Experimental Assessment of the Wear Preventive Characteristics of Waste-Recovered Biodegradable Total-Loss Lubricants Produced from Sunflower Oil

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Abstract: Bio-lubricants represent a reliable alternative to classical, petroleum-based lubricants. This paper aims to assess the tribological properties of two bio-degradable lubricants, based on sunflower waste cooking oil. Prior to the tribological tests, the rheo-physical properties of the two lubricant samples were determined. The load-carrying properties of both lubricants were evaluated using a four-ball testing machine by employing methods which covered the load-wear variation and the assessment of extreme pressures. The coefficient of friction (COF) and wear scar diameter (WSD), as well as the influence of lubricant temperature, were evaluated. The results obtained for the bio-lubricant candidates were compared with similar studies from literature, and both tested bio-lubricants were proven to have good potential.

Keywords: biodegradable, four-ball machine, sunflower oil, total-loss lubricants

1 INTRODUCTION

The need for environmental protection, which became more evident in recent years, imposes among other regulations, stricter conditions regarding the use of lubricants. This implies increased attention with regard to the management of oil residues, as well as the attempt of replacing lubricants based on mineral oils with alternative solutions, of vegetable or alimentary origin, namely biodegradable lubricants. The latter condition becomes essential for applications involving total-loss lubrication systems, where the used lubricant cannot be recovered.

Such a situation can be found in the case of lubricants used for guide bar – chainsaw contacts (chainsaw lubricants, for short) which, after fulfilling their function, are completely retrieved into nature: one-third of the lubricant is soaked into the soil or transformed into aerosols and the other two-thirds are adhering to the cut wood [13].

A relatively recent study [16] targeting the impact of the waste lubricants on the forest contamination in Central Europe revealed up to 7 million liters of various mineral oils being soaked annually into forest soil, the majority being produced by chainsaws, extensively used in forestry activities.

Bio-lubricants represent an attractive alternative to petroleum-based lubricants, being both an economic and ecological solution since they are produced mainly from disposable materials. Despite their slightly lower performances, in comparison with their classic, petroleum-based counterparts, bio-lubricants exhibit low toxicity and high levels of biodegradability, and thus, increased security for users.

Bio-lubricants are highly effective in applications involving chainsaws, lawnmowers, and other agricultural equipment, where lubricants are getting absorbed into the soil or water, where living organisms cannot decompose them.

Renewable sources are preferred for biolubricants, and vegetable oils represent candidates with high potential. For many bio-lubricants, triglyceride esters (plant fats) derived from vegetable oils are used. An extensive survey of the literature reveals a lack of standard evaluation methods for chainsaw lubricants. The few papers dealing with the subject present different approaches, some of them relying on classical testing procedures for petroleum-based products.

However, chainsaw lubricants have some particularities which must be considered during their testing. For chainsaw applications, the same lubricant operates in three contacts: guide bar-chain, chainsprocket, and chain-lumber, each contact having different operating conditions. At the same time, the durability of the lubricant is not a major concern, since the life of the lubricant is rather short, being of the order of minutes.

It is worth mentioning that for total-loss lubrication systems, a high level of biodegradability is required. Even though there are several standardized testing methods for biodegradability (CEC-L-103-12 [3], CEC-L-33-A-93 [2], OECD 301 B/C [1], DIN EN ISO 9408 [7], DIN EN ISO 9439 [6]), none of these methods are conclusive for bio-lubricants and the reproducibility is poor [17].

In view of the aforementioned, one of the main questions before commencing to assess the performances of a chainsaw lubricant is: "Which of the available tests reflects better the qualification of a bio-lubricant for chainsaw applications?"

Firstly, there are several standard physical and chemical tests of major interest, which include (but are not limited to): rheological tests, pour point tests, flash point tests, etc. Rheological tests provide information on the viscosity of the lubricant at different temperatures (typically at 40°C and 100°C) and on the viscosity index (VI).

However, there is no consistent information about lubricant viscosity, and the products that can be found on the market have values in a large range. Some sources [19] consider as a reference viscosity that of SAE 10W-30, but there is no scientific support for it.

Obviously, as chainsaws operate in a large interval of temperatures (from negative temperatures up to $+100^{\circ}$ C), the viscosity index is of major interest. On the other hand, mechanical tests are necessary to be

performed, in order to assess the values of the friction coefficient and the wear resistance performances.

During the last two decades, one could remark two different experimental approaches for the evaluation of total-loss lubricants: classical tribological tests performed on standard test rigs (four-ball test rig, blockon-ring tribometer, pin-on-disk tester) and tests made on in-house chainsaw testing devices [15].

Rac and Vencl [14] report the performances of two chainsaw lubricants, based on new sunflower oil, additivated with antioxidants, pour point depressants (PPD), and thickeners. The tribological evaluation performed on a block-on-ring test rig has shown a very low friction coefficient (average upper and lower values of 0.53 and 0.074, respectively).

Stanovský et al. [20] evaluated a chainsaw biolubricant, in contrast with a classical mineral oil competitor, based on the average guide bar-chainsaw temperature. The temperature was measured with an infrared camera during in-situ tests which used two chainsaws to cut several species of trees. The results obtained showed that the two tested lubricants were similar, with a slight performance advantage for the mineral oil, which was compensated by the rapid degradation in the soil and very low ecotoxicity of the bio-lubricant.

One of the most comprehensive evaluations of several chainsaw lubricants was published by Orawiec et al. [15] Two petroleum-based and two bio-degradable chainsaw lubricants have been compared along with two standard engine oils (SAE 30 and SAE 5W-30) in terms of mean guide bar temperature, which was proposed as a performance criterion.

The temperature was measured with an infrared camera on a custom-built precision chainsaw testing apparatus that replicated the real operating conditions. The authors concluded that the mean temperature is slightly higher for bio-lubricants (64°C, compared to 58°C for petroleum-based lubricants), and the average viscosity at the mean temperature that minimizes the friction is close to 30cSt. The paper also included a series of experimental results obtained on a four-ball tester.

2 MATERIALS

The analysis within the current paper targets testing the behavior of two biodegradable lubricants. The chemical processes necessary to produce the tested samples are still on an exploratory stage. Since neither of the two bio-lubricants are yet on the current market, for reasons concerning the intellectual property, the exact quantities and procedures involved within their recipes will not be mentioned. For the are sake of simplicity, the terms "sample A" and "sample B" shall be further used to reference one or the other lubricant.

Both lubricants are derived from the same substance, i.e. sunflower waste cooking oil. Sunflower oil is a triglyceride, being a mixture of saturated and unsaturated acids, with about 15% saturated and 85% unsaturated fatty acids. Sample A is a polyglycerol ester – a polyester obtained from fatty acids of sunflower oil and polyglycerol, known for having amphiphilic properties.

The main chemical processes which led to the production of sample A are the polymerization of glycerol, followed by esterification with fatty oleic acids. To create polyglycerol, glycerol is subjected at temperatures above 200 °C in the presence of an alkaline catalyst. The sunflower oil fatty acids are separately heated above 200 °C, which leads to inter-esterified oleic fatty acids. Further, the polyglycerol and the inter-esterified oleic fatty acids are mixed, thus resulting in the final composition of the sample.

For sample B, the fatty acids from sunflower oil were mixed with a diethanolamine, resulting in an oleic diethanolamide, a compound renowned for its corrosion inhibition and great lubrication and antistatic proprieties.

In order to reduce the influence of the composition upon the results obtained during the experimental work, lubricants originating from the same two individual batches were used.

Among their former envisioned application – that is bio-lubricants used in total-loss systems, both lubricants could be reliable candidates for other areas, such as the food, cosmetical, and pharmaceutical industries, due to their non-toxic behavior.

3 EXPERIMENTAL WORKS

Prior to performing tribological tests, the physical properties of interest (i.e. densities, kinematic and dynamic viscosities, viscosity indices, and pour points) for the two bio-lubricant samples and a petroleum-based chainsaw lubricant (PBCL 3) found on the market were determined using standard methods.

Firstly, the densities of the lubricant samples were determined at room temperature, using a hydrometer. For the viscosity measurements, a rotational viscometer with coaxial cylinders (Anton Paar ViscoQC 300) was used. The output given by the viscometer was in terms of dynamic viscosities, which alongside the previously-determined densities, were used to compute de kinematic viscosities. The kinematic viscosities obtained for two reference temperatures (40°C and 100°C, respectively) were used to assess further the viscosity indices (VI) of each lubricant. The VIs were determined based on the ASTM D2270 standard [9]. In order to find the pour points corresponding to the tested lubricants, the ASTM D97 [10] testing procedure was used.

The obtained results were compared with results found in the literature [14],[15] for both biodegradable and petroleum-based chainsaw lubricants, and are presented in table 1. As one can notice, in terms of viscosities, the two candidate bio-lubricants were the most viscous among the lubricants within the analysis. The VIs of the tested samples were in the middle range, with respect to the VIs of similar products. The pour points were expected to be higher, since high pour points are correlated within lubricants deriving from a larger proportion of plant material. The tribological tests used to assess the load carrying properties of the two bio-lubricants were performed on a four-ball test rig (fig. 1). The tested balls were made of a chrome alloy steel (AISI E-52100 [4]) with a diameter of 12.7mm and a hardness in the range of 64-66 HRC. The four-ball machine employs a low sliding speed between the balls in contact (under 1m/s) which is sensibly lower than actual sliding speed of the chainsaw. However, the main advantage of using the four-ball machine is the standardized experimental procedure, which allows an easy comparison of the obtained results with others from similar studies.



Fig. 1 Four-ball machine setup: 1 – electric motor; 2 – tachometer; 3- force sensor; 4 – cup assembly; 5 – thermocouple; 6 – data acquisition system; 7 – loading arm.

The tribological tests covered the wear preventive properties, as well as the extreme pressure limits of the tested lubricants . These methods were based on a series of standards for oil lubricants (ASTM D2783 [5], ASTM D4172 [8] and ISO 20623 [11]).

The first series of tests performed in order to study the wear preventive properties of the tested lubricants were based on the standardized method from ASTM D4172 [8]with a constant load of 392N and a rotational speed of 1200 rpm (a sliding speed of 0.46m/s), for an hour. The temperature of the lubricant was kept almost constant during the experiments, at $75\pm2^{\circ}$ C, by using a heating gun. The wear scar diameters (WSD) were measured using an optical microscope (Nikon SMZ1000). For the same load used previously (i.e. 392N) a higher speed (1450rpm) was employed. Thus, in order to maintain the same relative sliding distance, the duration of the tests was reduced to 50 minutes, instead of an hour.

In order to achieve a better understanding of the wear-preventing capacity of both tested lubricants, supplementary tests were performed for a lower speed (1000rpm corresponding to 0.38m/s relative sliding speed) and a lower load (294N). This series of tests were performed without controlling the temperature values,

the tests being performed starting with room temperature.

Another set of tests focused on determining the load wear index (LWI), using the method provided by the ISO 20623 [11] standard. Three types of tests are described in the standard to determine the correlation between wear and load (wear-load curve) and to find the seizure point at a sliding speed of 0.556m/s (corresponding to 1450rpm) during 1 minute tests. Consecutively increasing loads with an increment of 98N, in the range 588N up to 1667N, were used and the wear scar diameter was measured for each test. The lubricant was initially at room temperature.

To better understand the wear behavior of the tested lubricants, extreme wear tests were used. Thus, the four-ball machine was loaded successively at a given time interval for a constant rotational speed, in order to find the seizure limit (corresponding to the welding effects at the surface of contact of the tested balls) for each bio-lubricant.

4 RESULTS AND DISCUSSIONS

The measurements of the WSD under the microscope (fig. 2 and fig. 3) were done on parallel and perpendicular directions with respect to the texture lines and averaged (table 2). Comparing the values, the WSD was noticed to be significantly higher for sample B, regardless of the tests spanning on 50 minutes or one hour.





Fig. 2 WSD [mm] for sample A (392N, 60min, 1200rpm)

Fig. 3 WSD [mm] for sample B (392N, 60min, 1200rpm

The average friction coefficient was calculated based on the variation of the friction torque measured during the tests and it is presented in figure 4, alongside the temperature variation of the two tested lubricants, starting from room temperature.

The graph from figure 5 presents comparatively, for the two tested bio-lubricants, the average friction coefficient recorded during an extreme load test. For this test, the load was increased every 2 minutes with an increment of 98N. One can notice that, for either lubricant, the friction coefficient is almost constant, despite the load having increased. The same graph is also depicting the variation of the temperature, starting from room temperature. The seizure limit was reached for a load of 1177N for both bio-lubricants, at a temperature of approximatively 60°C.



Fig. 4 Friction coefficient for sample A and sample B at a constant load of 294N and 1000rpm for 60min



Fig. 5 COF and temperature variation during extreme wear tests for sample A and sample B (increment of 98N at 2min interval)

During the experiments, the loading was performed manually, by adding weights at the end of an amplification lever, and thus, peaks of variation which mark the increase of the load can be observed on the graph from fig. 5.

Using the ISO 20620 [12] specifications, it was possible to study the variation of the WSD with the load. These results are presented in figure 6, where one can notice a higher WSD for sample B. The seizure was observed for a load of 1470N, which was also higher, compared with the value obtained for the previous extreme wear tests. This can be justified by the low reproducibility, specific to wear tests.

The results obtained for the two candidates biolubricants were compared with results obtained for rapeseed oil – a product which is currently intensively studied, due to its eco-friendly qualities when used as a lubricant. Rapeseed-based products are also of high interest because large quantities of the raw material are available, surpassing the sunflower production. When comparing the results with results obtained for rapeseed oil (pure and with additives) [18][21][22], one discovers that the performances are similar. Our results for COF and WSD compared with the results obtained for rapeseed oil [22] are presented in figure 7. One can see that the coefficient of friction is similar, and some differences were found for WSD, within the limits of precision related to the reproducibility levels of wear tests.



Fig. 6 WSD variation with load for sample A and sample B



Fig. 7 Comparison of COF and WSD for sample A and rapeseed oil [22]

4 RESULTS AND CONCLUSIONS

The paper followed the assessment of the tribological properties of two biodegradable lubricants, based on sunflower waste cooking oil. For this matter, exploratory tests were made for the candidate lubricant samples, as well as for a petroleum-based chainsaw lubricant found on the market (PBCL 3). Further, the obtained results were compared with other results found in the literature for various chainsaw lubricants, either laboratory-made or from the market. The comparison was performed in terms of rheo-physical properties (table 1), wear scar diameter (WSD) and coefficient of friction (COF) (table 3).

The two tested bio-lubricants were proven to have a good potential, with sample A exhibiting better performances than sample B, and overall, the results for the two tested lubricant samples were comparable from a range point of view with the ones found in the literature.

Lubricont type WSD [mm] COF []					
Lubricant type	won [mm]				
Sample A	0.69	0.073			
Sample B	1.24	0.081			
BCL 1 [15]	/	/			
BCL 2 [15]	/	/			
BCL 3 [14]	0.47	0.126			
BCL 4 [14]	0.70	0.070			
Rapeseed oil [22]	0.58	0.084			
ROA 1 [18]	0.3	0.078			
ROA 2 [21]	0.4-0.55	0.082-0.16			
ROA 3 [21]	0.42-0.7	0.055-0.14			
PBCL 1 [15]	0.49	/			
PBCL 2 [15]	0.47	0.103			
PBCL 3	1.36	0.120			
CI – biodegradable chainsaw lubricant					

Table 3.	Tribological properties of various	chainsaw
	lubricants	

BCL= biodegradable chainsaw lubricant ROA=rapeseed oil with additives PBCL=petroleum-based chainsaw lubricant

However, similar pieces of work found in literature dealt mostly with the performances of additivated lubricants of vegetal origin. In most situations, additives were proven to enhance the performance of lubricants [22],[18],[21]. Even so, there are situations when the performances and overall desirable properties of lubricants could deteriorate during additivation (especially the bio-degradability and non-toxicity, which could further impact the water and soil pollution levels). In other cases, even though the performances of the lubricant do not improve with additivation, the duration of service does improve. Since the chainsaw lubricants are subjected to mild conditions of load and temperature variation, their durability is rather short, but the extension of the lubricant durability is not a factor of paramount importance. Nonetheless, the current formulae used for the tested samples were intended to create reliable bio-lubricants, with minimum financial investment with respect to the ingredients used, which are still able to perform the job with reduced pollution of soil or water. Withal, the composition of the samples does have a shortcoming, that is, the raw material used (i.e. waste sunflower cooking oil) did not come from the same batch, and thus, the chemical composition for future samples, regardless of the recipe used, could be influenced by the age, impurities, and range of temperatures at which the cooking oil has been previously used.

Table 1. Rheo-physical properties of various chainsaw lubricants

Lubricant type	Viscosity (40°C) [cSt]	Viscosity (100°C) [cSt]	Viscosity Index (VI) [-]	Density (15°C) [kg/m ³]	Pour point [°C]	
Sample A	154	28.9	228	844	-17	
Sample B	442	32	105	890	-13	
BCL 1 [15]	31.2	7.7	228	/	/	
BCL 2 [15]	95.2	20.8	248	/	/	
BCL 3 [14]	47.6	10.5	218	917	-20	
BCL 4 [14]	99.6	11.2	98	881	-25	
PBCL 1 [15]	63.9	10.4	154	/	/	
PBCL 2 [15]	67.6	7.6	63	/	/	
PBCL 3	101	22.2	249	767	/	

BCL= biodegradable chainsaw lubricant PBCL=petroleum-based chainsaw lubricant

Table 2 The average WSD in parallel and perpendicular directions for sample A and sample B

Lubricant type	Load [N]	1200rpm&60minWSD [mm] (ASTM D4172)		1500rpm&50min WSD [mm]			
		PAR //	PERP⊥	AVG	PAR //	PERP⊥	AVG
Sample A	- 392	0.61	0.65	0.63	0.51	0.55	0.53
Sample B		0.90	0.92	0.91	0.68	0.70	0.69

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