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EXPERIMENTAL INVESTIGATION ON RHEOLOGICAL PROPERTIES OF A COMPLEX LITHIUM-CALCIUM GREASE IN FRESH AND USED CONDITION

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Abstract: *The purpose of the paper is to investigate the rheological and tribological properties of a complex lithium - calcium grease, using a cone and plate viscometer and a pin-on-disk tribometer. The experiment was performed on two samples of the same grease, in fresh and used condition (during use in a worm gear). In the course of the experiments, the effect of tackiness and adhesion phenomenon was studied, for both fresh and used grease. Finally, it has been observed a strong dependence between the operating time and technical performances of greases: decreasing the values of the rheological parameters (viscosity and yield stress), increasing of the friction coefficient between tested surfaces and decreasing of the threads length during the adhesion test. The principal result of this research is an alternative method for the evaluation and quantification of the wear degree and lubricants durability.*
Key words: *Lubricating grease, Degradation, Rheology, Adhesion*

1. INTRODUCTION

Lubricating greases are colloidal systems composed of two phases, the dispersion medium forming the outer phase of the system, and the dispersion the inner phase. Phases can consist of a single component or several. They also contain active surface substances that do not form an independent phase, i.e. system stabilizers. The dispersion medium is liquid, and the dispersion can consist of solid, plastic or liquid bodies.

The vast majority of solid greases contain in their composition a solid phase (thickening agent) and a liquid phase (mineral oils), forming a colloidal dispersion [1]. Sodium, calcium, aluminum, barium, lithium, etc. soaps are used as thickening agents. As a dispersion medium, refined mineral oils are used in proportion of 75%...90%, of well-specified quality [2].

Lubricating greases are used in a wide range of industrial products such as bearings, gears, guides etc. [3–6]. Determining the life of solid greases is especially important because users and operators must properly schedule relubrication intervals and prevent catastrophic component failure [7].

From this point of view, it is very useful to establish a few criteria for the grease life time, as follows: modification of the physical and chemical properties of the greases [8-9], modification of the rheological parameters [10], modification of the microscopically grease structure and apparition of the wear particles [12], evaluation of the effect of tackiness and adhesion phenomenon [13] etc.

The procedure used in the present work was to assess the modifications in rheological and tribological properties for a lithium-calcium complex grease, in fresh and used state. Its purpose is the development of a fast diagnoses method for lubricants, with minimal investments and a high precision level, easy to use.

The testing program, using a Brookfield viscometer and a Gunt pin-on-disk tribometer, analyzes the influence of the lubricant state of degradation, considering the changing of the rheological and tribological properties during the working time. The degradation process of the grease was performed in a double worm gearbox. For both samples of fresh and used grease, the effect of tackiness and adhesion phenomenon was studied.

The main result of this research is an alternative method for the evaluation and quantification of the wear degree and lubricants durability.

2. METHODOLOGY

2.1 Lubricating grease

The lubricating grease used for testing was a lithium-calcium complex grease, in fresh and used state, added with molibdenum disulphide. This grease is characterized by good mechanical and thermal stability, being used for a range of temperatures between -30...110^{0}C. It is recommended to be used for lubrication of bearings, gears and various devices or mechanisms.

Table 1 summarizes the information regarding the properties of the grease in fresh state.

Table 1

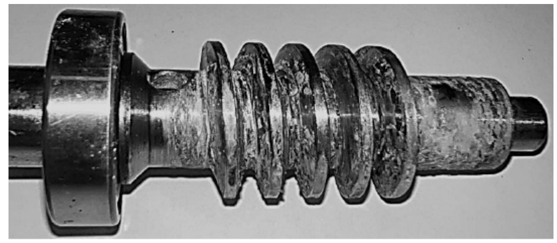
Properties of the lithium-calcium complex grease.

Characteristic	Value
NLGI penetration class	3
Aspect	pasty
Color	brown
Temperature range, ^{0}C	-30 … +160
Kinematic viscosity at 40^{0}C, cSt	190
Kinematic viscosity at 100^{0}C, cSt	17
Dropping point, ^{0}C	170
Base oil	Mineral oil - naphtenic
Base oil viscosity at 40^{0}C, cSt	220
Base oil viscosity at 100^{0}C, cSt	23

The grease was used in a double worm gearbox from air conditioning installation of passenger cars for the railway, for an operating time of 7000 hours. The main technical parameters of the gearbox are:

- Power: 0.4 kW;
- Rotation: 2000 rpm;
- Transmission ratio: 630:1;
- Maximum torque: 1.96 Nm.

The worm gear sets, worn after operation for two years (7000 hours) are presented in Figure 1. It can be easily observed the appearance of traces of adhesive wear on the flanks of the worm and on the wheel.



a. Worm

b. Wheel

Fig. 1. Worm gear sets, worn after 7000 hours operation

2.2 Rheological test

The rheological test was performed using a cone and plate Brookfield Cap 2000+ viscometer, which can measure viscosity in a varied range of temperature and speed conditions [13]. The main technical characteristics of the viscometer are:

- Main shaft speed: continuously variable in the range of 5 – 1000 rpm;
- Variation range of shear rates: between 10 and 13330 s^{-1};
- Testing temperature: adjustable with a precision of 0.1^{0}C in the range 5 … 75^{0}C.

The entire system is controlled by a specialized viscometer software Capcalc 32, which performs the functions of programming, command and data acquisition, numerical data processing, as well as the general setting of the working parameters of the viscometer.

In order to determine the rheological model for the analyzed grease, a "velocity imposed gradient" type test was used, at the reference temperature of 20 ^{0}C, with a grease soaking time of 60 seconds. The working geometry of the cone was number 5, having the following characteristics: cone radius 9.53 mm; cone angle 1.8^0; shear rate 16 ... 167 s^{-1}.

As variant of proposed rheological model was Bingham model [14]:

$$\tau = \tau_0 + \mu \frac{\partial u}{\partial y} \quad (1),$$

where: τ – shear stress;

τ_0 – yield stress;

η – dynamic viscosity;

$\frac{\partial u}{\partial y}$ – shear rate.

2.3 Tribological test

The tribological test was carried out at room temperature, using a Gunt pin-on-disk tribometer [15], with the following technical parameters:

- Rotation speed for the output shaft of the drive system: continues variable in range 0 ... 200 rot/min;
- Maximum torque on the output shaft: 18.5 Nm;
- Maximum load of the force transducer: 50 N;
- Amplification ratio of loading system: 2:1;
- Weights: 1 x 5 N, 1 x 10 N and 1 x 20 N;
- Pin diameter: 4 mm

The testing pin and disk were made of C45 steel, with the surface roughness R_a = 0.8 μm. During the test, the friction force results were recorded and each measurement was repeated three times.

Finally it obtains a mean value of the friction coefficient for each testing speed.

2.4 Tackiness (adhesion) test

The tackiness (adhesion) property of lubricating greases to the surfaces of a friction couple is defined as the ability of the grease to form threads when two surfaces are separated from each other [16]. At this moment, the most used method for evaluating thread formation is the finger test, which involves applying a film of grease between two fingers and separating them. According to this test, the increase of the threads length implies a higher degree of adhesion for the lubricating grease.

Apart this empirical method, there were developed other experimental methods, with a higher degree of objectivity, which are based on the measurement of interaction forces between a sample and a layer of lubricating grease [17, 18].

In this work, we propose to use a semi-empirical method, which assumes that the lubricating grease film is deposited between the two semi-couples of the viscometer (cone and plate); their separation is carried out in a controlled manner, with the same movement speed, but without the possibility of measuring the film breaking force. So, it will be possible to evaluate the tackiness (adhesion) of the lubricating grease, in a qualitative way, and same time to visualize the formation of grease threads characteristic of the surface separation process.

3. RESULTS AND DISCUSSION

3.1 Rheological test

The experimental test consists of a load from the 17 s^{-1} to 167 s^{-1} shear rate gradient, followed by an unload in order to highlight the thixotropy of the lubricant – "shear memory".

The rheograms are obtained by plotting shear stress as a function of the shear rate, as 50 measuring points at each shear rate, using the software Capcalc 32 specific for the viscometer [13].

Figure 2 presents the rheograms obtained for fresh and used grease, obtained with the viscometer.

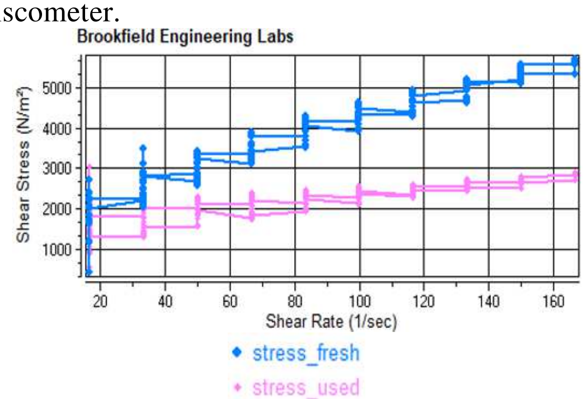


Fig. 2. Rheograms for fresh and used grease

The values of the rheological parameters, for both fresh and used grease (Table 2), are obtained by regression method, according the Bingham rheological model (eq. 1).

Table 2

Rheological parameters for fresh and used grease.

Grease type	Bingham model		
	τ_0 [Pa]	η [Pa.s]	Correl. coef.
Fresh	1721	24.271	92.3%
Used	1473	8.238	89.2%

Analyzing these values it can observe a strong dependence between the operating time and rheological parameters of greases: decreasing of the values for the yield stress (with 15%) and dynamic viscosity (with 66%) for the used grease by comparison with fresh grease.

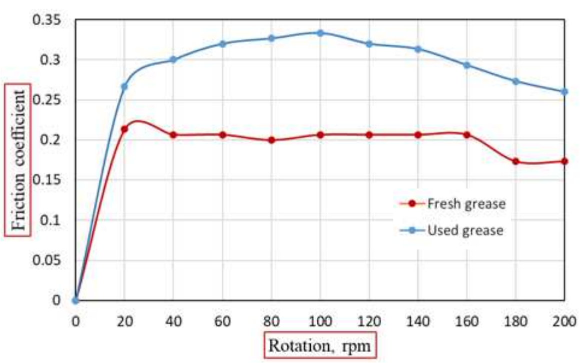
3.2 Tribological test

Tribological tests present the variation of the friction force versus load and rotation, highlighting the influence of the operating time over lubricant state of degradation.

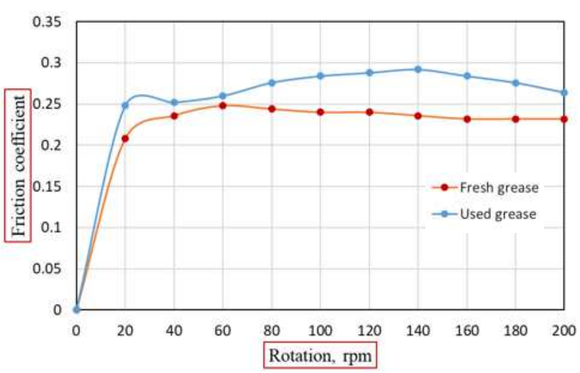
The Stribeck curves for fresh and used grease are obtained in the following conditions:

- Loadings: 15, 25, 35 and 45 N;
- Equivalent contact pressure: 1.2, 2, 2.8 and 3.2 MPa;
- Rotation: 20 … 200 rpm;
- Equivalent sliding speed: 0.003 … 0.03 m/s;

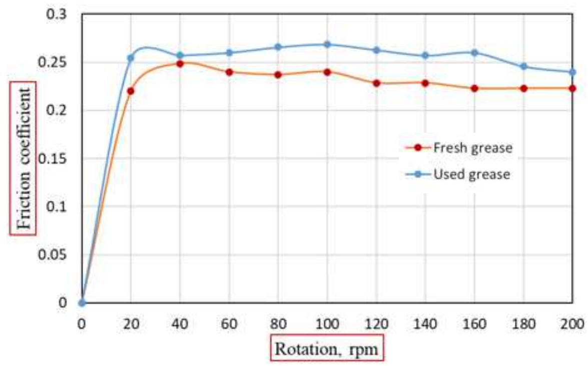
Figure 3 present the obtained Stribeck curves, for the four proposed loadings.



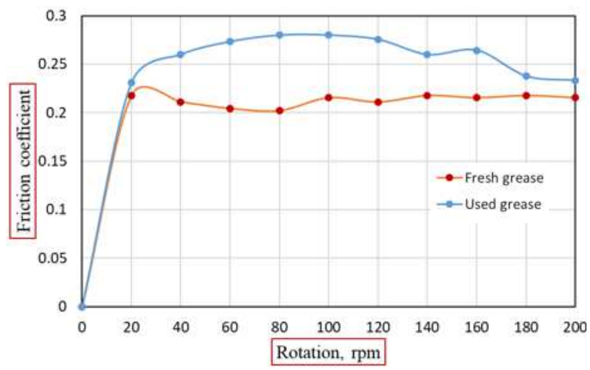
a. Load 15 N (contact pressure 1.2 MPa)



b. Load 25 N (contact pressure 2 MPa)



c. Load 35 N (contact pressure 2.8 MPa)



d. Load 45 N (contact pressure 3.2 MPa)

Fig. 3. Stribeck curves for fresh and used grease

The mean values obtained for the friction coefficient (μ), the mean square deviation (σ) and the 95% confidence limits (CL) are presented in Table 3.

Table 3

Friction coefficient for fresh and used grease.

Load [N] / **Grease type**	**15**			**25**		
	μ	**σ**	**CL**	**μ**	**σ**	**CL**
Fresh	0.200	0.0144	0.00029	0.241	0.0086	0.00022
Used	0.301	0.0264	0.00052	0.267	0.0265	0.00031

Load [N] / **Grease type**	**35**			**45**		
	μ	**σ**	**CL**	**μ**	**σ**	**CL**
Fresh	0.234	0.0106	0.00019	0.213	0.0056	0.00011
Used	0.253	0.0147	0.00017	0.259	0.0191	0.00038

Figure 4 presents the variation of the mean friction coefficient versus load for fresh and used grease.

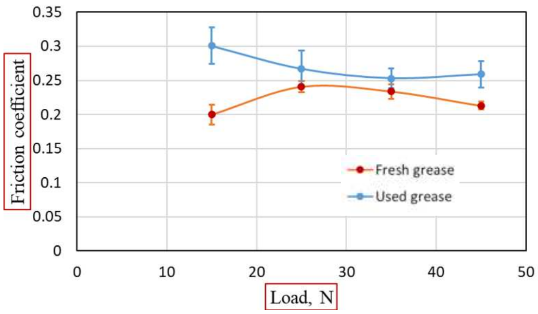


Fig. 4. Friction coefficient versus load for fresh and used grease

Analyzing the experimental results regarding the tribological test, it can observe that the friction coefficient between the tribometer pin and disk is always greater for the used grease by comparison with the fresh grease, for all values of rotations and loads. For the fresh grease, the friction coefficient has a maximum value for the load of 25 N, while for the used grease, the same friction coefficient has a minimum value for the load of 35 N.

3.3 Tackiness (adhesion) test

The tackiness (adhesion) of the lubricating grease and visualization of the of grease threads formation, characteristic of the surface separation process, was realized at the end of the rheological test. Figure 5 shows the same rupture process of the grease film, for fresh and used grease.

a. Threads formation for fresh grease

b. Threads formation for used grease

Fig. 5. Visualization of the film rupture for fresh and used grease

Analysing these visualizations, it can observe that used grease has a reduced degree of tackiness by comparison with fresh grease. Also, the threads before separation are longer for the fresh grease than the used grease, where the threads are practically absent.

4. CONCLUSION

In this paper was performed the study of the rheological and tribological properties of a complex lithium - calcium grease added with molybdenum disulphide, fresh and used in a double worm gearbox. The rheological properties were investigated in range of 17 s^{-1} to 167 s^{-1} shear rate gradient and the results were fitted according to Bingham model.

The tribological properties (Stribeck curves) were determined at low sliding velocities (0.003 … 0.03 m/s) and normal contact pressures (1.2 … 3.2 MPa). It can be observed an increasing of the friction coefficient and a decreasing of the yield stress and viscosity for the degraded grease, by comparison with the same fresh grease. Therefore, the rheological parameters and the friction coefficient are both a very precisely indicator of the grease degradation level.

The adhesion, cohesion and tackiness (ability to forms threads before separation) are also a criteria of the degradation level, only that they present a higher degree of subjectivity and they are more difficult to quantify.

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CERCETĂRI EXPERIMENTALE PRIVIND PROPRIETATILE REOLOGICE ALE UNEI UNSORI COMPLEXE PE BAZA DE SAPUNURI DE LITIU-CALCIU, IN STARE PROASPATA SI UZATA

Rezumat: Scopul lucrării este cercetarea proprietăților reologice și tribologice ale unei unsori consistente pe bază de săpunuri complexe Li-Ca, utilizând un viscozimetru con-placă și un tribometru pin-on-disk. Experimentul a fost efectuat pe două probe din aceeași unsoare consistentă, în stare proaspătă și uzată într-un angrenaj melcat. Pe parcursul experimentelor s-a studiat efectul fenomenului de aderență, atât pentru unsorile proaspete, cât și pentru cele uzate. În final, s-a observat o dependență puternică între gradul de uzură și performanțele unsorilor: scăderea valorilor parametrilor reologici (viscozitate și prag de tensiune), creșterea coeficientului de frecare între suprafețele testate și scăderea lungimii filamentului în timpul testului de aderență. Principalul rezultat al acestei cercetări este o metodă alternativă de evaluare și cuantificare a gradului de uzură și durabilitatii lubrifianților.

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