Proceedings of BALTTRIB'2017 edited by prof. J. Padgurskas
eISSN 2424-5089/eISBN 978-609-449-127-6

https://doi.org/10.15544/balttrib.2017.18

OPTIMIZATION OF LITHIUM SOAP-BASED GREASE TESTING METHODOLOGY BASED ON MEASUREMENTS REPEATABILITY

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Abstract: The purpose of the paper is to investigate the rheological properties of a lithium soap-based grease, using a Brookfield viscometer. An optimized methodology was proposed, applicable to the tested grease, which takes into account the possibility of reducing the testing time by analyzing the degree of measurements repeatability. Finally, the thermal variation of the rheological parameters (yield stress and viscosity) was obtained, in the temperature range 10...75 ^{o}C. The main result of the paper is the reducing of the testing time from 6 hours to 3 hours for one temperature, with the same precision of measurements, according to economical effects.

Keywords: Rheology, Grease, Thermal.

1. INTRODUCTION

Lubricating greases are frequently used as lubricants for journal bearings and rolling element bearings. During the last years, a lot of researches regarding the performances of grease lubrication were performed, due to the necessity to predict the life of bearings.

By comparison with oil lubrication processes, where hydrodynamic theory can be applied with certain confidence, in the case of lubricating greases there are some difficulties in order to predict the film thickness using elastohydrodynamic theory. The explanation consist in the insufficient understanding of the mechanism of lubrication and the complexity of the rheological properties of greases [1, 2].

Lubricating greases are colloidal systems which consist in a soap thickener mixed with synthetic or mineral oil, presenting non-Newtonian characteristics. Therefore, the use of performant rheometers has become a challenge for researchers to find a suitable methodology for evaluating the rheological behavior of these lubricants [3–5].

The purpose of this paper is to investigate the rheological properties of a lithium soap-based grease, using a Brookfield viscometer [6]. An optimized methodology was proposed, applicable to the tested grease, which takes into account the possibility of reducing the testing time by analyzing the degree of measurements repeatability.

The tested grease, based on lithium soap, is produced in Romania, has a smooth texture, it is very adhesive and also is water-resistant [7]. It has a very high load carrying capacity and presents reduce wear rates, due to the antiwear additives specific to the grease content. The main characteristic parameters of the grease are presented in Table 1.

Table 1. Physical-chemical properties of the lithium soap-based grease [7].

Soap thickener	Penetration at 25^{0}C, 1/10 mm^{-1}	Dropping point, ^{0}C	Temperature range, ^{0}C	Testing results on four ball machine	
				Maximum load, N	Wear diameter, mm
Lithium	260-300	185	-25 … 110	2400	1.7

2. STAND AND METHODOLOGY

The experimental test stand is a Brookfield CAP 2000+ viscometer, the components of which are

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presented in principle in Figure 1 [6]. It is a rotational viscometer that measures the flow behavior and viscosity of liquid and semisolid materials, with working geometry Figure 2.

The whole system is controlled by a specialized software called CAPCALC 32, which has the ability to control the stand, to acquire and process the data. The software has the possibility to determine parameters of the rheological model of the lubricant analyzed in the hypothesis of the validity of five rheological models:

- Bingham model: $\tau = \tau_0 + \eta\dot{\gamma}$; (1)
- Casson model: $\tau^{1/2} = \tau_0^{1/2} + (\eta \cdot \dot{\gamma})^{1/2}$ (2)
- Herschel-Bulkley model: $\tau = \tau_0 + m\dot{\gamma}^n$ (3)
- Power law model: $\tau = m\dot{\gamma}^n$ (4)
- Newtonian model: $\tau = \eta\dot{\gamma}$ (5)

Figure 1. Brookfield viscometer – general view [6]

Figure 2. Brookfield viscometer – working geometries [6]

The viscometer also has the possibility to perform thermal determinations, namely the variation of rheological parameters with temperature on the 10–75 °C range.

In order to determine the rheological model for the analyzed lubricant, a "velocity imposed gradient" type test was used at a constant temperature using a working geometry (cone 5) with the dimensions shown in Table 2. As variants of proposed rheological models were the Bingham model (Eq. 1) and the Herschel-Bulkley model (Eq. 3), their parameters being determined by the regression analysis method [8].

Table 2. Geometry and viscosity measuring range corresponding to the viscometer cones [6].

Cone number	Cone radius, mm	Top cone angle, grd	Range of viscosity, Pa.s	Range of shear rate, s^{-1}
5	9.53	1.8	0.333 ... 7.50	10 … 2200

In order to determine the variation of the shear stress and viscosity with temperature, similar tests were performed, such as the "velocity imposed gradient", but for a temperature range, using cone number 5 as working geometry.

The experimental results on the rheological temperature variation were processed numerically with a program developed in MathCAD, starting from the Reynolds model, for viscosity and yield stress:

- viscosity variation with temperature: $\eta = \eta_{50} e^{m_\eta (t-50)}$ (6),

where: η – viscosity;
η_{50} – viscosity at 50 °C;
m_η – temperature parameter;
t – temperature.

- yield stress variation with temperature: $\tau_0 = \tau_0^{50} e^{m_\tau (t-50)}$ (7),

where: τ_0 – yield stress;
τ_0^{50} – yield stress at 50 °C;
m_τ – temperature parameter;
t – temperature.

3. EXPERIMENTAL RESULTS

The experimental tests were performed in three steps:

Step 1: *Determination of the parameters of the theoretical rheological model that approximate with the highest level of confidence the experimental behavior of the lubricating grease.*

In this case, the test performed consisted of a load from the 16.66 s^{-1} to 3333 s^{-1} shear rate gradient, followed by an unload in order to highlight the thixotropy of the lubricant – "shear memory ». The reference temperature at which the tests were performed is 20±0,1 °C and the duration of homogenization (soaking time) of the sample at a certain shear rate was 3 min 10 sec. The measurements were made in 39 points and the total duration of the data acquisition was approximately 2 hours.

From the analysis of the experimental data, it is found that the rheological model that approximates exactly the behavior of the lithium grease is the Bingham model. The reogram obtained in this case is presented in Figure 3, and the values of the rheological parameters are centralized in Table 3, first line.

Step 2: *Determination of the degree of repeatability of the measurement of the parameters of the theoretical rheological model corresponding to the lubricating grease.*

For this case, the testing performed consisted of 3 repeated loading / unloading cycles, from a 16.66 to 3333 s^{-1} shear rate, with the acquisition of a total of 117 experimental points over a period of approximately 6 hours. The reference temperature at which the tests were performed is 20±0,1 °C and the duration of homogenization of the sample at a particular shear rate was 1 min 10 sec.

Analyzing the experimental data presented in Figure 4, it is found that all three curves obtained by repeating the test on the same sample of material are almost superimposed. This observation confirms the repeatability of the measurements. The values of rheological parameters obtained in this case are centralized in Table 3, second line.

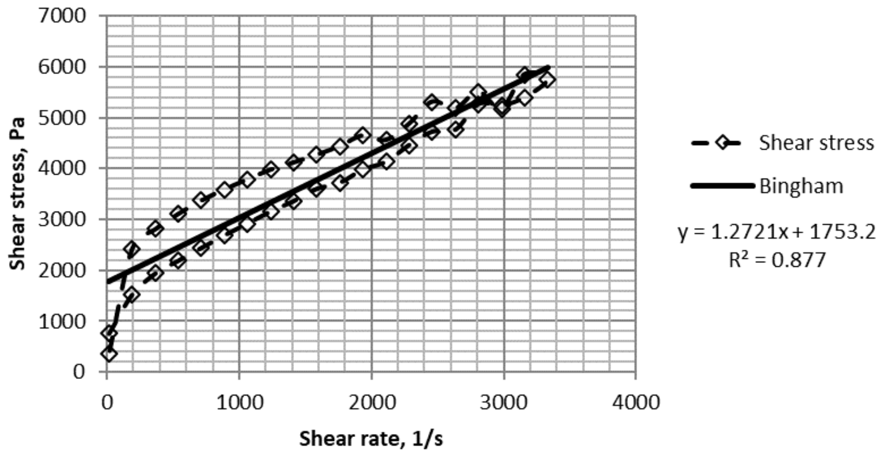


Figure 3. Lubricant rheogram for lithium grease, for 39 measuring points during approx. 2 hours.

Table 3. Rheological parameters of the lithium grease.

Number of measuring points / testing time \ Rheological parameters	Yield stress, Pa	Viscosity, Pa.s	Correlation coefficient
39 points / approx. 2 hours	1753	1.2721	87.70%
117 points / approx. 6 hours	1962	1.3345	88.20%

Comparing the results presented in Table 3, it can observe that reducing the test time three times leads to close values of viscosity and yield stress, resulting in a decrease of only 1% of the correlation coefficient of the determinations. But from an economic point of view, testing costs are almost half-life.

This observation is very important for the thermal tests, because it reduce the test time from 6 hours to 2 hours for a single temperature value, with cost reductions of almost half.

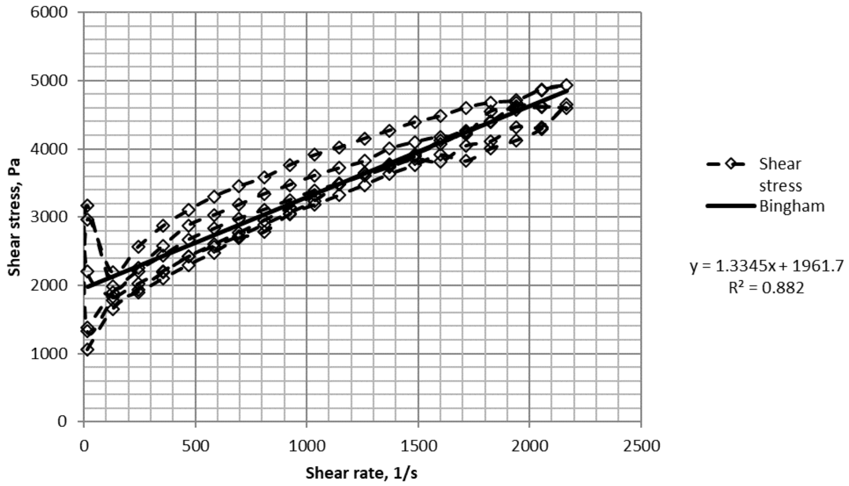
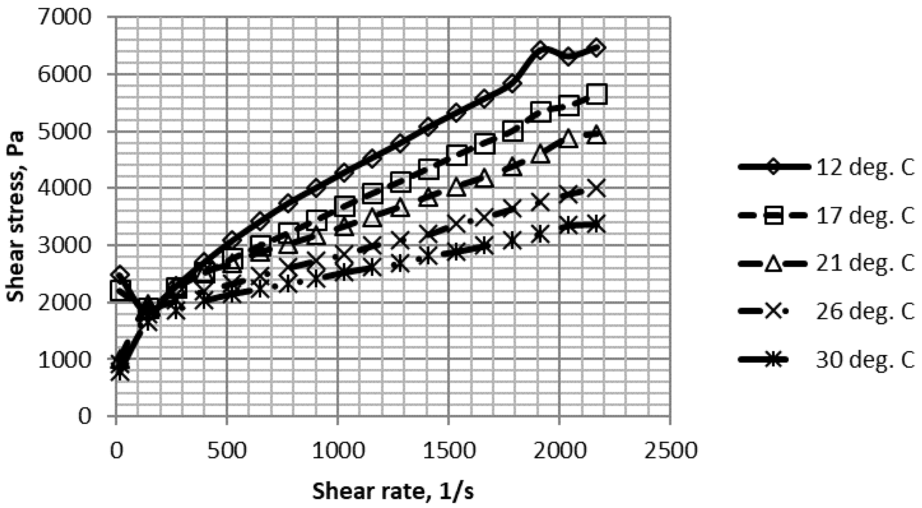


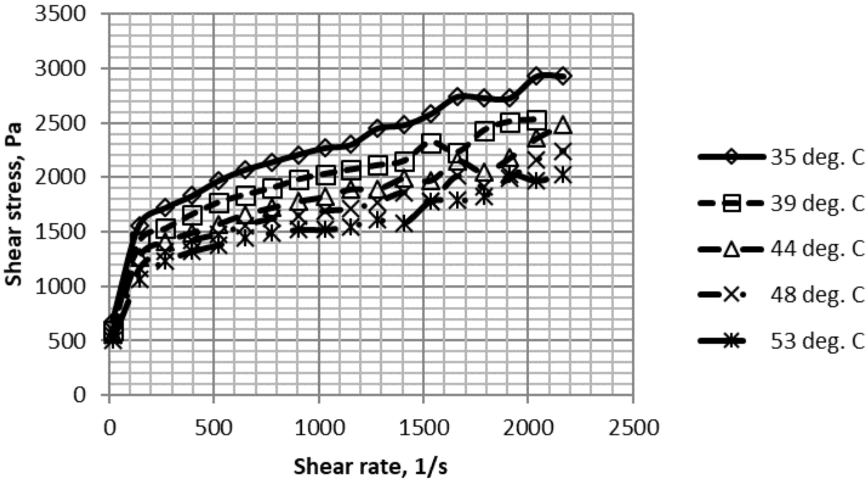
Figure 4. Lubricant rheogram for lithium grease, for 117 measuring points during approx. 6 hours.

Step 3: *Determination of the temperature variation of the parameters of the rheological model of the lubricating grease.*

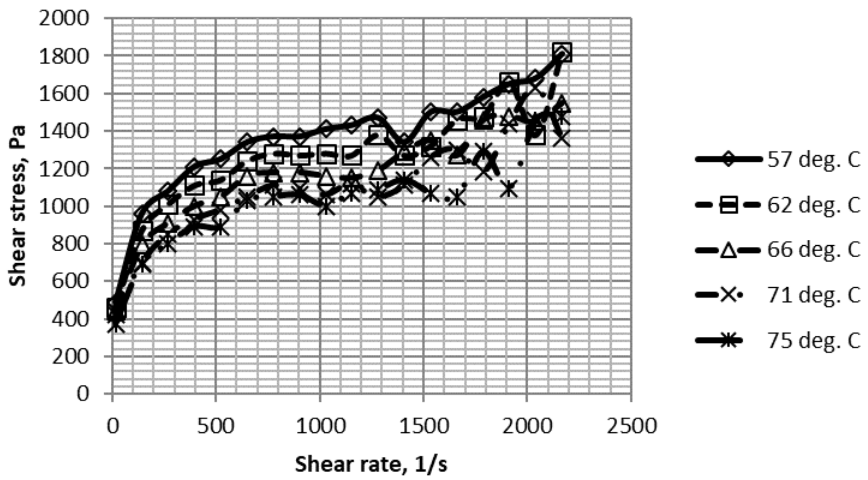
For this case, the test performed consists of one loading / unloading cycle, from a 16.66 s^{-1} to 3333 s^{-1} shear rate, with the acquisition of a total of 39 experimental points over a period of approximately 2 hours, repeated for 15 measuring temperatures, on a temperature range 10–75 ºC. Comparative curves are presented in Figure 5 a, b and c.



a) Temperature range 12–30 ºC.



b) Temperature range 35 ºC ... 53 ºC.



c) Temperature range 57 ºC ... 75 ºC.

Figure 5. Lubricant rheogram for lithium grease corresponding to thermal tests.

By performing the experimental results presented in Figure 5 a, b and c by the regression analysis method [9], rheological parameters characteristic of investigated lithium grease (yield stress and viscosity) for the whole temperature range (10–75 ºC) can be determined. The values obtained are centralized in Table 4.

Table 4. Rheological parameters of the lithium grease corresponding to whole temperature range.

Temperature, ºC	Bingham model		
	Yield stress, Pa	Viscosity, Pa.s	Correlation coefficient
12	1946	2.1928	98.18%
17	1864	1.7652	99.32%
21	1702	1.5413	96.27%
26	1576	1.1693	94.67%
30	1485	0.9296	90.65%
35	1394	0.7746	86.57%
39	1253	0.6672	84.79%
44	1129	0.6064	85.61%
48	1073	0.5373	84.04%
53	979	0.5083	84.96%
57	927	0.3942	78.71%
62	842	0.3833	76.78%
66	768	0.3682	84.21%
71	693	0.3641	81.06%
76	664	0.3344	77.21%

Tables 5 and 6 show the values of the characteristic parameters corresponding to the Reynolds model of viscosity variation and yield stress with temperature (Eq. 6 and 7) for the analyzed lithium grease.

Table 5. Characteristic parameters of viscosity variation with the temperature for analyzed lithium grease.

Parameter	Value
Viscosity at 50 ^{0}C (η_{50}), Pa·s	0.5373
Temperature parameter (m_η), $^0C^{-1}$	-0.034
Interpolation correlation coefficient	97.06%

Table 6. Characteristic parameters of yield stress variation with the temperature for analyzed lithium grease.

Parameter	Value
Yield stress at 50 ^{0}C (τ_0^{50}), Pa	1073.3
Temperature parameter (m_τ), $^0C^{-1}$	-0.0162
Interpolation correlation coefficient	98.84%

The results presented in Tables 6 and 7 are very important for the study of the friction couples lubricated with greases, functioning in hydrodynamic conditions. The integration of Reynolds equation that governs hydrodynamic lubrication processes, along with the energy equation, inevitably leads to the thermal approach of the phenomenon. Hence the necessity of knowing the variation laws of the yield stress and the viscosity with temperature, the literature being very poor in such information.

4. CONCLUSIONS

1. From a rheological point of view, the considered lithium grease 2 is modeled most accurately by the Bingham model.
2. The lithium grease shows a reduced thixotropy, regardless of the number of cycles it has been subjected to, for the entire range of shear rates investigated.
3. The repeatability of the tests at 20 ^{o}C is an indicator of the degree of homogeneity of the analyzed structure of the grease as well as a quality indicator of the product.
4. For the lithium grease it is found that the Reynolds model of viscosity and yield stress variation with temperature approximate with the same accuracy the experimental values, leading to correlation coefficients above 95%.
5. The proposed testing methodology is validated for the analyzed lithium soap-based grease, leading to reduced experimentation costs at almost half.

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