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LUBRICATING ABILITY OF A CHOLINE-AMINO ACID IONIC LIQUID AS NEAT LUBRICANT AND ADDITIVE TO A NON-POLAR OIL

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ABSTRACT

Although lubricants play an essential role in reducing wear and friction in mechanical systems, environmental issues persist. In the past decades, Ionic Liquids (ILs) have arisen as environmentally friendly alternatives to conventional lubricants and additives. ILs are low–volatile and non-flammable salts that possess low melting points (below 100 ºC). Their tunable properties, achieved by selecting the appropriate cation and anion, make them ideal candidates for different applications, including lubricants. In recent times, Protic Ionic Liquids (PILs) have attracted attention in the tribological community as a cost-effective alternative to conventional aprotic counterparts.

In this work, a choline-amino acid ionic liquid, derived only from renewable, biodegradable, and biocompatible products, was synthesized, and investigated as both neat lubricant and additive to non-polar oil. The lubricating properties of [CHO][GLY] were studied both as a neat lubricant and as a 1 wt. % additive to a polyalphaolefin (PAO) oil using a ball-on-flat reciprocating friction tester. AISI 52100 steel disks were tested against AISI 52100 steel balls using either [CHO][GLY] or the mixture of PAO+[CHO][GLY]. For comparison purposes, the commercially available base oil, PAO, was also tested. Preliminary results showed no major differences in friction between the lubricants used. Nevertheless, the addition of 1 wt.% to the PAO demonstrated a remarkable 30% reduction in wear on the steel disk. This encouraging improvement in anti-wear characteristics raises the potential advancement of lubrication technology with the choline-amino acid ionic liquid, coupled with its environmentally friendly nature. Energy-dispersive X-ray (EDX) spectroscopy, non-contact profilometry, and scanning electron microscopy (SEM) were used to study the worn steel surfaces and elucidate the wear mechanisms.

Keywords: Friction, Wear, Lubricants, Additives, Ionic Liquids

1. INTRODUCTION

Humanity, throughout history, has struggled with friction, looking for creative and innovative solutions to curb its impact on machinery and transportation. From lubricating using water and oils by ancient civilizations [1] to recent developments in tribology, the importance of reducing friction is becoming increasingly evident for both economic and environmental reasons. Studies from industrialized countries, including the influential "Jost Report" from the UK in 1966, have acknowledged how enhanced tribological practices can lead to significant reductions in energy consumption and financial losses [2]. Moreover, recent advances in tribology are motivated by the dual objectives of reducing economic losses due to friction and mitigating the adverse environmental impact of lubricants.

The effective use of lubricants and additives has been reported to be critical in improving the lubricity of contact surfaces, which consequently reduces the associated friction and wear [3,4]. Lubricants and lubricant additives decrease the direct surface-to-surface contact by forming a protective coating between the surfaces in motion, thus lowering the frictional resistance and the corresponding energy consumption [5].

Over the past two decades, ILs have attracted much attention for of their outstanding qualities, including, but not limited to, high solubility, recycling capabilities, and efficient acid-base catalytic processes which contribute to the formation of protective coating on surfaces [4]. Charged species are present in ionic liquids (ILs), which causes positive and negative charges to delocalize within their molecular structure [6,7]. These charges are electrostatically attracted to the metal surface forming an adsorption layer. The ability of ILs to create the thin film over surfaces that are undergoing sliding friction is the key advantage of using ILs as lubricants [8]. During this process, the ions in the IL are adsorbed on the metal surfaces in contact, forming layers that are closely spaced and have a certain configuration that significantly reduces wear and friction, especially in boundary lubrication [6,9]. Furthermore, ILs has shown effectiveness as neat lubricants and as additives [10], offering a means to enhance the lubricating properties of base oils. Thus, ILs are the best choice in lubricant additives compared to conventional additives to either polar or non-polar base oils like polyalphaolefin (PAO).

Since the onset of the use of ILs, aprotic ionic liquids (APILs) have been the focus of tribology research. This subset of ILs has been found to be costly due to their complex synthetic routes. In addition, the presence of halogen components in their structure causes corrosion of surfaces and toxic species when subjected to moisture [4]. However, another subset of ILs, known as protic ionic liquids (PILs), have easy synthesis process and can be designed to be environmentally friendly by avoiding halogen and other undesirable elements in their structure [11,12]. With the growing demand for sustainable and environmentally friendly, halogen-free lubricants, research on PILs as additives and neat lubricants is drastically increasing.

Following the Zwitterion structures of amino acids which are essential components in biological processes, these molecules have both carboxyl and amino groups, which permits them to operate as both anions and cations. Their molecular structures have a wide range of polar, non-polar, and aromatic functional groups, which give them their versatility for participating in a wide range of chemical reactions, thereby enabling diverse biological functions and potential applications in various fields, including lubrication technology. Like ILs, amino acids create thin films through surface adsorption that lessen friction [13]. Besides preventing surface corrosion [14], they also have ability to change lubricant's viscosity which impacts the lubricant's flow and film thickness [15].

On the other hand, choline, an important nutrient in many biological processes, can be used as cation in PILs. The charges in choline amino acid PILs associate with the charges on the metal surface resulting into formation of the ordered adsorption layer [16]. Understanding the molecular and structural characteristics of choline and amino acids is important for knowing how they affect lubrication. Their dipolar property helps in how they interact with metal surfaces, which makes them better lubricants.

Non-polar oils are an important class of lubricants widely used in industrial and automotive applications. These base oils are mostly composed of long hydrocarbon chains and are characterized by their low polarity and non-reactive nature. In lubrication, non-polar oils help in dissipating heat, preventing wear, reducing friction, and eliminating pollutants from entering gaps in tightly fitted surfaces [17]. By creating barriers against moisture and corrosive materials, they provide corrosion protection [18]. However, for several crucial reasons, additives are essential to maximizing the performance of non-polar oils in lubricating applications, such as polyalphaolefin (PAO). Even though non-polar oils have natural lubricating qualities, they frequently need to be improved to withstand the demanding requirements of contemporary machinery and working environments. Through the formation of boundary layers or protective coatings on metal surfaces, additives efficiently reduce wear and friction and increase the lubricity of non-polar oils. Furthermore, additives are essential for improving non-polar oils' thermal stability since they lessen the impact of oxidation and thermal deterioration at high temperatures [19]. Antioxidant and anti-corrosion additives prevent non-polar lubricants from degrading, maintaining their lubricating effectiveness and prolonging equipment life by preventing oxidation and corrosion.

In this work, a choline amino acid protic ionic liquid derived from the amino acid Glycine and choline is studied. Its performance both as a standalone lubricant and as an additive to a PAO-based non-polar oil, is assessed under steel-steel contact.

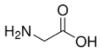
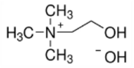
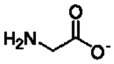
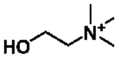
2. MATERIALS AND METHODS

2.1 Synthesis

Choline glycine PIL was synthesized in our laboratory using the following procedure: equal molar amounts of glycine amino acid and choline hydroxide (CHO) were combined in a round-bottomed flask with a magnetic stir bar and distilled under reduced atmosphere at 125–135°C in an oil bath to remove water from the samples using a Dean-Stark apparatus. The resultant PIL was subjected to bulb-to-bulb distillation using a Kugelrohr apparatus to remove any residual water. The molecular structure and code used in this study are shown in Table 1.

In this research, a readily available wind turbine gear lubricant, Mobil SHC XMP 320, was used as the base oil. This oil has a polyalphaolefin base in its formulation with its own additives. For the rest of this paper, this commercially available polyalphaolefin-based oil will be referred to as PAO. When the choline glycine PIL is used as an additive, one weight percent of the synthesized PIL was mixed with the non-polar oil PAO using a magnetic stir bar for 30 minutes followed by 30 minutes ultrasonication to form a homogeneous mixture. ILs are often efficient in lubrication enhancement even at very low concentration. One percent by weight of the additive can achieve a substantial improvement in base oil's performance.

TABLE 1: MOLECULAR STRUCTURE OF GLYCINE PROTIC IONIC LIQUID

IL code	Chemical		PIL	
	Acid	Base	Anion	Cation
[CHO][GLY]				

2.2 Ionicity and Dynamic Viscosity

To investigate the influence of the ionicity of lubricants on their tribological behavior, the conductivity of the PIL and mixture was measured using the Orion Star^{TM} A212 Conductivity Benchtop Meter. These measurements were taken at room temperature, 40°C, 70°C, and 100°C. Dynamic viscosity tests were also executed to evaluate the PILs' flow features and their influence on the lubricating behavior. These tests were conducted using Discovery Hybrid Rheometer with TRIOS software installed in computer for instrument control and analysis. Measurements were taken at different temperatures to account for temperature-dependent differences in viscosity.

2.3 Thermal Analysis

For thermal stability analysis, thermogravimetric analysis was performed using TA Instruments Q500 on the PIL, base oil, and the mixture of the base oil and 1 wt.% PIL. Under a nitrogen atmosphere, the lubricants were heated from 10°C to 600°C with a temperature ramp of 10°C per minute to simulate the process of thermal degradation and oxidation. Each lubricant's onset decomposition temperature was determined.

2.4 Tribological Tests and Wear Measurements

Experiments to investigate friction were conducted at room temperature with the use of a reciprocating ball-on-flat friction tester. Over time, Instantaneous friction coefficient was recorded for each run, and the average data was recorded. The length of the sliding stroke was 3 mm and one hour was the test period. 52100 steel balls of diameter 1.5 mm were sliding on 52100 steel disks at a frequency of 5 Hz for this study, yielding a sliding distance of 108 m. A 3 N normal load was tested on each lubricant, equivalent to a maximum contact pressure of 2.4 GPa. Under the experimental conditions studied, the sliding contacts are in the boundary lubrication regime. Each test began with the addition of 2 ml of lubricant. Each condition was tested for each lubricant at least three times and the average friction coefficient value was obtained. Isopropyl alcohol was used to clean the specimen both before and after each test.

After that, the wear marks on used disks were examined and the width of the wear track calculated. Each track was measured 30 times, and the average value was calculated. Wear volume of the disks was computed using the equations (1) and (2) from [20]. The examination of the wear marks and the taking of the images of the used steel balls were both done using Olympus BH-2 optical microscope.

$$V_f = L_s\left[{R_f}^2 arcsin\left(\frac{W}{2R_f}\right) - \frac{W}{2}\left(R_f - h_f\right)\right] + \frac{\pi}{3}{h_f}^2\left(3R_f - h_f\right) \tag{1}$$

$$h_f = R_f - \sqrt{{R_f}^2 - \frac{W^2}{4}} \tag{2}$$

2.5 Surface Characterization

Tescan-Bruker Vega 3 scanning electron microscope (SEM) with energy dispersive X-ray spectrometer (EDX) was used to analyze the wear surfaces of the steel disks and the surface interactions between the lubricants and the disks. For 3D wear track profiles, a NANOVEA PS50 3D profilometer was also employed.

3. RESULTS AND DISCUSSION

The graph in Figure 1 illustrates the variation of dynamic viscosity of the three lubricants – PAO, [CHO][GLY], and PAO+[CHP][GLY] – with temperature. There is a consistent tendency of the viscosities to decrease as temperature rises. Precisely, over the whole temperature range, [CHO][GLY] consistently exhibited the highest viscosity of all the lubricants. Interestingly, adding 1 wt. % of PIL to PAO, slightly decreased the viscosity of PAO at room temperature and 100°C, but increased at 40°C (Table 2). In any case, the difference in viscosity among the three lubricants is minimal. Furthermore, since all three lubricants are operating in the boundary regime of lubrication, characterized by conditions where the lubricant film is not thick enough to separate the direct contact between the sliding surfaces, differences in viscosity will not directly affect the friction behavior. In the boundary lubrication regime, other factors such as the lubricant's chemical composition to form adsorbed layers or its affinity with the surfaces play more crucial roles in reducing friction [21].

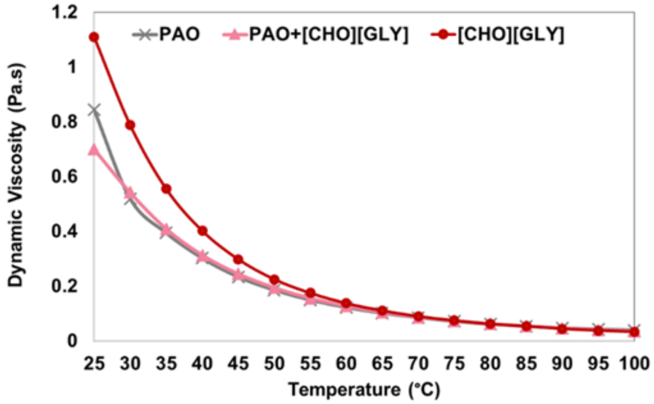


FIGURE 1: DYNAMIC VISCOSITY OF THE LUBRICANTS AS A FUNTION OF TEMPERATURE.

TABLE 2: DYNAMIC VISCOSITY OF PAO, PAO+[CHO][GLY] AND [CHO][GLY] AT DIFFERENT TEMPERATURES.

Dynamic Viscosity (Pa.s)			
	25°C	40°C	100°C
PAO	0.8451	0.3023	0.0389
PAO+[CHO][GLY]	0.7498	0.3114	0.0358
[CHO]GLY]	1.1091	0.4023	0.0340

Figure 2 shows the temperature dependence of electrical conductivity of the three lubricants. The electrical conductivity of [CHO][GLY] is significantly affected by temperature, displaying a non-linear dependence. As shown in the figure, there is a smooth increase from room temperature to 40°C, followed by a rapid change in conductivity after this point, reaching a maximum value of 2800 μS/cm at 100°C. This rise in ionic conductivity with temperature is anticipated due to the heightened mobility of the ions [3]. As expected, the non-polar based oil (PAO) exhibited negligible electrical conductivity.

Although the addition of the PIL to the base oil was anticipated to increase its conductivity [22], the amount added was not sufficient to have a significant effect. Consequently, the electrical conductivity of the PAO and PIL mixture remained negligible within the studied temperature range.

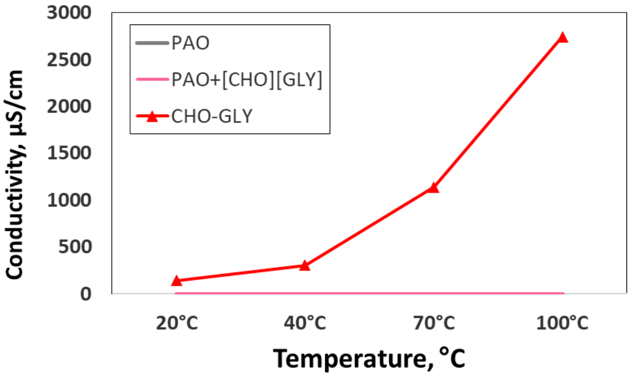


FIGURE 2: CONDUCTIVITY OF PAO, PAO+[CHO][GLY] AND [CHO][GLY] AT DIFFERENT TEMPERATURES.

One of the key factors in deciding the type of lubricant suitable for specific applications is its thermal stability. In this study, thermogravimetric analysis (TGA) findings for examining the thermal degradation behavior of three lubricants is illustrated in Figure 3. For each lubricant, the onset temperatures — a measure of the temperature at which degradation starts — are shown in Table 3.

From Figure 3, all three lubricants exhibited thermal stability well above 100 °C, the boiling point of water, signifying that the content of water is negligible. The amino acid-based ionic liquid, [CHO][GLY], exhibited an onset decomposition temperature of around 181°C, consistent with previous findings [23]. The short length of the glycine anion alkyl chain was expected to result in lower thermal stability compared to other choline PILs with different amino acids as anions [23].

The stronger intermolecular forces present in the hydrocarbon-based molecules of PAO led to higher thermal stability (Tonset ≈270°C) compared to [CHO][GLY]. The addition of 1 wt.% of the PIL to the base oil resulted in a slight increase in thermal stability.

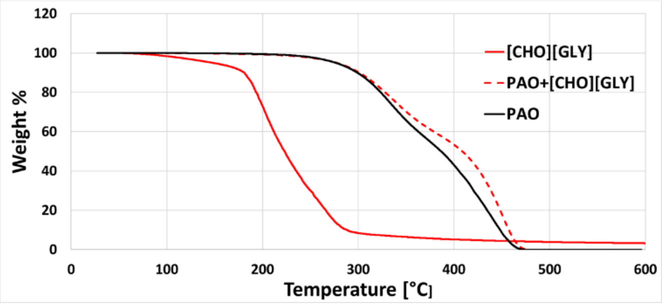


FIGURE 3: TGA CURVES OF PAO, PAO+[CHO][GLY] AND [CHO][GLY].

TABLE 3: ONSET TEMPERATURE OF PAO, PAO+[CHO][GLY] AND [CHO][GLY].

Ionic Liquid	Onset Temperature [°C]
PAO	271
PAO+[CHO][GLY]	271
[CHO][GLY]	181

The average values of coefficient of friction of the base oil, neat [CHO][GLY], and PAO+[CHO][GLY] are shown in Figure 4. In addition to the average values of friction, the change in the friction coefficient over the sliding distance for one of the runs for each lubricant can be found in Figure 5. From Figure 4, it can be observed that under the experimental conditions studied, no significant differences were noted in the average friction coefficient among the lubricants.

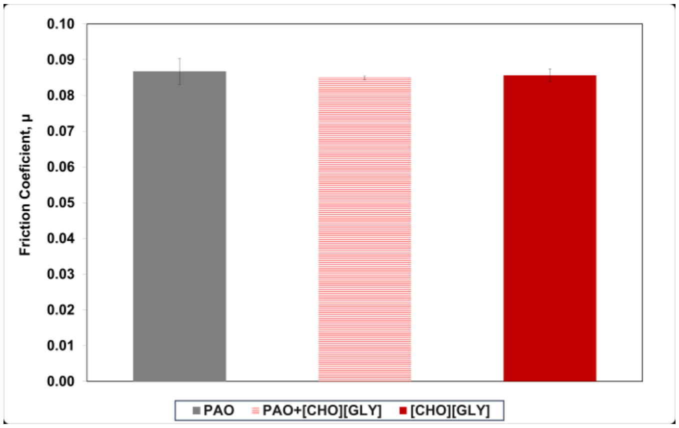


FIGURE 4: AVERAGE FRICTION COEFFICIENT AFTER TESTS USING PAO, POA+[CHO][GLY] AND [CHO][GLY].

Although in general, no major differences were found between the lubricants in the friction behavior over the sliding distance (Figure 5), when PAO was used as a lubricant, lower friction values were observed at the beginning of the test, which increased after approximately 20 meters and maintained an upward trend for the remainder of the test. Conversely, when neat [CHO][GLY] was used as a lubricant, the test started with higher friction values and exhibited a downward trend. The mixture displayed more consistent friction values throughout the test.

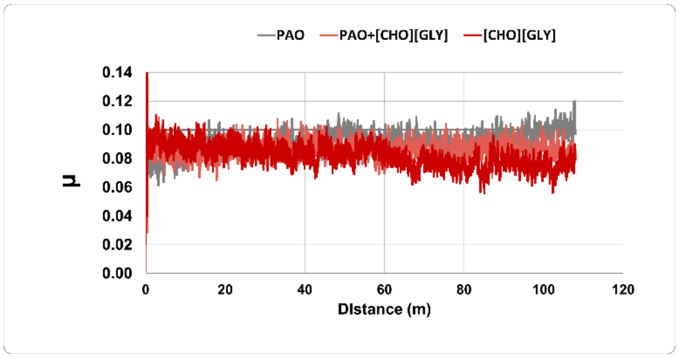


FIGURE 5: FRICTION COEFFICIENT VARIATION WITH SLIDING DISTANCE.

The average wear volume of the wear tacks after testing with the three lubricants is shown in Figure 6. No wear was observed on the steel balls for any lubricant under the experimental conditions tested as observed in the optical images in Figure 7. From Figure 6, the highest value of volume of the wear on the steel disks were obtained when the lubricant used was neat [CHO][GLY]. When PAO was used as the lubricant, the average wear on the steel disk was 2.26 x10^{-3} mm^3, which is approximately half of the wear volume obtained when [CHO][GLY] was used as the lubricant. Furthermore, the addition of 1 wt.% of [CHO][GLY] resulted in a further reduction of wear volume on the steel disks. The mixture displayed superior anti-wear properties, reducing wear volume by nearly 30% compared to PAO and an impressive 64% compared to using neat [CHO][GLY].

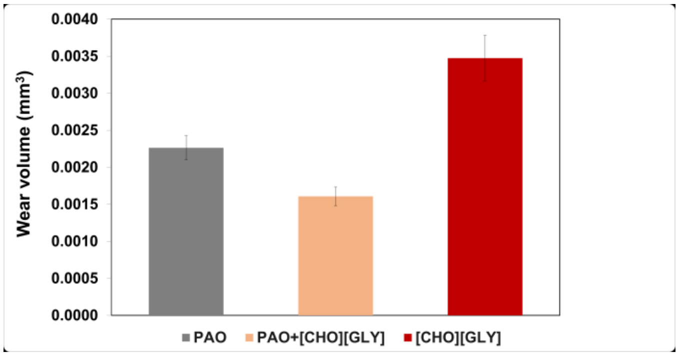


FIGURE 6: STEEL DISKS WEAR VOLUME AFTER TESTS USING PAO, PAO+[CHP][GLY], AND [CHO][GLY]

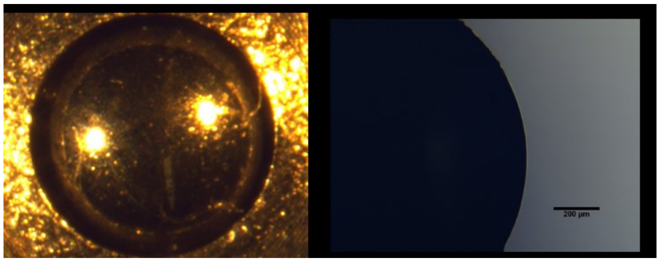
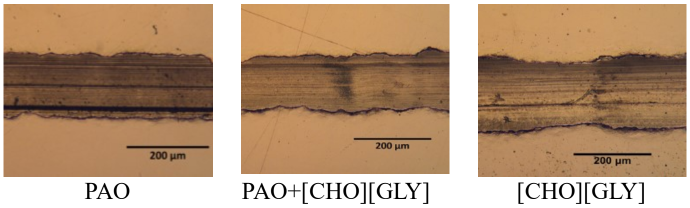


FIGURE 7: STEEL BALL IMAGES FOLLOWING A TEST WITH [CHO][GLY] LUBRICANT.

The images of the worn steel disks after the frictional tests taken by an optical microscope are displayed in Figure 8. These images not only illustrate that the wear track on the steel disk after a test lubricated with neat [CHO][GLY] is wider, but also reveal that the wear mechanisms are similar for all three lubricants. There is a significant abrasive component observed, particularly for PAO and [CHO][GLY], along with materials formed due to plastic deformation along the borders of the wear tracks.



PAO PAO+[CHO][GLY] [CHO][GLY]

FIGURE 8: STEEL DISKS WEAR TRACK IMAGES.

The 3D and 2D profiles in Figure 9 reveal that the wear track associated with neat [CHO][GLY] is not only wider but also deeper. Additionally, it is evident in this figure the significant wear reduction achieved with the addition of only 1 wt.% [CHO][GLY], where the wear track is not only smaller but also more uniform.

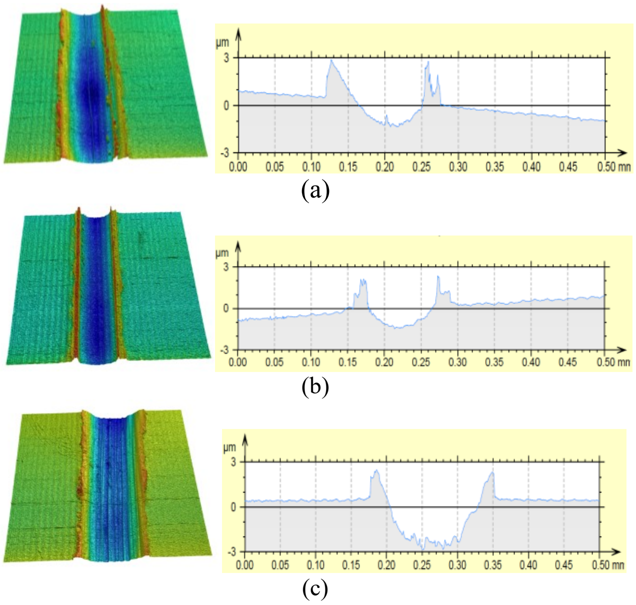


FIGURE 9: 3D AND 2D WEAR TRACK PROFILES ON STEEL DISKS FOLLOWING LUBRICATION USING (a) PAO, (b) PAO+[CHP][GLY], AND (c) [CHO][GLY].

To gain deeper insights into the wear mechanisms associated with the use of neat choline amino acid PIL as a lubricant or additive to PAO, SEM images of the wear tracks (Figure 10) and EDS spectra in and out of the wear tracks were obtained (Figure 11).

SEM images confirm the presence of abrasive wear, evident by the ploughing marks along the sliding distance, particularly pronounced when the test was lubricated with [CHO][GLY]. Under higher magnification, the wear track lubricated with [CHO][GLY] also exhibited micro-cracks characteristic of a fatigue component and some dark spots, suggesting corrosive wear when this neat choline amino acid PIL was used as a lubricant.

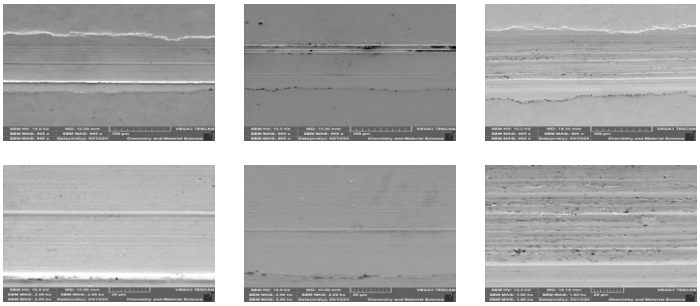

PAO PAO+[CHO][GLY] [CHO][GLY]

FIGURE 10: SEM WEAR TRACK IMAGES AFTER TESTS LUBRICATED WITH PAO, PAO+[CHP][GLY], AND [CHO][GLY].

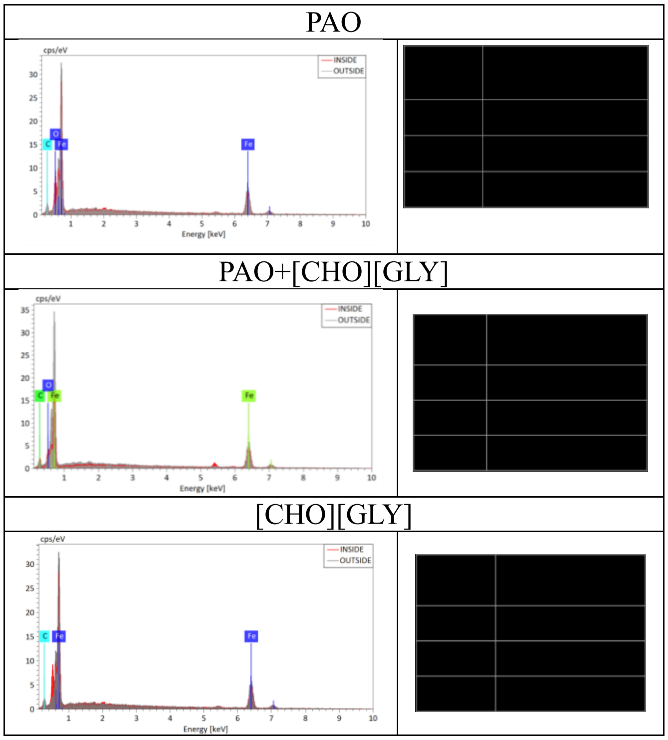


FIGURE 11: EDS SPECTRA INSIDE AND OUTSIDE OF THE WEAR TRACKS FOLLOWING TESTS WITH PAO, PAO+[CHP][GLY], AND [CHO][GLY] LUBRICANTS.

EDS analysis inside and outside of the wear tracks confirmed higher amounts of carbon inside the wear tracks when [CHO][GLY] was present in the lubricant, indicating that a tribolayer rich in carbon [24–26] formed on the surface of the disks. The significantly higher carbon content on the surface of the worn steel when [CHO][GLY] is used as a neat lubricant suggests that this PIL, when used alone, promotes detrimental tribocorrosion processes, accelerating the wear process. However, when this PIL was used as an additive to PAO, just 1 wt.% was sufficient to form a protective carbon- and oxygen-rich tribolayer, preventing the tribocorrosion reactions observed when [CHO][GLY] was used as a neat lubricant. The higher quantity of oxygen inside the wear track with PAO is used as a lubricant also suggests the formation of a less effective oxygen-rich protective tribolayer.

4. CONCLUSION

A renewable and biodegradable PIL has been investigated both as a neat lubricant and as a 1 wt.% additive to a non-polar oil under steel-steel contact. From this study, the following deductions can be made:

- The electrical conductivity of [CHO][GLY] increased with temperature following a non-linear dependence. The addition of 1 wt.% of [CHO][GLY] to PAO oil was not sufficient to change the non-polar character of this base lubricant.
- No substantial differences were noted in the average friction coefficient among the three lubricants.
- The addition of 1wt.% to PAO reduced wear volume by almost 30% compared to PAO alone. This reduction in wear volume may be attributed to the formation of a carbon- and oxygen-rich tribolayer when [CHO][GLY] is used as an additive.
- When neat [CHO][GLY] was used as a lubricant, detrimental tribocorrosion processes were observed, accelerating the wear process.
- An adhesive component of wear, characterized by plastically deformed materials at the edges of the wear tracks, along with an abrasive wear component, were observed across all three lubricants. The abrasive component of wear was more evident when neat [CHO][GLY] was used and was also accompanied by fatigue cracks and small spots of corrosion wear.

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