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Assessment of the tribological performance of ionic liquid additive in engine oil for automotive application

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KEYWORDS

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ABSTRACT

Ionic Liquids (ILs) have been explored as possible additives in engine oil for optimizing the tribological performances of Internal Combustion engines. Several articles published in the recent past have discussed Trihexyltetradecyl phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate as a potential additive due to its high miscibility and low corrosive nature. This study focuses on the tribological behaviour of various concentrations of Trihexyltetradecyl phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate and ZDDP additives and their blends in mineral base oil and engine oil. Wear of cylinder sleeve–pin specimens was analysed using a reciprocating wear test rig. Further, surface morphology was analysed using SEM analysis. Addition of a 3% concentration of IL Trihexyltetradecyl phosphonium bis (2, 4, 4-tri-methylpentyl) phosphinate into engine oil demonstrated significant reduction in friction and wear.

1.0 INTRODUCTION

Lubricants are one of the most important components that influence the working of any machine with moving parts. From the basic manufacturing processes to the complex mechanical inventions, their usage is inevitable. Lubricants create a fluidic layer between the contact surfaces and reduces the friction. Various types of additives are added to the base stock of a lubricant in order to enhance its tribological as well as other properties (Li et al., 2018; Tang et al., 2014; Lee et al., 2009; Zhou et al., 2009)

In recent years, many literature published were exploring the potential of ionic liquids as a possible additive in non-polar engine oils. Ionic liquids are molten salts with boiling points below

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the normal boiling point of water. Low miscibility and the corrosive nature of ILs in non-polar oils have limited the use of ILs as possible lubricant additives. In the family of ILs, phosphonium based ILs stood out as a potential candidate as an engine oil additive due to its ability to offer several advantages over other groups of ILs, including but not limited to, higher thermal stability, lower viscosity, and higher stability in strongly basic or strongly reducing conditions.

Ionic liquids (ILs) are in essence, low melting point organic salts. Their characteristics such as negligible vapor pressure, high thermal stability, electrical conductivity and controllable physical and chemical properties have raised their potential to be a high-performance green lubricant. With these properties, they are being researched as a solution to many lubrication problems in engineering applications (Palacio and Bhushan, 2010). Some studies proved that ILs could be effective due to a molecularly ordered layer formed at a static IL-solid interface. Further formation of a low friction boundary layer is facilitated when surfaces are set in relative motion (Horn, Evans, and Ninham, 1988). Even so, these ordered layers are found to be disrupted in a rubbing contact and resulted in the missing of an effective slip plane (Atkin and Warr, 2007; Gebbie et al., 2013; Min et al.,2009; Perkin et al., 2010; Perkin, 2012; Smith et al., 2012).

In 2001, Ye et al. were the first to investigate the use of ILs as lubricants and propose ILs as an alternative to conventional liquid lubricants. Since then, the number of published papers on the topic has increased steadily. In the past few years, researches have shown that ILs possess properties like non-flammability, high polarity, non-volatility, high viscosity and thermal stability. Various potentials of ILs have been developed and introduced to function as part of industrial devices, equipment and machinery (Bermúdez et al., 2007; Dörr, 2012; Thuy Pham et al., 2009; Wasserscheid and Welton, 2007).

Due to the presence of ions, ILs have been shown to have influence in ready adsorption onto metal ions, which typically possess some form of charged layers. The reduction in friction and wear in boundary lubrication is due to the formation of relatively thick and low friction layers, which are thought to be because of surface adsorption and long alkyl chains (Somers et al., 2013). ILs have the ability to form an adequate lubricant layer which can provide higher load carrying capacity on the metal asperities during different lubricating regimes such as hydrodynamic, elastohydrodynamic and boundary lubrication. It also has the capability of supplying wide temperature range for cooling action, which is larger, compared to conventional fluid lubricants (Amiril et al., 2017).

Bowden and Tabor (Popov, 2010) proposed a mechanism illustrating the formation of boundary lubricating film at the interaction area. This contact mechanism had an effect on the viscosity levels of lubricants and their anti-wear and anti-friction abilities were well explained. According to Mendonça et al., 2012, chemical reactions occurring at molecular level has a prominent impact on the performance of IL lubricants on the interacting surfaces. The main chemical reaction involved is the atomic adsorption of ILs on the interaction area. Generation of mechanical and thermal energy during the boundary and mixed lubrication conditions boosts the rate of these chemical reactions (Somers et al., 2013). Heat energy is produced as a result of rubbing motion and leads to the formation of tribofilm in the interface territory (Zhang et al., 2009). Besides this, higher load carrying capacity and wear and friction reduction were achieved with the development of adsorbed molecules or films. The strong electrostatic interactions of ILs and metallic surfaces plays a vital role in this process (Jim enez et al., 2006; Palacio and Bhushan, 2010). Researches have shown that these layer formations are much faster than mineral water and oil. Covalent bonds are formed between IL molecules and metal substrates during the chemical process. This causes chemical reactions, which could lead to faster adsorption rate during metal-IL interaction (Brinksmeier et al., 2015; Davis et al., 2015; Guoet et al., 2015).

From the several investigations done on the capability of ILs as lubricant additives, maintaining an optimum lubrication performance level was found difficult. This was due to the corrosive attack of ILs on lubricated metal surfaces and low miscibility of ILs in the non-polar oils (Qu *et* al., 2009; Yu *et* al., 2012). Besides this, considering the large cost of ILs, they are being used only in smaller quantities than bulk quantities for engineering applications. Nevertheless, the overall cost of using ILs in real applications can be brought down by the multiple recycling of ILs after use (Plechkova *et* al. 2008). This cost efficiency factor of IL inspired further investigations into this matter. Anand *et* al. in 2016 performed a study on the interaction between ILs and prevailed additives in engine-aged lubricants and their tribological behavior of ring-liner tribo-system. The ionic liquids used were Trihexyltetradecyl phosphonium bis (2, 4, 4-tri-methylpentyl) phosphinate and Trihexyltetradecyl phosphonium bis (2-ethyl- hexyl) phosphate. The phosphonium based ILs used in this case helped in the recovery of tribological performance of aged lubricants towards the end of their service life.

Zinc dithiophosphates (also known as ZDDP) are among the most successful and effective additives ever invented for lubricant application due to its corrosion and oxidation inhibition capacity, low cost and multi-functionality. Introduced in over 60 years ago, these chemical compounds are still widely used and present in almost all the engine oil produced (Spikes. H, 2004).

From the literature, it was observed that research work related to analysing the tribological behaviour of the blended mix of IL and ZDDP with a fully formulated engine oil at various temperatures is limited. The present research work focuses on analysing the friction and wear behaviour of ionic liquid, Trihexyltetradecyl phosphonium bis (2, 4, 4-trimethylpentyl) phosphinate and ZDDP at various proportions when blended with mineral base oil and engine oil.

2.0 EXPERIMENTATION

2.1 Sample Preparation

Tribological studies were planned in such a way as to simulate the contact conditions in an engine where the piston ring makes contact with the cylinder sleeve. In a cylinder-piston assembly, while assembling, the ring is compressed slightly and placed inside the cylinder. The outer diameter of the dismantled ring will be more than the inner diameter of the cylinder sleeve. Hence when the piston ring and cylinder sleeve are cut to segments, the ring samples may not conform perfectly to the cylinder sleeve segments. To avoid this uncertainty and to ensure high contact pressures, a hemispherical tipped pin was used to make contact with the sleeve segment.

For preparing the sleeve samples, a 115 mm diameter cast iron cylinder sleeve that is used in IC engines was cut into 9 equal segments with a 40-degree profile angle for each segment. The sleeve samples were produced using a wire electrical discharge machine (Wire EDM) to achieve precise dimensions. To ensure a point contact, a hemispherically tipped pin was used as the counter facing body. The pin was machined from an EN31 cylindrical rod by turning operation. EN31 is an alloy steel rich in carbon and chromium and offers high hardness and abrasion resistance. The pin diameter was reduced to 10mm gradually and one of the two ends were formed into a hemisphere by turning operation. The pin and sleeve samples are shown in Figure 1.

Figure 1: Pin and cylinder sleeve samples after cutting and machining.

2.2 Test Lubricant Preparation

The test lubricant solutions were prepared using mineral base oil, Trihexyltetradecyl phosphonium bis (2,4,4-trimethylpentyl) phosphinate and Zinc dialkyl dithiophosphate (ZDDP). ZDDP is used here to investigate its interaction with IL. The IL for the present experiments was procured from Zigma Aldrich®. To mix the IL and ZDDP at an optimum concentration, the concentration of ZDDP that offers the minimum coefficient of friction and wear was determined first. Table 1 presents the properties of the mineral base oil SAE 30.

Table 1: Properties of the mineral base oil

Property	Standard	Value
Viscosity Index		97
Flashpoint ‘C	D-92	252
Sulphur,% mass	D-129	0.0178(T)
KV @ 100’C, cSt	D-445	10.72
KV @ 40’C, cSt	D-445	92.77
PMCC Flashpoint, ‘C	D-93	234
Pour point ‘C	D-97	-3

To conduct the experiments, lubricant blends were prepared with concentrations of the additives as given in Table 2. To obtain the desired concentration, a pre-calculated amount of additive was measured using a micropipette and poured into a premeasured quantity of base oil. This mixture was then stirred thoroughly to ensure a homogeneous mixture.

Table 2: Prepared lubricants with varying concentrations of the additives

Test set-I	Test set-II	Test set-III	Test set-IV
% of IL in base oil	% of ZDDP in base oil	IL and ZDDP in base oil	IL and ZDDP in engine oil
1	1	1% IL with 1% of ZDDP	
3	0.5	3% IL with 1% of ZDDP	
5	0.1	5% IL with 1% of ZDDP	

2.3 Experimental Setup

The experiments to analyze the friction and wear characteristics were performed on a reciprocating wear testing machine (Figure 2). The machine is provided with sample holders to fix the lower sample – cylinder sleeve segment and the reciprocating upper sample-pin as shown in Figure 3.

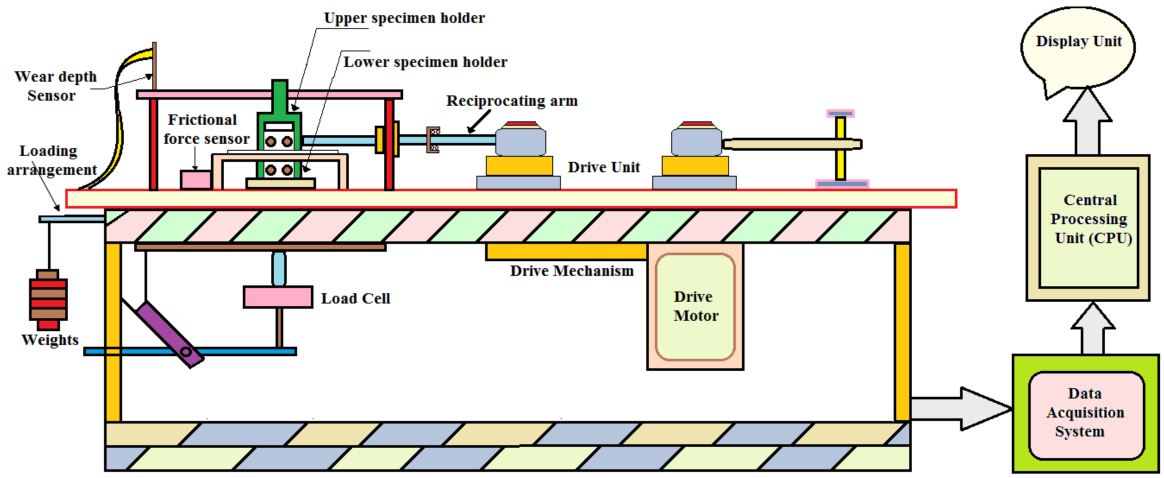


Figure 2: Schematic sketch of the reciprocating friction and wear testing machine.

The equipment is provided with various sensors for measuring wear depth, load, temperature and frictional force. A dead weight loading arrangement is used to apply the normal load acting on the upper sample. A heating coil positioned below the lower sample fixture controls the sample temperature. The frictional force is recorded by a pressure transducer. A linearly variable resistance transducer (LVRT) measure the cumulative wear of the upper and lower samples.

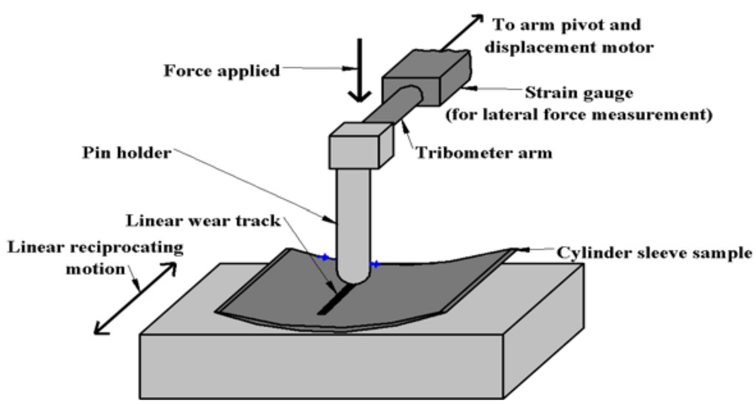


Figure 3: Sample setup on the wear testing machine.

The acquired data from the machine is processed using a computer with Winducom® software. In the experimental setup as shown in Figure 3, a linear wear track is produced due to the reciprocating motion. Table 3 presents the test parameters. The load 50 N was selected based on a previous research (Anil and Vasudevan, 2017) to nearly simulate an initial contact pressure of 2 GPa. The stroke length of 15mm was set to ensure the presence of boundary lubricated conditions at the reversal positions of sliding.

Table 3: Test parameters.

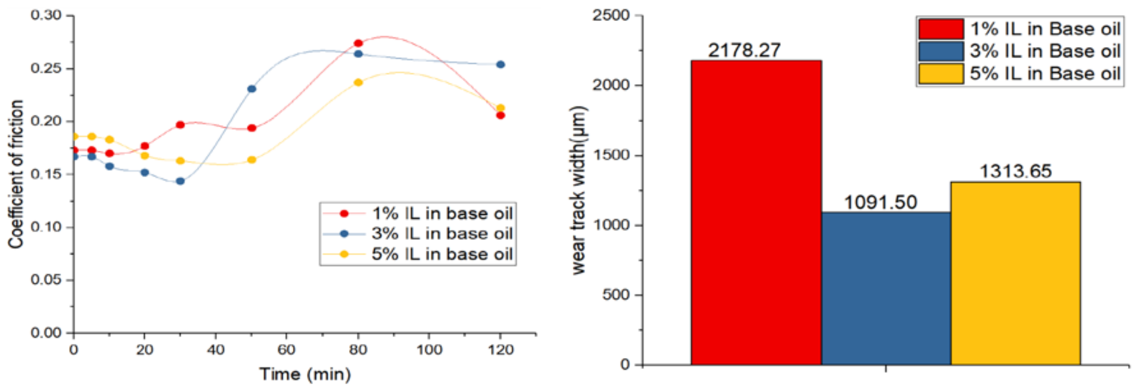
Parameters	Value
Load (N)	50
Temperature (°C)	100
Duration (min)	120
Stroke length(mm)	15
Oscillation frequency(Hz)	10

The tests were performed for a duration of 120 minutes with the four sets of blends (Test set-I, II, III and IV) as mentioned in Table 2. After completion of each test, the wear scar on the sleeve specimen was analyzed under a high-resolution digital microscope to determine the wear track width. This data was used to compare the wear track width obtained with various samples. Since the length of the wear track correspond to 15mm stroke length in the wear testing machine, the widths of the wear tracks were compared in order to determine the test solutions with the least wear.The coefficient of friction data was analyzed and comparative graphs were plotted. The surface of the tested specimens was analyzed using a Scanning Electron Microscope (SEM) and the composition of the material on the surface was examined by Energy dispersive X-Ray spectroscopy (EDS).

3.0 RESULTS AND DISCUSSION

3.1 Wear Tests with Lubricants with 1%, 3% and 5% of IL in Mineral Base Oil (Test set-I)

Figure 4 presents the variation of the coefficient of friction and wear track width obtained for various samples. It was observed that, the COF was the least for 3% IL lubricated sample in the initial stages. After about 30 minutes of sliding, the COF increased considerably. This can be explained by the breakdown of the mineral base oil. Without the presence of additives, mineral base oil has a short life. The breakdown occurs due to the breakdown of hydrocarbon chains. The behaviour of coefficient of friction is reflected in the wear track width also. After 120 minutes of running, wear track width was found to be least for 3 % IL in base oil. The trend is nearly the same for 5% IL also.

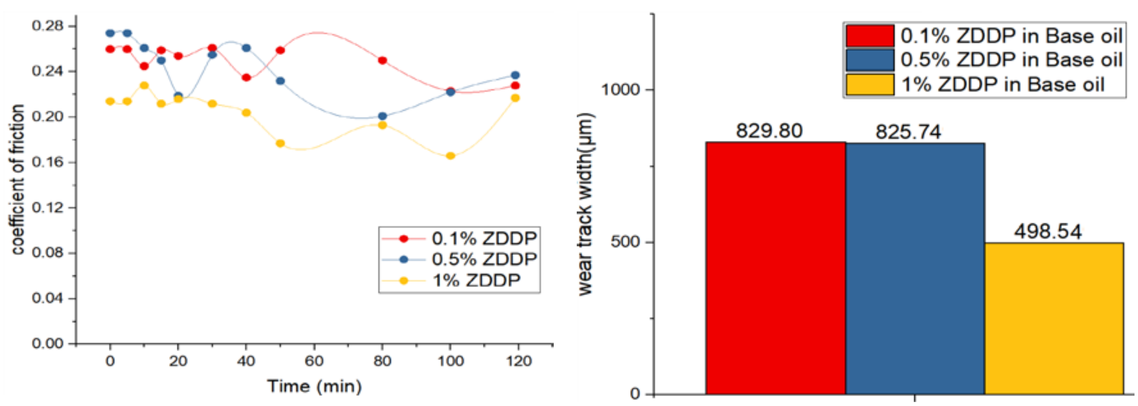


(a) (b)

Figure 4: (a) Comparison of COF of samples tested with 1%, 3% and 5% IL in base oil, (b) Comparison of wear track width of samples tested with 1%, 3% and 5% IL in base oil

3.2 Wear Tests with Lubricants with 0.1%, 0.5% and 1% of ZDDP in Mineral Base Oil (Test set-II)

Figure 5 presents the test results of Test set-II. The plot clearly shows that the least average coefficient of friction was obtained when 1% ZDDP was added in mineral base oil. The coefficient of friction was about 0.22 in the beginning and reduced progressively to 0.16 at the end of 120 minutes of sliding. Furthermore, the coefficient of friction values obtained with 0.1 % ZDDP blend exhibited values close to 0.26 initially and progressed with a fluctuating trend. For the 0.5% blend, it was found to highly fluctuating from the beginning. This fluctuating trend in coefficient of friction is due to the breakdown of the protective film (Qu et al., 2012). Wear track width also showed a minimum value in the case of 1% blend. However, the wear track width was nearly the same for 0.1 % and 0.5 % blends. The resemblance in wear track widths were also reflected in the coefficient of friction values for these blends.



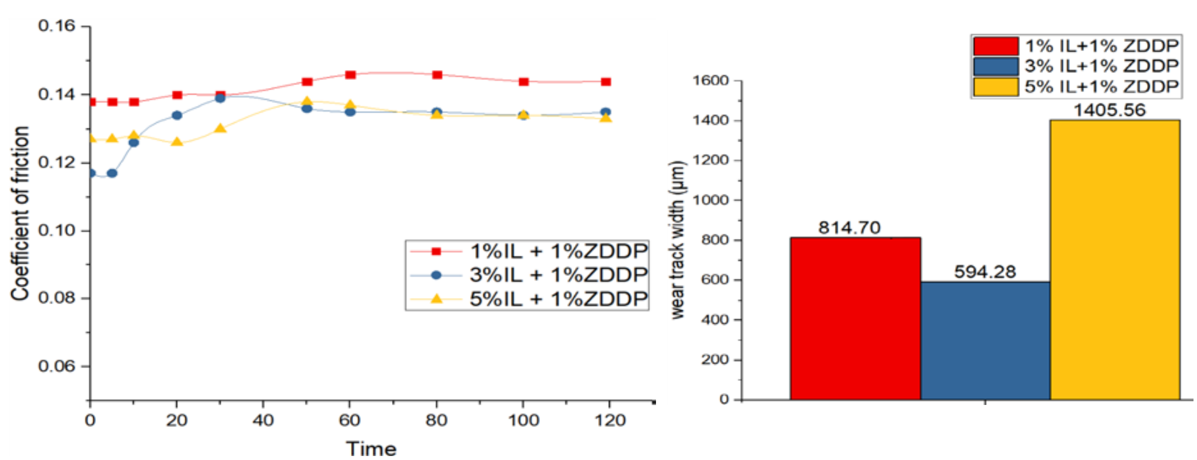
(a) (b)

Figure 5: (a) Comparison of COF of samples tested with 0.1%, 0.5% and 1% ZDDP in base oil, (b) Comparison of wear track width of samples tested with 0.1%, 0.5% and 1% ZDDP in base oil.

3.3 Wear Tests with the Mixture of 1% ZDDP and Varying Concentrations of IL in Base Oil (Test set-III)

Tests conducted with ZDDP in the base oil showed that 1 % exhibited the lowest wear and coefficient of friction. When tested as per Test set-I, it was confirmed that 3 % IL in base oil exhibited the least coefficient of friction and wear track width. Similarly when tested as per Test set-II, it was demonstrated that 1% ZDDP presented the best tribological behaviour. Furthermore, the interaction of these two additives have to be analysed in base oil and engine oil. For achieving this, blends were prepared in base oil with each having 1% ZDDP and 1%, 3% and 5% concentrations of IL as mentioned in Table 2 for Test set-III. Similar to the previous tests, the COF and wear track widths were compared.

From Figure 6, it may be noted that the initial coefficient of friction varied between 0.115 and 0.14 for the three blends. The coefficient of friction was found to be progressively increasing for the 1% IL blend with ZDDP and remained at 0.145 at the end of 120 minutes of sliding. 3% and 5% blends with 1% ZDDP exhibited a similar friction behaviour from 60 to 120 minutes. However, from the wear track width plot, it was observed that 3% IL exhibited the least width. Furthermore the 5% IL blend showed the maximum wear track width that is nearly 2.5 times that of 3% blend.

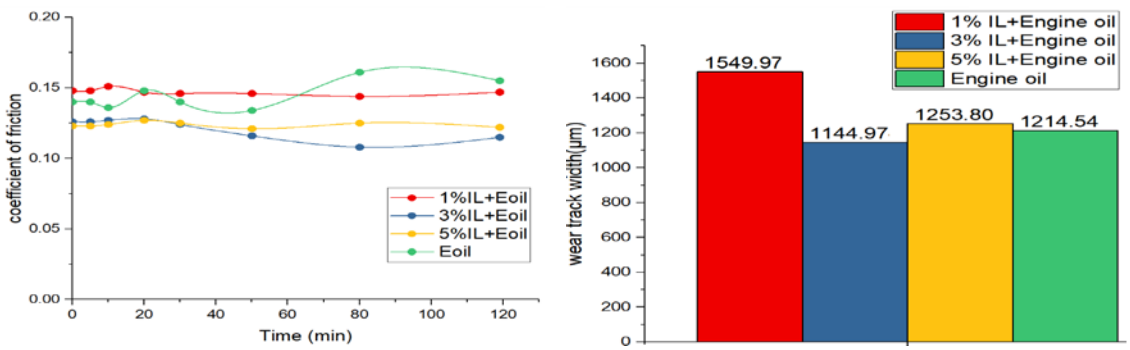


(a) (b)

Figure 6: (a) Comparison of COF values of specimens tested with blends of IL and ZDDP in base oil (b) Comparison of wear track width of specimens tested with blends of IL and ZDDP in base oil

3.4 Wear tests with IL in fully formulated engine oil (Test set-IV)

From the mineral base oil tests with different blends of IL and ZDDP, it was observed that 3% concentration of IL and 1% concentration ZDDP exhibited the best tribological performance. This has to be verified in fully formulated engine oil. For this, blends were prepared in fully formulated engine oil as presented in Test set -IV in Table 2. Tests were performed with these blends in engine oil as well as in unblended engine oil. The coefficient of friction data obtained showed a significant reduction with the 3% IL + Engine oil when compared to pure engine oil. This can be clearly observed from the Figure 7.



(a) (b)

Figure 7: (a). Comparison of COF values of specimens tested with blends of IL and engine oil, (b) Comparison of wear track widths of specimens tested with blends of IL and engine oil

Table 4: Comparison of wear track widths.

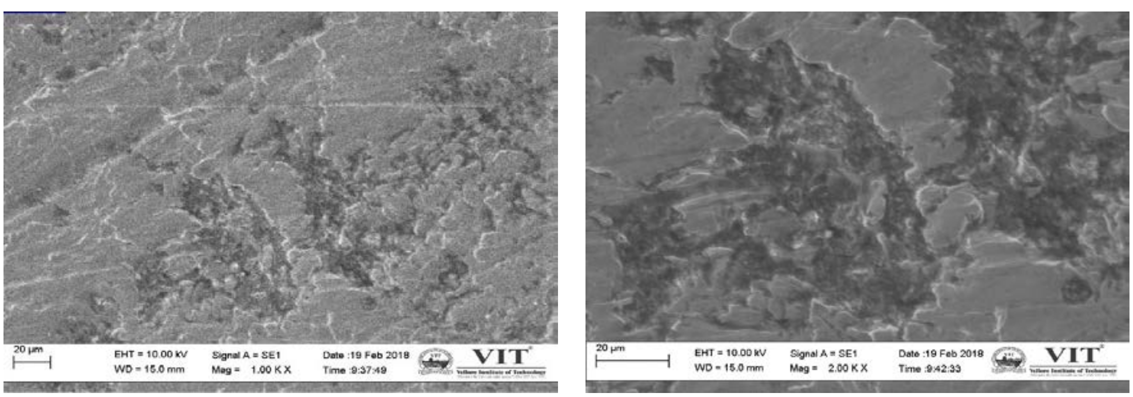
Test	Minimum coefficient of friction in the test sets at the end of 120 minutes of sliding	Minimum Wear track width (micrometers) in the test sets at the end of 120 minutes of sliding
Set-I	0.21	1091.50
Set-II	0.22	825.74
Set-III	0.13	594.28
Set-IV	0.11	1144.97

A comparison of the wear track width also confirms the findings of reduced wear width for 3% IL lubricated sample. However, the wear track width was found to be higher when compared to Tests with Set-I, II and III. The obtained values of wear track widths with various test sets are presented in Table 4. Even though the coefficient of friction obtained with 3 %IL and 1% ZDDP in engine oil was found to be low, the wear track width with this blend was found to be high (1144.87 micrometres). The same blend in base oil showed the least wear track width (594.28 micrometres) and coefficient of friction of 0.13. The wear track width of all the 13 samples varied between 2178.27 and 498.54 micrometres. The least value was obtained when tested with 1% ZDDP in base oil. The increase in wear track width for the Test set-IV should be analysed carefully. Ionic liquids have a tendency to cause chemical reactions on steel surfaces and cause chemical wear. Qu et al., (2012) have reported a synergy between ZDDP and phosphonium based IL in 5W-30 engine oil with reduced wear rate of nearly 70% when compared to PAO base oil and IL blend. In the present study with 3% IL and 1% ZDDP in engine oil, even though the coefficient of friction reduced by almost 45 %, the wear track width increased by about 5% than when blended with 3% IL in mineral base oil. This may be attributed to the corrosive nature of the IL. From the Figure 7, it can be seen that there is no fluctuating behaviour for the coefficient of friction. This indicates simultaneous occurrence of a steady coefficient of friction and progressive removal of material resulting in an increased wear track width.

3.5 Surface Analysis

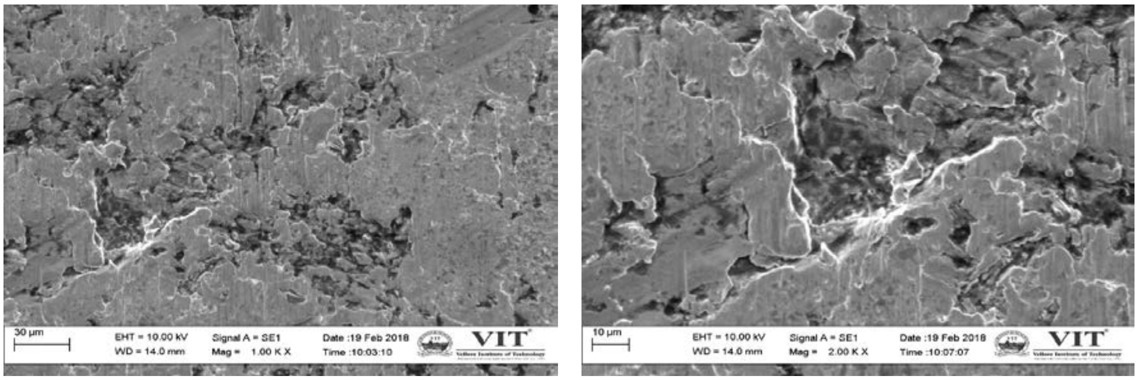
Figures 8-10 show the SEM morphological analysis of the wear scar on the surface of samples tested with 1%, 3% and 5% of IL and 1% ZDDP blends in mineral base oil. Figure 11 presents the unworn surface of the cylinder sleeve specimen. The honing marks can be clearly observed on the surface. The samples were observed under two magnifications, 1000X and 2000X. Figure 8 presents the samples with 1% IL lubrication, Figure 9 presents 3% IL lubricated samples and Figure 10 presents 5% IL lubricated samples.

From an initial observation at 1000X magnification, it is evident that all the samples had mild scuffing damage from the wear test. Material has been pulled away spontaneously from the surface similar to a spalling process. The counter facing pin used was hemispherically tipped. As the contact zone are is very small, it resulted in high initial contact pressures. High contact pressures along with sliding produced sufficient tractive forces to pull away the material from the surface. The flake like appearance much evident on the surface of 3% IL tested sample (Figure 9) indicates high contact pressures and traction. Furthermore, the mechanism of wear is a combination of mild adhesion as well as mild abrasion, even though the dominant mechanism is adhesion. Since the pin material was AISI 52100 steel and has a higher hardness compared to the cast cylinder sleeve sample, mild abrasion is seen on the 5 % IL tested sample (Figure 10). Surface films were not detected in any of the three samples.



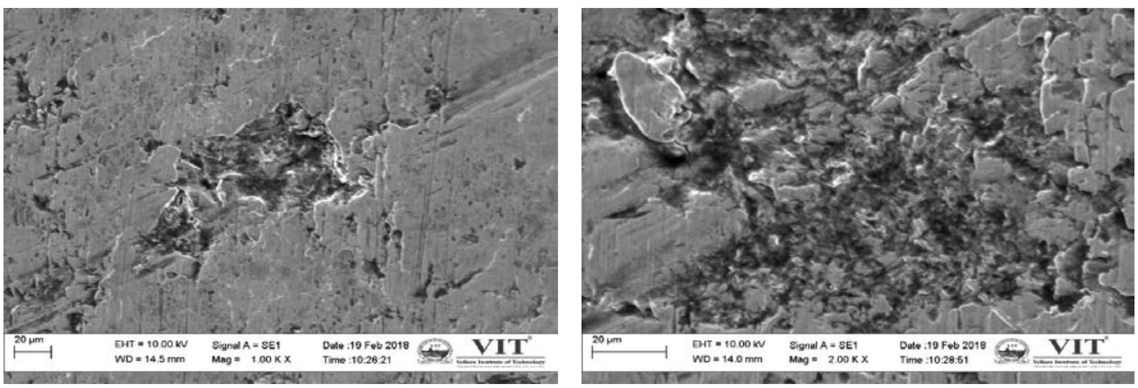
(a) (b)

Figure 8: SEM of the wear scar on the cylinder sleeve surface with 1% IL (a) 1000X (b) 2000X.



(a) (b)

Figure 9: SEM of the wear scar on the cylinder sleeve surface with 3% IL (a) 1000X (b) 2000X.



(a) (b)

Figure 10: SEM of the wear scar on the cylinder sleeve surface with 5% IL (a) 1000X; (b) 2000X.

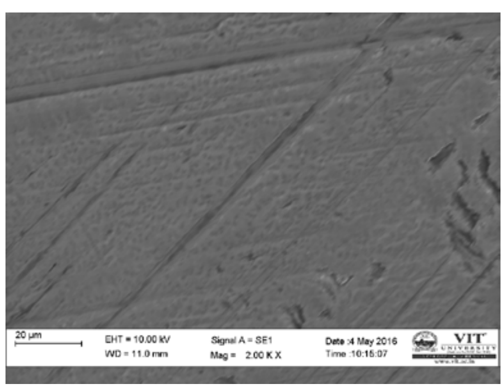


Figure 11: SEM image of the unworn surface of sleeve specimen at 2000X.

Table 6: Comparison of the EDS results.

Element	Weight % of Elements		
	1% IL Sample	3% IL Sample	5% IL Sample
C K	12.76	7.44	8.06
O K	7.62	4.31	5.47
P K	0.55	1.02	1.66
Fe L	78.75	82.03	80.50
Zn L	0.33	2.17	3.37
Si K	0.0	1.90	0.0
S K	0.0	1.14	0.93

Table 6 presents the EDS results of the samples tested with 1%, 3% and 5 %ILs. The presence of constituents of ZDDP and IL are observed on the wear track surface. The presence of P as well as Zn is found to be maximum in the 5% sample. The higher percentage of phosphorous may be due to the presence of the same in both IL as well as ZDDP.

4.0 CONCLUSIONS

The ionic liquid Trihexyltetradecyl phosphonium bis (2,4,4-tri-methyl pentyl) phosphinate was mixed with base oil at different weight percentages, viz. 1%, 3% and 5% by volume. Base oil blends with varying concentrations of Zinc dialkyldithiophosphate (0.1%, 0.5%, and 1%) were also prepared. These blends were tested individually and also in blends of specific combinations. One set of tests were conducted with engine oil blended with IL and ZDDP. The observations were that:

(a) The blend of 3% IL and 1% ZDDP in base oil exhibited a low coefficient of friction and wear track width compared to the other samples.
(b) The blend of 3% IL and 1 % ZDDP in engine oil also exhibited a low coefficient of friction without any fluctuations during the entire test. A good synergy was observed in this case with the IL and ZDDP.
(c) SEM analysis of the wear track surfaces revealed a combination of mild adhesive and abrasive wear when tested with IL and ZDDP in base oil. However, a tribofilm formation was not detected in any of the samples.

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