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TRIBOLOGICAL EVALUATION OF THE PERFORMANCE OF TRIMETHYLOLPROPANE ESTER BIOLUBRICANTS BASED ON FATTY ACIDS FROM SUNFLOWER OIL

Petrică TURTOI, Julien DELCUZE, Ionuț-Răzvan NECHITA, Alice MARINESCU

Abstract: *The efforts for finding a solution for environmental sustainability issues have prompted researchers to look for alternative resources for lubricant oils. Bio-based lubricants were proven to have certain advantages, such as being less expensive, showing good lubricating properties, and most importantly, being highly bio-degradable. Trimethylolpropane esters (TMPEs) are someof the most commonly used polyols in the production of bio-based lubricants, due to their thermal stability. Therefore, the current study follows the tribological evaluation of the performance of a single non-additivated TMPE produced from sunflower oil fatty acids. Tribological tests were carried out in different loading conditions, using a four-ball tribo-tester. The coefficient of friction, the variation of wear with the applied load, as well as the seizure load, were determined. The results showed relatively good anti-wear properties for the tested TMPE. A comparison of the results with similar ones found in the literature was made.*
Key words: *Biolubricant, Trimethylolpropane Ester, Wear, Four-Ball Tester, Fatty Acid.*

1. INTRODUCTION

Biolubricants are an attractive alternative to petroleum-based lubricants. The increase in environmental pollution caused by many synthetic substances, (including lubricants), facilitates the use of biolubricants in applications such as chainsaws, lawn mowers, and other agricultural equipment. Nevertheless, the EU policies for environmental concerns have a strong influence on bio-industry related decisions. Now, biolubricants represent a very small part of the global lubricant market, with over 56% being used in the automotive industry in 2015 [1]. Renewable sources are preferred when it comes to biolubricants, with vegetable oil presenting great potential.

Due to their strong interactions with contact surfaces, especially metallic ones, vegetable oils can act as anti-wear additives and friction mitigators. Even at relatively severe regimes [2] the long molecular chains of fatty acids have the ability to adhere and hold on to surfaces in contact. Experimental results [3] have shown that the composition of fatty acids contributes to a better lubricity and has an increased efficiency in wear reduction, when compared to mineral and synthetic oils.

Even though biolubricants offer several advantages over petrol-based lubricants, they are not a "one size fits all" solution and may not be suitable for all applications or industries. Hence, they might not work well with all equipment varieties, particularly those that integrate seals or other components made of synthetic materials, as these materials are typically more compatible with petrol-based lubricants.

Thus, it is necessary to consider the limitations of the lubricant before selecting it for a specific application. Another possible issue that might arise is the compatibility of biolubricants with petroleum-based environments, since biolubricants are biodegradable and do not persist in such environments. The downside of this aspect is the limited usage of biolubricants, resulting in a shorter shelf life than petrol-based lubricants, due to their biodegradability. Owing to the costs of renewable source materials and their processing, biolubricants could also be slightly more expensive than petrol-based alternatives.

Despite these mild inconveniences, biolubricants compensate by being, in most cases, non-toxic and safe for usage in sensitive environments, such as food processing plants and marine ecosystems. Substituting traditional petrol-based lubricants with biolubricants allows companies to lessen their environmental footprint. Thus, by making this switch, companies can contribute to reducing greenhouse gas emissions, decreasing dependence on finite fossil fuel resources, and minimizing the risk of environmental contamination from petroleum-based products.

For the tribological analyses performed within this paper, a trimethylolpropane ester (TMPE) biolubricant derived from sunflower waste vegetable oil was used. The development of plant-based TMPE lubricants targets different applications, such as food-safe lubrication, engine oils, metal working fluids, or hydraulic oils. Sunflower oil is biodegradable, readily available, eco-friendly, and renewable. Due to its widely spread availability and its role in human nutrition, substantial waste is generated daily as a by-product of its usage. The literature contains several experimental studies that assessed the performance of crude sunflower oil using four-ball testing machines [4][5].

In order to increase the potential of TMPE-based lubricants, surface additives multifunctional additives, and other solid nano particles [6][7] can be added to their composition. Information regarding the tribological characteristics of TMPE biolubricants, as assessed using the four-ball machine, is available in the literature. For a comprehensive understanding, it is important to consider the performance of base oils and alternative esters. More results can be found when the bibliographical study is extended towards other biolubricants, such as rapeseed oil, palm oil and soybean oil.

The tribological behavior of lubricants based on rapeseed oil and two nano additives (ZnO and TiO_2) was evaluated in [8][9]. The results proved that the inclusion of additives reduced the WSD; however, an increase in the COF was observed as well. Arumugan et al. [10] conducted tribological investigations which showed that the pentaerythryl ester of rapeseed oil exhibited a better lubricity behaviour compared to a synthetic grade compressor oil. Guglea et al. [11] analysed coarse rapeseed oil with a 1% hexagonal boron nitride additive with no significant results with respect to the COF, but Swith a reduction of the WSD. Georgescu et al. [12] studied the potential of rapeseed oil reducing extreme pressures. They measured the seizure load, which was lower when compared with a transmission oil.

Oil Palm *(Elaeis oleifera)* thrives in hot, humid tropical regions, being mostly produced and exported by Malaysia. Its main derived product – palm oil – is a good source of fatty acids. Hamdan et al. [13] present a tribological characterization using Lateral Force Microscopy of palm oil based TMPE used as boundary lubricant. Their findings have shown that it could act well as a wear reducer. Zulkifli et al. [14] studied wear prevention characteristics of a palm oil based trimethylolpropane ester, used as additive for an engine lubricant. The authors found that an addition of up to 3% of Palm oil TMPE decreases the maximum WSD and reduces the COF up to 30%. Hussain et al. [15] presented experimental results and proposes a model for ranking biolubricants based on their performance and properties. Yunus et al. [16] evaluated experimentally both palm oil and palm kernel oil based TMPEs. For both oils, oxidative stability was observed, being comparable to other high oleic base fluids. An improvement in wear was also found.

Nettlespurge (*Jatropha*) seeds contain 30–40 % oil and the crude oil contains around 14 % free fatty acids [17]. Talib and Rahim [18] studied the performances of crude jatropha oils, methyl ester and TMPE, used as a sustainable metalworking fluid. Ruggiero et al. [19] investigated the tribological performance of a methyl ester and two plant seed-based oils (jatropha oil and hydrotreated rapeseed oil) using a ball-on-flat reciprocating tribometer. The COF for the ester and jatropha oil was in the range of 0.11-0.14, and a lower wear was measured for rapeseed oil.

Soybean is rich in oil and protein, and it is used mainly as a food supplement; it has also been observed to have good potential for biolubricants production. Guru et al. [20] proved that PTFE and MoS_2 improve the tribological performance of the base soybean oil. Cristea et al. [21] presented results obtained from testing the soybean oil with nano graphite additives on a four-ball machine. The COF increased for the additivated oil, but no evident dependency on concentration and test conditions was noticed.

Results were also found for cottonseed oil methyl ester [22] and mastwood (*Calophyllum Inophyllum*) based TMPE [23]. For both, the results on the four-ball machine showed a lower WSD when compared to commercial lubricants - petroleum diesel, respectively.

The current paper targets to assess the performance of a TMPE biolubricant using the four-ball machine; the coefficient of friction (COF), wear scar diameter (WSD) and seizure load were the parameters of interest of the current study, since they could be easily compared to other similar results from the literature. A series of standards for oil lubricants (ASTM D4172 and ASTM D5183) were used to design the tests. Specific conditions for envisioned applications were considered (chainsaws, lawn mower, etc.).

2. EXPERIMENTAL SETUP

2.1 Trimethylolpropane ester oil (TMPE)

Many studies have been conducted to evaluate the tribological performance of TMPEs and most of them have shown promising results. The studied TMPE was derived from a fatty acid obtained from waste sunflower oil. Compared to other biolubricants, the TMPE based ones have good lubrication properties, generally a good viscosity at 100°C (can be used at relatively high temperatures), and a very high viscosity index (greater than 100).

2.2 Viscosity measurements

The viscosity of the tested TMPE was measured using an Anton Paar ViscoQC 300 (L) Rotational Viscometer at different temperatures, ranging between 60°C and 100°C. The measurement method consisted in using a rotating spindle positioned in an open beaker (infinite gap). The beaker, filled with oil, was continuously and slowly heated, to increase the temperature. The viscosity was measured at different temperatures, and its variation with temperature is presented in Fig. 1.

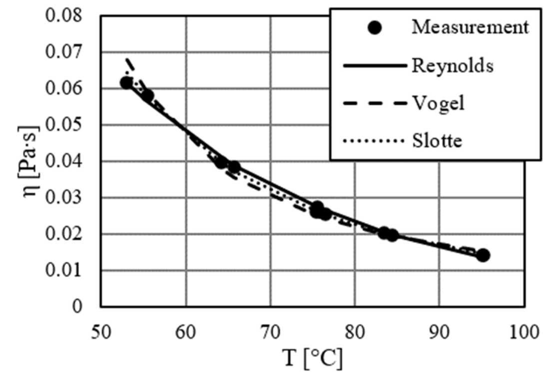


Fig. 1. The cup with testing balls submerged in oil

The experimental results were fitted using the least squares method with different well-known laws from the literature that describe the viscosity-temperature variation: Reynolds, Vogel and Slotte. Table 1 presents the values of the specific coefficients. Using ASTM D2270 and the viscosity measurement results, the obtained viscosity index was VI=167.

Table 1

Equations for variation of viscosity with temperature

Equation		Coefficients		
Reynolds	$\eta = \eta_0 e^{-\beta(T-T_0)}$	β=0.035		
Vogel	$\eta = ae^{b/(T-c)}$	*(a=)* 2.4	*(b=)* 177	*(c=)* 0
Slotte	$\eta = a/(b+T)^c$	*(a=)* 1575	*(b=)* 0.028	*(c=)* 2.55

Following the viscosity measurement, the density of the TMPE was also measured. For increased precision, two measurement methods were used: one using a graduated tube and a high precision scale (gravimetric method), and a second one using an analog density meter. The results obtained using the two methods were similar to the average measured value at room temperature (~22°C), $\rho_{TMPE} = 0.975 g/ml$,

2.3 Four-ball machine

The experimental device used to assess the performance of the tested TMPE was a four-ball tester, which is a standard machine used to measure the extreme pressure and anti-wear properties of lubricants, greases, and other fluids used in mechanical applications. The use of the four-ball machine spans many fields, including automotive, aviation, and industrial applications. The frictional contact was obtained by pressing one rotating bearing ball against three stationary ones, firmly held together inside a cup and a holding support. The contact was immersed in the tested lubricant which was added inside the cup (Fig. 2). The three lower balls were pressed against the upper ball using dead weights and a lever (with a factor of amplification of 10). A force sensor was used to measure the rotation force of the cup at the end of a small loading arm, mounted on the side. The friction torque M_f was obtained by multiplying the force F_s with the distance R=184*mm:* $M_f = F_s \cdot R$. The oil temperature was measured with a thermocouple inserted into the holding support. The temperature and force were recorded using 5B47 Analog Devices, respectively SCM5B38 Dataforth modules and a 5B08 Analog Devices backplanes 98 (8 channels) to support data acquisition.

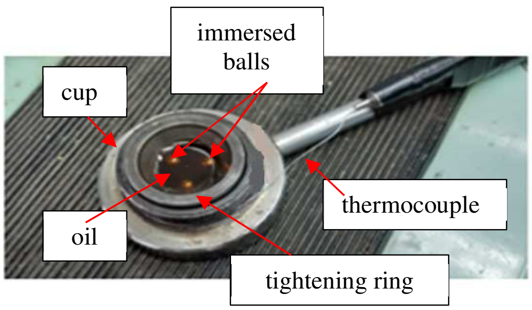


Fig. 2. The cup with testing balls submerged in oil

2.4 Experimental procedure

For each test, new sets of AISI 52100 alloy steel balls were used. The diameter of the balls is d=12.7mm (0.5inch) and the hardness is in the range of 64-66 HRC. A sufficient amount of lubricant was applied over the balls to ensure they were covered with a layer measuring 1-2 mm in thickness.

Before the test commences, the computer and data acquisition software are launched. The motor is activated one minute before the test begins, and the timer starts simultaneously with the applied load (at that moment, the contact is established between the cup's small lever and the force sensor). Upon the completion of the test, the load is removed, the motor is stopped, and the experimental data is saved, to be further processed.

The tribological tests focused on the extreme pressure limits of the tested lubricants. Two test methods were used based on the ASTM D4172 and ASTM D5183 standards, respectively. Following the ASTM D4172 method, the preliminary evaluation of the anti-wear properties of fluid lubricants in sliding contact was performed, by studying the variation of the WSD and the COF. This standard method implies testing the lubricant with a constant load of 392 N, a constant temperature of 75±2°C and a rotational speed of 1200±60 rpm, for 60±1 mins. During these tests, the COF and the WSD on the bottom three balls are measured.

The second series of tests were made according to the ASTM D5183 standard. For the first phase of the tests, the fourth ball was pressed with the same force of 392 N. The temperature of the wear-in lubricant was also regulated at 75±2°C, and the top ball was turned at 600 rpm for 60 min. The WSD was examined on each of the three lower balls using an optical microscope (Nikon SMZ1000). The images of the wear scars were captured using a digital camera and their sizes were measured with free source software.

An average value of 0.67±0.03 mm was obtained for the measured wear scars. It was in accordance with standard values. The repeatability was verified by means of three consecutive tests.

For the second phase, the balls tested in the first phase were further used in tests employing a load increment of 98.1N(10kg), which increased after each 10min, up to a point where the friction measurement indicated an incipient seizure. The COF was measured at the end of each 10min interval.

3. EXPERIMENTAL RESULTS

Fig. 3 shows the variation of the COF over time in the case of the test performed using the ASTM D4172 standard. The sample of TMPE showed comparable trends in the COF variation throughout the experiment. A high initial peak was seen at the start of most tests. Subsequently, the COF decreased to a value that remained relatively constant during the remainder of the test (Fig.3). The presence of the extreme peak (observed in the first minute, in Fig.4) was attributed to the loading system with manual release. The operator can reduce the influence of shock by carefully releasing the lever that is pressing the balls. Nevertheless, at the beginning of the test, the balls were in dry contact until lubricant was introduced between them through sliding. For some results (test 13 and 14 from Fig.4) the peak cannot be found at the beginning of the test and the COF was significantly lower for the duration of the entire experiment. A plausible explanation for this difference could be that the friction between the balls was characterized by an important boundary regime.

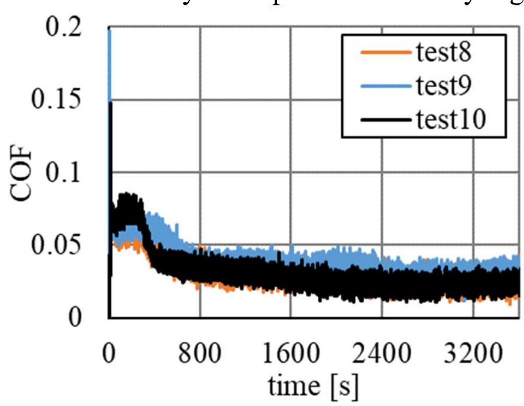


Fig. 3. Complete COF variation during 60min for selected tests in accordance with ASTM D4172

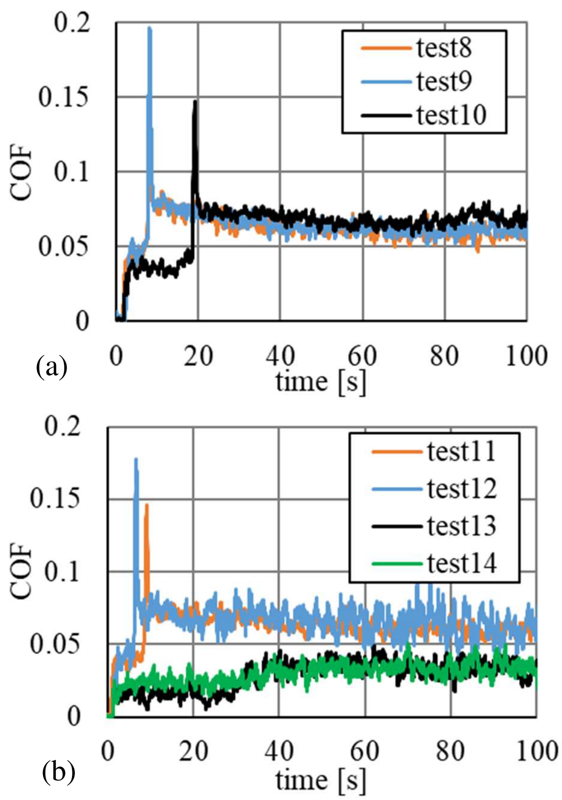


Fig. 4. COF variation during the start of the tests in accordance with ASTM D4172

The wear scar measurements indicate the severity of the wear: the higher the WSD value, the higher the wear. Some observations about the wear surface condition (type of wear, shape of wear scar, etc.) could be made using a high-definition microscope. A graph of the WSDs measured on the steel balls was plotted, as shown in Fig. 5. Results were compared with two other biolubricants based on sunflower oil fatty acids, found in the literature. In a previous paper, Turtoi et al. [24] presented similar results for other sunflower based lubricants: polyglycerol ester (sample A) and a mixture between a fatty acid and diethanolamine (sample B). Jabal et al. [25] found that under low load, the sunflower oil shows good anti-friction and anti-wear characteristics when compared with petroleum oil samples. Fig. 5 also depicts results from these two papers, and one can see that the wear is similar and the COF relatively higher.

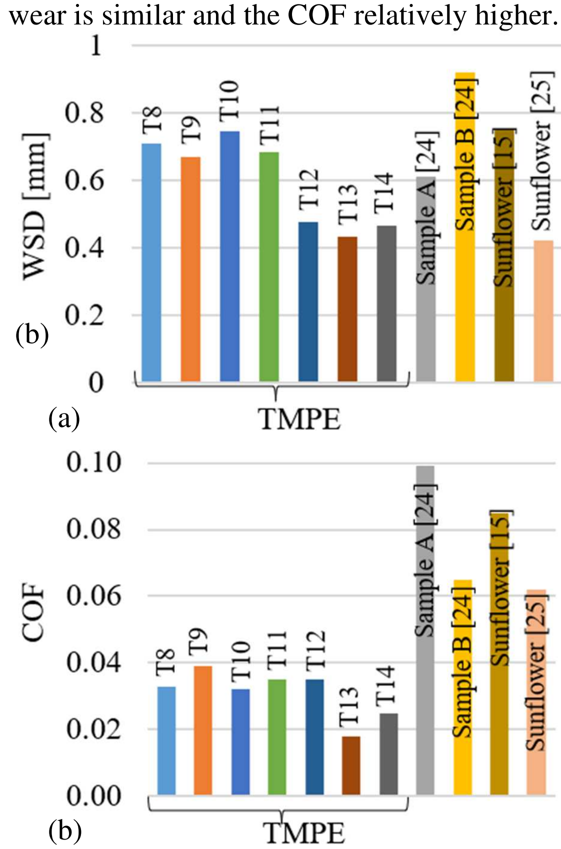


Fig. 5. WSD (a) and COF (b) for the tests in accordance with ASTM D4172 and other results from literature

Tests designed in accordance with ASTM D5183 were used to determine the COF, by means of the four-ball wear test machine. These tests offered information about the COF for each increment of 100kgf, the incipient seizure load (measured in kilogram force) and the final average wear scar (measured in mm). All tests started with a wear-in first phase, when the balls were loaded with 392N for 60min. The initial wear obtained was characterized by a WSD of 0.67±0.03 mm. Fig.6 presents phase two of the tests, where the seizure occurrence (marked by the rapid growth of COF) can easily be identified. Based on these results, one can notice that a good repeatability was obtained.

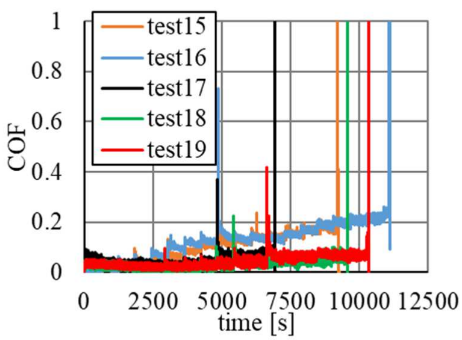


Fig. 6. COF variation during tests in accordance with ASTM D5183 and the occurrence of seizure

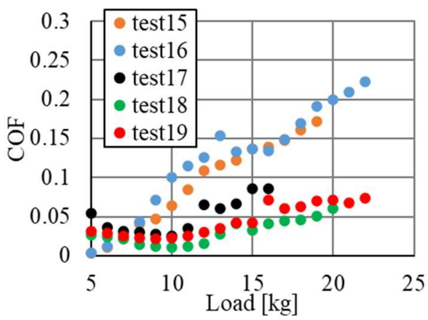
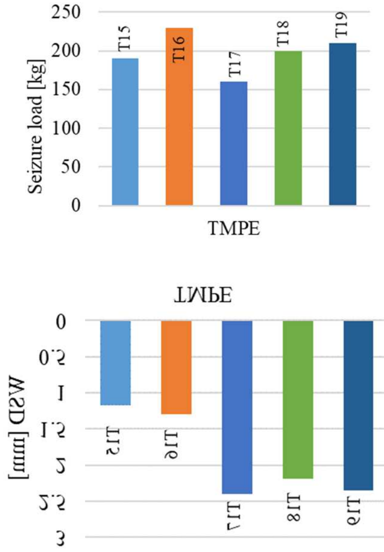


Fig. 7. COF variation during tests and the seizure occurrence

Fig.7 presents the evolution of the average COF for each load increment. This graph shows some inconsistencies and one can see that for tests 17-19, the COF was high at the beginning and relatively constant during the tests. For tests 15 and 16, the COF was low at the beginning, for low loads, but had a more pronounced variation.

The load, which was recorded for each test when the first signs of seizure were spotted, is presented in Fig.8 (a). The results are similar, and the tests offered good reproducibility. The balls were removed and with the WSD was measured for all three balls from the cup.

(a)



(b)

Fig. 8. Seizure load (a) and WSD (b) for the tests in accordance with ASTM D5183 and other results from the literature

Table 2

Tribological properties of various chainsaw lubricants

Lubricant	WSD* [mm]	COF* [-]
Sunflower oil TMPE**	0.5-0.7	0.07
Rapeseed oil [8]	0.4-0.8	0.04-0.06
Rapeseed oil + ZnO [8]	0.4-0.5	0.06-0.08
Rapeseed oil + TiO_2 [8]	0.4-0.6	0.04-0.06
Rapeseed oil [9]	0.5-2.0	0.05-0.08
Rapeseed oil + TiO_2 [9]	0.5-1.7	0.05-0.1
Pentaerythryl ester of rapeseed oil [10]	0.4	0.08
Rapeseed oil [11]	0.3-0.6	0.04-0.06
Rapeseed oil + hBN [11]	0.2-0.6	0.05-0.07
Rapeseed oil [12]	0.2-0.35	0.05-0.08
Palm oil TMPE[16]	0.2-0.33	0.07-0.11
Palm kernel oil TMPE [16]	0.2-0.4	
Crude jatropha oil [18]	0.7	0.05
Cottonseed oil methyl ester [22]	0.3-0.35	0.07-0.08
Calophyllum Inophyllum TMPE [23]	0.85-1.0	0.075

**Average value, ** ASTM D4172 tests*

Fig. 8 (b) presents the average WSD for each test. In the case of the WSD, the repeatability is lower, and the results are scattered. The main reason for this is the manual release of the lever used to apply the 10kg increment load. A high release shock can worsen the wear between balls, leading to a higher WSD.

Similar pieces of work dealt mostly with the performance of oil plant-based esters. The tested biolubricants proved to have good potential, and overall, the results were comparable with the ones found in the literature (obtained on the four-ball machine) (Table 2). Overall, rapeseed oil and derivatives have shown a higher capacity to reduce wear, while there was no significant difference regarding the COF.

4. CONCLUSIONS

In this paper, the tribological performance of a TMPE based on a fatty acid obtained from waste sunflower oil was analyzed. In general, the fatty acid composition influences significantly the tribological performance of biolubricants. It is known that biobased TMPEs have important wear reduction properties. A notable decrease in the COF was achieved for TMPEs, compared to other sunflower-based biolubricants found in literature. The results were compared for similar tests, in accordance with the ASTM D4172 standard. In terms of WSD, the results are not as conclusive as the ones for the COF. This is due to the high scattering of the measurements.

The extreme pressure characteristic of TMPE was studied using standard tests, according to ASTM D5183, by increasing the load until seizure. Results for seizure load were relatively consistent, and an average of 198kg was found. Wear was evaluated using the WSD, which was measured at the end of the test, after the first signs of seizure were noticed. A high spread of results was found, similarly to previous ones, obtained for constant load. The friction torque was recorded during the entire duration of the tests and the variation of the COF was found. For this test, no similar results in accordance with ASTM D5183 were found for sunflower oil or other derivatives.

The investigations of the worn surfaces of the stationary ball were limited only to the measurement of the dimensions of the wear scar using an optical microscope. No measurements of roughness or analyses with scanning electron microscopy (SEM) were made.

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TESTAREA TRIBOLOGICĂ A PERFORMANȚEI UNUI BIOLUBRIFIANT PE BAZĂ DE TRIMETILPROPAN OBȚINUT DIN ACIZI GRAȘI PROVENIȚI DIN ULEI DE FLOAREA SOARELUI

Eforturile de soluționare a problemelor de sustenabilitate a mediului i-au determinat pe cercetători să caute resurse alternative pentru lubrifianții lichizi. Este dovedit faptul că biolubrifianții au anumite avantaje: sunt ieftini, prezintă proprietăți de lubrifiere bune și, cel mai important, sunt biodegradabili. Trimetilolpropanul (TMP) este unul dintre cei mai des utilizați polioli în producția de biolubrifianți, datorită stabilității termice. Prin urmare, prezentul studiu urmează să evalueze tribologic performanța unui singur tip de TMP neaditivat produs din acizi grași din ulei de floarea soarelui. Testele tribologice au fost efectuate folosind o mașină cu patru bile în diferite condiții de încărcare. S-au determinat coeficientul de frecare, variația uzurii cu sarcina aplicată, precum și sarcina de gripare. Rezultatele au arătat că TMP-ul utilizat are proprietăți anti-uzură relativ bune. Rezultatele au fost comparate cu studii similare găsite în literatură.

Petrică TURTOI, Assistant professor, National University of Science and Technology POLITEHNICA Bucharest, Machine Elements and Tribology Dept., 313 Splaiul Independenţei, Bucharest, Romania, petrica.turtoi@upb.ro.
Julien DELCUZE, Eng**.,** INSA Lyon, 20 Av. Albert Einstein, 69100 Villeurbanne, France, julien.delcuze@insa-lyon.fr.
Ionuț-Răzvan NECHITA, PhD student, Nat. Univ. of Science. and Technology POLITEHNICA Bucharest, Machine Elements and Tribology Dept., 313 Splaiul Independenţei, Bucharest, Romania, ionut_razvan.nechita@upb.ro.
Alice MARINESCU, PhD student, Nat. Univ. of Science. and Technology POLITEHNICA Bucharest, Machine Elements and Tribology Dept., 313 Splaiul Independenţei, Bucharest, Romania, alice.marinescu@upb.ro.