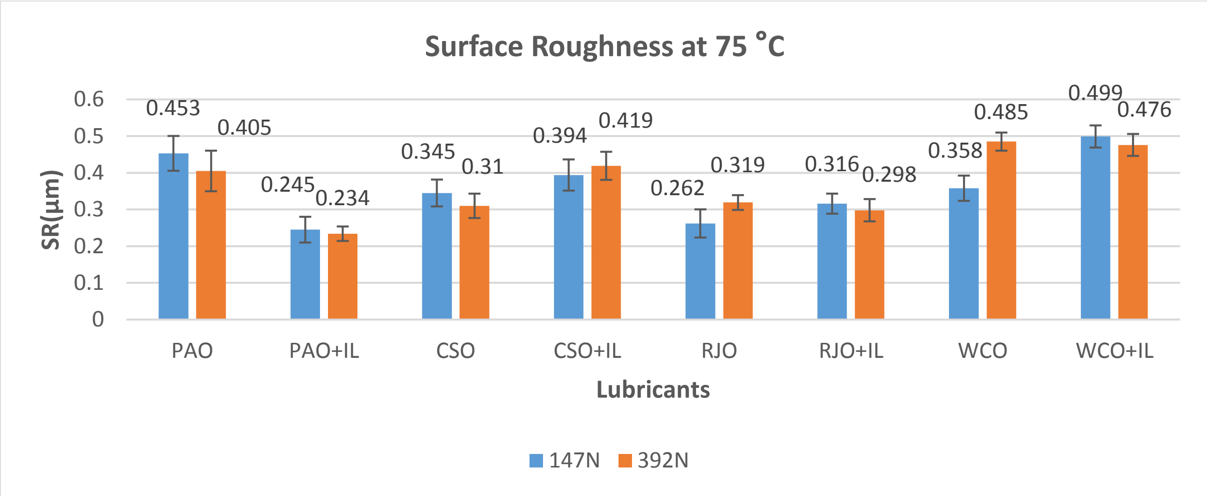


Fig. 11. Surface roughness of lubricants under different loads at 75°C temperature.

Fig. 12 shows the average surface roughness (R_a) values for all lubricants. At 100°C, comparable trends between COF and surface roughness values are visible. As shown in Fig. 12 and Fig. 7, for CSO+IL, RJO+IL, and RJO, an increase in load causes a rise in surface roughness values while their COF falls. These surface roughness patterns highlight how operating conditions have a major impact on both COF and surface roughness.

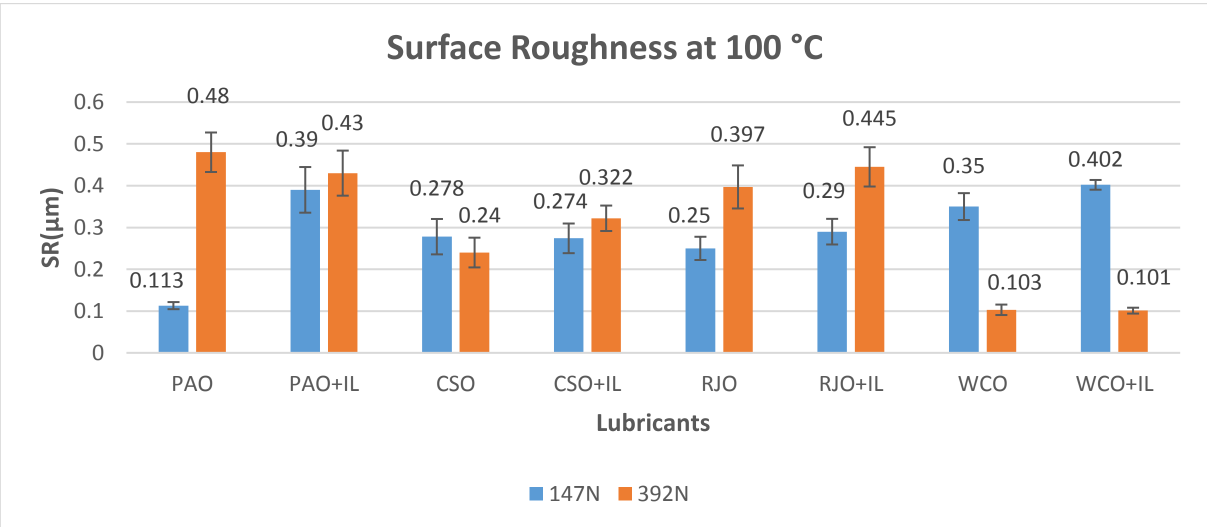


Fig. 12 Surface roughness of lubricants under different loads at 100°C temperature.

Wear scar images for all lubricants taken with an OLYMPUS microscope at a magnifying power of 280x are shown in Fig. 13 to Fig. 16.

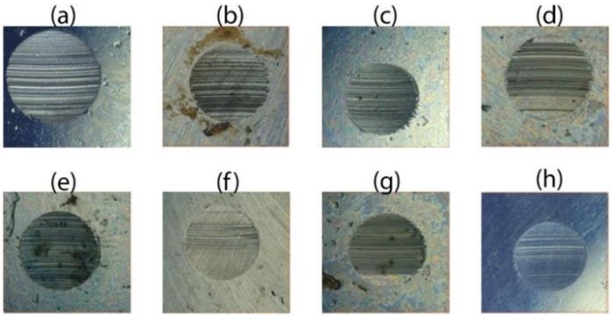


Fig. 13. WSD at 147N and 75°C for (a) PAO (b) PAO+IL (c) CSO (d) CSO+IL (e) RJO (f) RJO+IL (g) WCO (h) WCO+IL.

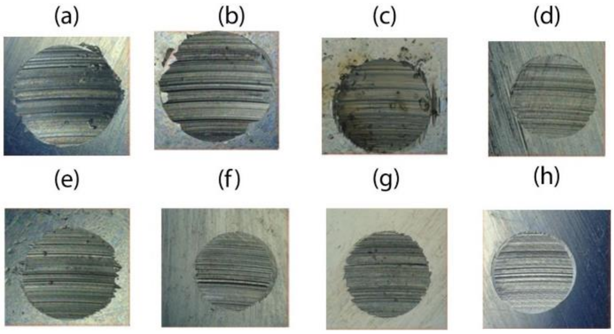


Fig. 14. WSD at 392N and 75°C for (a) PAO (b) PAO+IL (c) CSO (d) CSO+IL (e) RJO (f) RJO+IL (g) WCO (h) WCO+IL.

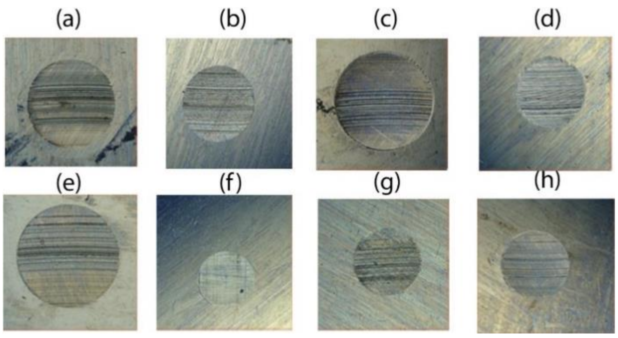


Fig. 15. WSD at 147N and 100°C for (a) PAO (b) PAO+IL (c) CSO (d) CSO+IL (e) RJO (f) RJO+IL (g) WCO (h) WCO+IL.

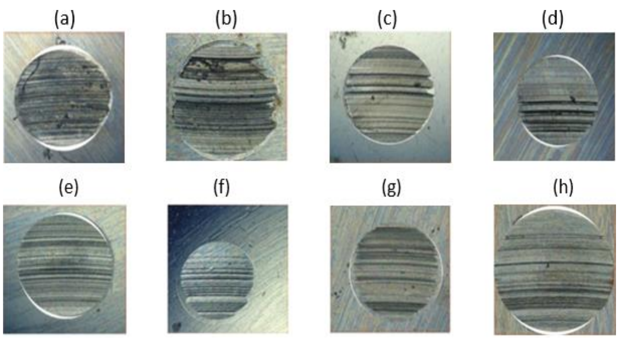


Fig. 16. WSD at 392N and 100°C for (a) PAO (b) PAO+IL (c) CSO (d) CSO+IL (e) RJO (f) RJO+IL (g) WCO (h) WCO+IL.

Wear scar images are analyzed at a scale of 200 µm in image width. Abrasive wear can be seen for all samples. It can be observed from these images that for higher loads the abrasive wear has increased for all samples. This trend is due to increase in load, more heat is generated, and with increased contact pressure it causes increased mechanical wear and hence decreases the strength of the material. Also, with an increase in operating temperature, the wear has increased for all bare lubricants. In the case of lubricants containing ionic liquids, a trend of decrease in wear scars can be seen. This can be explained by the higher thermal stability of ionic liquid-based lubricants. It can be observed that the abrasive wear in the case of PAO is more intense as compared to bio-lubricant. Also, the addition of ionic liquids has no significant effect on the WSD of PAO samples.

RJO with the addition of ionic liquid has outperformed all the other lubricants in terms of WSD under all operating conditions. Also, lower values of COF and wear can be seen for all bio-lubricants as compared to synthetic lubricant PAO. This enhancement in tribological behavior of bio-lubricants is because of its higher thermal stability which is caused by the high oleic acid concentration in it [43-46]. Oleic acid is a mono saturated fatty acid present in bio lubricants and it contributes to higher thermal stability of bio lubricants. Amongst bio-lubricants, RJO shows the best results with ionic liquid as compared to WCO and CSO.

4. CONCLUSION

In this research, a Vertical Universal Friction Testing Machine (MMW-1A) was used to study the effect of a phosphonium-based ionic liquid on the tribological performance of CSO, RJO, and WCO bio-lubricants and their results were compared with PAO. The tests were conducted at two loads and operating temperatures. Several conclusions regarding the performance of bio-lubricants and their comparison with PAO can be made from this study. The study shows that bio-lubricants when compared with PAO showed better tribological performance. Moreover, the introduction of ionic liquid does not produce a positive effect on the tribological performance of PAO because of its incompatibility with non-polar lubricants. Additionally, bio-lubricants containing ionic liquid typically yield superior tribological outcomes.

It can be observed that at higher temperatures, bio-lubricants except for WCO with the addition of ionic liquids, performed better as compared to untreated lubricants. Moreover, this study indicates that ionic liquid demonstrated better performance when used as an additive to Rattan Jot Oil as compared to other lubricants.

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