

# The Innovative Research Methodology of Tribological and Rheological Properties of Lubricating Grease

R. Kozdrach<sup>a,\*</sup>

<sup>a</sup>Research Network - LUKASIEWICZ – Institute for Sustainable Technologies, Radom, Poland.

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Tribological properties  
Friction factor  
Limiting load of wear  
Rheological properties  
Mean Square Displacement  
Storage modulus  $G'$   
Loss modulus  $G''$

## \* Corresponding author:

Author: Rafal Kozdrach   
E-mail:  
[rafal.kozdrach@itee.lukasiewicz.gov.pl](mailto:rafal.kozdrach@itee.lukasiewicz.gov.pl)

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## ABSTRACT

*The article presents the methodology of tribological and rheological research of typical lubricating composition.*

*The analysis of friction factor, friction work, frictional force, frictional power values during the test for the tested lubricating composition indicates a significant change in this parameters for lubricants. A change of the value of evaluated tribological parameters leads to a change in the structure of the composition carried out tests and a change in the effectiveness of the tribological protection of the tribosystem. The content of the thickener, base of oil and additive in the lubricant structure affects the level of the anti-wear properties, as evidenced by the results obtained during the tribological tests presented in this article.*

*The DWS (Diffusing Wave Spectroscopy) technique provides the ability to obtain information about the viscoelastic state of non-Newtonian liquids and provide the monitoring of structural changes in time. The observation of the movement of the dispersed phase particles and the evaluation of the microstructure change in the solutions based on the results of the correlation function MSD (Mean Square Displacement) in time, complex viscosity, complex modulus and the research of the  $G'$  and  $G''$  modulus in wide frequency range provides a comprehensive microstructure characteristic of the tested systems at the microscopic level.*

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## 1. INTRODUCTION

The problem of the analysis of changes in the structure of dispersion systems as a result of the interaction of individual components forming the dispersion, despite the fact that it is the subject of many studies, is not sufficiently recognized. The literature of the subject

describes the research results characterizing the rheological properties of heterogeneous and homogeneous dispersions with a relatively low content of dispersed phase [1-3]. However, there are no results characterizing the changes of the microstructure of substances with an increased content of the dispersed phase, and, in particular, the evaluation of lubricants.

The diffusing microrheology DWS (Diffusing Wave Spectroscopy) as an analytical instrument is used to test non-Newtonian fluids in a wide range of frequencies and viscoelasticity. The DWS technique provides the measurement of the dynamics and local intermolecular displacements and is based on the assumption that light transport in the media consists in the diffusion of particles in the dispersion phase.

The DWS method provides the test of rheological properties in a wide frequency range from 0.1-1.0 MHz and temperature of 283-363K. The interpretation of the obtained modulus  $G'$  and  $G''$ , especially in the case of polydispersion systems, in the linear viscoelasticity range is a very complex problem based on the dispersed phase displacements.

The optical microrheology is widely used to determine the dynamics of particle movement, changes in the structure and mechanical properties of emulsions, and to evaluate the physical stability of pharmaceutical emulsions based on the value of MSD (Mean Square Displacement) function in time, as well as changes in  $G'$  and  $G''$  values from frequency to explain destabilization processes of emulsion [1,2, 4-6].

Using the DWS technique, tests were also carried out that, provided a series of interesting sets of data about the movement of particles in concentrated fluids, such as colloids and microemulsions [7] and about the heterogeneity of biopolymer materials [8-10]. It was observed that the evolution of microstructure and mechanical properties of different types of gels is based on the changes in the value of correlation function MSD [11-15]. The rheological characteristics and microstructure of submicron emulsions deposited on a modified matrix based on waxy starch were determined. The change in microstructure of tested emulsions were evaluated and the mechanism of structure destabilization was confirmed [16]. By means of optical microrheology, the sizes of colloidal particles were measured which determined the gelation point, described the process of destabilization of colloidal systems as well as the process of the aggregation of particles and monitoring their displacement [17,18,24]. The rheological properties of micellar fluids and colloidal dispersions, polymers, and biomaterials,

on the basis of changes in the value of the MSD function over time were evaluated, and the relaxation modes of adsorbed polymers also were evaluated [19,20].

The rheological properties of high-viscosity silicone oil were also evaluated using the DWS technique. The kinetic change in structure was evaluated and a mathematical model describing viscoelastic properties was proposed [21-24,25]. So far, there have been no reports regarding the evaluation of the lubricants microstructure of the DWS method, including greases with a plastic consistency containing a dispersed phase in the form of thickener and oil as a dispersion phase. The evaluation of the stability of the lubricant on the microscopic level with the possibility of the elucidation of the instability of the microstructure has not been possible till now. The analysis of the previously described cases of the application of diffusing rheometry DWS provides, on ascertainment, that it is possible to identification and elucidation of changes declining in the microstructure of plastic lubricants and to correlate them with the viscoelastic states.

The analysis of phenomena occurring during the contact of the lubricant with machine elements in the surface layer, especially in the border layer have a great importance for the lubrication process [26]. The lubricating grease near of the machine construction elements creating a separate surface phase called the border layer. When in the range of action of the forces of machine elements are located the lubricating greases the interaction of these forces causes a greater arrangement of individual components of the lubricating grease, which was associated with an increase of intermolecular interactions between the lubricating composition and machine construction elements [27]. As a result of these interactions, a border layer are creating, which prevents of wear and friction processes. To effectively protect of device components before excessive wear, the phenomena between the lubricating composition and device components must be known. The general rules describing the interaction between lubricants and machine elements in the border layer were established using model substances, while the problem of testing border layers as a result of the influencing of real lubricating greases was not resolved [28,29].

The issues regarding the surface and areas of the interface between the surface layers have long been the subject of research in many scientific disciplines. The surface layer was a series of particles contained between the external and internal surface of the tested medium, which is the border of significant changes of physicochemical and structural properties. The elements of the testing substance (atoms, molecules) seek to achieve the minimum value of energy or enthalpy. In the absence of external forces, the solid fluid takes the form of a geometrically flat border surface with low surface energy. The energy of the fluid surface decreases with the reduced number of molecules on the surface in relation to the total number of molecules [30-32]. The surface particles from the outside of the fluid are subjected to force interaction, which differs significantly from the effect on the particles of the interior. The resultant force of pull of the surface molecule and the particles of several layers under the surface were directed inside the fluids. Since the chemical and physical properties of the testing substance depend on the value and direction of the force reaction of atoms and molecules, therefore the surface layer will determine the area from the surface deep into the fluid characterized by variability of properties or force effect. The presence of interaction forces on the surface causes the occurrence of surface energy. The energy increases the thermodynamic and chemical instability of surface atoms, leading to a decrease of system energy [40,42]. Under normal conditions, the fluid surface is in contact with the liquid, solid or gas phase. The surface energy, potential differences and other factors contribute to the solid-condition interface between physical and chemical reactions. A physically clean surface is covered with surface particles or reaction products. Therefore, the surface layer is defined as the area bordered from the outside with particles adsorbed on the surface, and from the inside with the surface of particles that differ significantly in energetic and properties from deeper located particles [33,38,41].

Therefore, the properties of the surface layer will depend on the type, configuration and energy state of the fluid molecules lying on the surface as well as under the surface, and the type and intensity of condition interactions. The

plastic deformation zone of the surface layer was created as a result of mechanical loads caused by the friction process. The deformation begins to evolve after reaching of critical stresses. These deformations were created when separate particles of network change their position, connection with other particles disappears, and appear the related with other elements. By changing location, the molecule effects on its direct conditions, leading to structure disruptions and increasing the number of structure defects, especially dislocations. Any infraction of the solid structure generally carrying on to its strengthening and increasing the resistance of plastic deformation. The depth of covering of plastically deformed layer for typical friction conditions doesn't exceed 60 nm [38-39]. As a result of plastic deformations of the surface layer, the area of own stresses was formed, whose distribution depends on the type of interactions, and the size depends on the force of interactions and the properties of the material surface layer. The identification of surface layer parameters such as: quantitative and qualitative analysis of the chemical composition, identification of microstructure of fluids, geometric structure of the surface and the degree of plastic deformation; size and state of stress, the degree of strengthening and thermal properties was an extremely important element in the analysis of the state of the surface layer [34,39,43].

The aim of the work was to discuss the methodology of the tribological tests on a rheometer with a tribological cell, the methodology of rheological tests on the DWS optical rheometer and to discuss of structural changes using the DWS technique resulting after the tribological tests on the example of a lubricating grease produced on a vegetable oil base.

## **2. METHODOLOGY OF RESEARCH**

To check the methodology of tribological tests was used the lubricating composition, which was prepared using non-toxic ingredients that are a dispersing and dispersed phase [35-37]. As a dispersing phase, vegetable oil with very good physicochemical properties was used. The Abyssinian oil, which was used in tribological tests, is characterized by the following physicochemical properties: 0.906–

0.911 g/cm<sup>3</sup> density; 48.20 cSt kinematic viscosity at 40 °C; 2.80 meq O<sub>2</sub>/kg peroxide number; 83.48 g I<sub>2</sub>/100 g iodine number; 170.50 mg KOH/g saponification number; and 0.34 mg KOH/g acid number. The Abyssinian oil consists of over 50 % of erucic acid (C22-1), which determines its qualities. It is an ultra-light oil, has a low viscosity, high absorbability, very good antioxidant properties, and characterized of excellent sliding, very good lubricating properties and high chemical resistance [44-46].

Lithium stearate was used as the dispersed phase. In the early phase of the experiment, research was carried out on the amount of the thickener that should be incorporated into the lubricating composition.

The lithium stearate used as a thickeners was introduced to the vegetable base oil in an amount of 8 % m/m. The lubricating composition prepared in this way were subjected to tribological and rheological tests.

To determine the tribological (anti-wear) properties of the tested lubricating composition, was used a compact MCR 102 rotational rheometer of the Anton Paar company with tribological cell T-PTD 200 (Fig. 1) with a concentric plate-ball contact point, in which three fixed cuboid steel plates were pressed with adequate force through a ball fixed in the spindle, rotating at the appropriate speed. The tribological device enables the execution of tests in the temperature range of -40÷200 °C. The balls with a diameter of 12.7 mm and plates with dimensions of 15 × 5 × 2 mm were made of bearing steel LH 15 (Ra = 0,3 µm; hardness 60–63 HRC). During the tests, immersion lubrication was used. Tribological tests (measurement of limiting load of wear -  $G_{oz}$ ) were carried out at the tribosystem at 10.00 N, rotational speed of 500 rpm, during 3600 s and temperature of 20 °C [47]. The parameters i.e. friction factor, friction work, frictional force and frictional power were also measured during the experiment. Before starting the tribological attachment, the plates were placed in the holder, pressed with springs, lubricant was introduced (approx. 5 cm<sup>3</sup>) and stabilized for 60 s at a set temperature. During the test, the tribological parameters was recorded, which was automatically converted into a coefficient of friction at 36-second intervals. Three test runs

were performed, and the final test results were averaged. The final result of the run was the value of 100 measurements registered during the test. The averaged results obtained during the tests were also given. For statistical processing of the results, the Q-Dixon test was used with a confidence level of 95 %. After testing, the components of the tribosystem were dismantled, washed with n-hexane and dried.

The limiting load of wear is a measure of the anti-wear properties of the lubricating composition. The determination of this parameter consisted in calculating its value in accordance with the formula (1):

$$G_{oz} = 0.52 * \frac{P_n}{d_{oz}^2} \quad (1)$$

where:

$P_n$ —load of the tribosystem equal to 10.00 N,

$d_{oz}$ —the diameter of the diathesis formed on the steel plates used for the test.

The optical microscope was used to determine the size of the trace of wear of the surface of rectangular test plates. The obtained results were used to determine the size of  $G_{oz}$ , i.e. the evaluation of anti-wear properties of lubricating composition subjected to tribological tests [48,49].

The friction factor value are calculating in accordance with the formula (2):

$$\mu = \frac{F_R}{F_L} \quad (2)$$

where:

$F_R$ —friction force,

$F_L$ —normal load.

The friction force are calculating with the formula (3):

$$F_R = \frac{M}{r * \sin \alpha} \quad (3)$$

where:

$M$  – torque,

$r$ — radius of friction force.

While the normal load are calculating with the formula (4):

$$F_L = \frac{F_N}{\cos \alpha} \quad (4)$$

where:

$F_N$ —normal force.

The sliding speed are calculating with the formula (5):

$$v_s = \frac{2\pi}{60 \cdot n \cdot r \cdot \sin \alpha} \quad (5)$$

where:

r- radius of friction force,  
n- number of rpm.

The sliding distance are calculating with the formula (6):

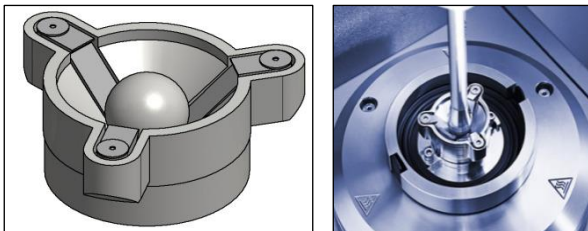
$$S_s = \varphi \cdot r \cdot \sin \alpha \quad (6)$$

where:

r- radius of friction force,  
 $\varphi$  – correlation coefficient.



a) The compact rotation rheometer MCR 102



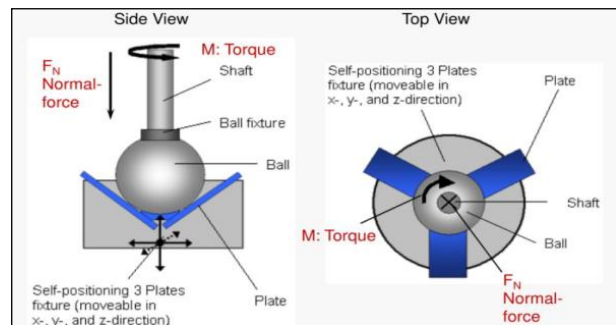
b) The model of tribosystem of rheometer MCR 102 [49,50]

Insert		Append		Delete...	
MCR102 BC12.7-SN27280		1  2 Pts. 30 s		2  200 Pts. 18 s	
Rotation $\dot{\gamma}, n, \varphi, \gamma$		n 1..500 1/min		n 500 1/min	
Rotation $\tau, M$					
Oscillation $\varphi, \gamma$					
Oscillation $\tau, M$					
$F_N$		$F_N$ 1..10 N		$F_N$ 10 N	
$d, v, d/d$					
T-PTD200-SH81146307		Accessory1 T		T 120 °C	

c) The rules of tribology measurements of rheometer MCR 102 [49,50]

Meas. Pts.	Temperature [°C]	Torque [mNm]	Sliding Distance [m]	Friction Factor [N]	Frictional Force [N]	Normal Load [N]	Friction Power [W]	Friction Work [J]	Shear Stress [Pa]	Normal Force [N]	Speed [mm/s]
Data Series Information											
Name:	60MIL_BA_test 1h-120C 1										
Operator:	NoLogin										
Number of Intervals:	2										
Application:	RHEOPLUS02 V3.61 21005880-33024										
Device:	MCR102 SH81146307, PW3.64, SH81146307, T1, AS(14.0d)										
Measuring Date/Time:	2018-02-26 11:19										
Measuring System:	BC12.7-SH82823, d=0 mm										
Accessories:	T1=T1-PTD200-H4-PTD200-SH81146307-8880599										
Calculating Constants:											
Cor [mm/s]	0.00074035488										
Cor [mm/s]	0.00070716679										
Start Delay Time [s]	305.957										
Substance Density [mg]	1.000										
Measurement Type:	14										
Axial Compliance [mN]	1E-6										
Position [mg]	1E-6										
Interval:	1										
Number of Data Points:	60										
Time Setting:											
60 Meas. Pts.											Meas. Pts. Duration 3 s
Measuring Profile:											
Speed	n = 0.1 .. 500 1/min in										
Normal Force	FN = 0.1 .. 50 N in										
Temperature	T [1] = 20 .. 120 °C in										
Meas. Pts.	Temperature [°C]	Torque [mNm]	Sliding Distance [m]	Friction Factor [N]	Frictional Force [N]	Normal Load [N]	Friction Power [W]	Friction Work [J]	Shear Stress [Pa]	Normal Force [N]	Speed [mm/s]
1	20	0.854	4.45E-7	0.113	0.146	1.29	7.12E-7	8.47E-8	0.00482	0.813	0.8021

d) The rules of tribology measurements of rheometer MCR 102 [49,50]



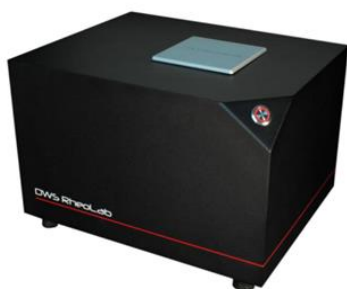
e) The rules of tribology measurements of rheometer MCR 102 [49,50]

**Fig. 1.** The view of triborheometer and rules of tribology measurements.

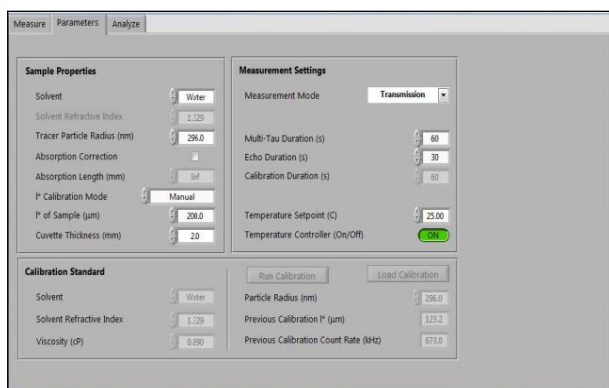
The research of rheological properties of the tested lubricating composition was carried out by means of the DWS RheoLab optical rheometer of the Swiss company LS Instruments AG (Fig. 2). This device uses Diffusing Wave Spectroscopy (DWS) for rheological characteristics of media, both solid and liquid, such as suspensions, emulsions, foams, lubricants, etc. [49-53]. The rule of operation of the DWS rheometer is based on the assumption that light transport can be treated as a diffusion process in optically turbid samples. The apparatus enables micro-rheological measurements of materials under static inter-molecular displacements in a wide range of frequencies and viscoelasticity. The optical rheometer enables testing in two different geometries, i.e. transmission and backscattering. In the transmission mode, scattered light is detected after passing through the sample, and intensity fluctuations are correlated using the correlation intensity function (ICF). In contrast, in the backscattering mode, the light that disperses back towards the incident beam is collected and its fluctuations are measured [54-56].

Before the measurements were commenced, the apparatus was calibrated using a model, i.e. emulsion of polystyrene with a particle size of 222 nm in water. Then, the refractive indices for the oil bases of the individual lubricating compositions, the duration of the measurement, the temperature and the size of the spectrometric cuvette in which the sample was placed were determined. The tested lubricating grease was prepared by adding a titanium dioxide marker with a particle size of 360 nm to their structure. The test sample was then homogenized and placed in a measuring cuvette. The rheological tests were carried out using a measuring cuvette with an optical path equal to 1 mm [57-59]. The duration of the measurement was 90 s.

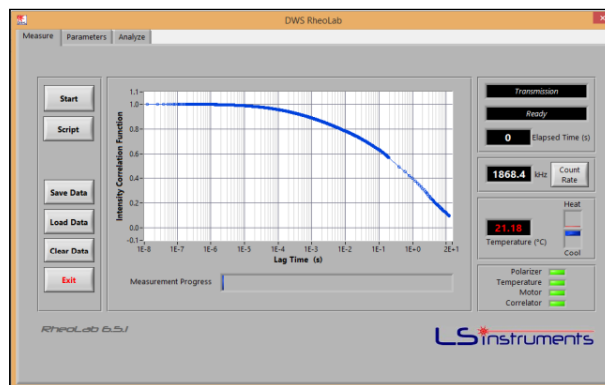
During the rheological tests, the correlation function of the MSD (mean square displacement of the molecule) from the time of delay, complex viscosity, the complex modulus  $G^*$  and the storage modulus  $G'$ , the loss module  $G''$  in static conditions were determined, depending on the frequency. The rheological measurements were carried out at the temperature of 20 °C. Based on the analysis of the determined rheological parameters, the change in viscoelastic properties of the investigated lubricating compositions was determined [48-51,57,59].



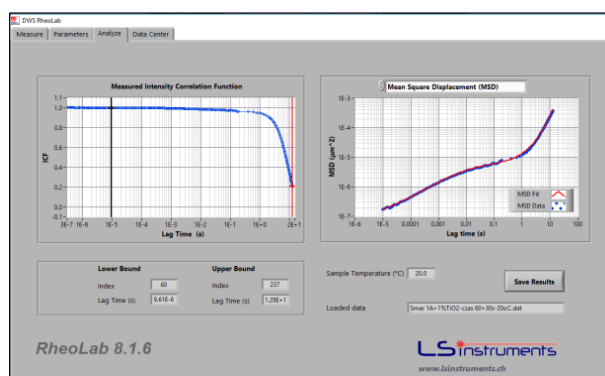
a) The optical rheometer DWS



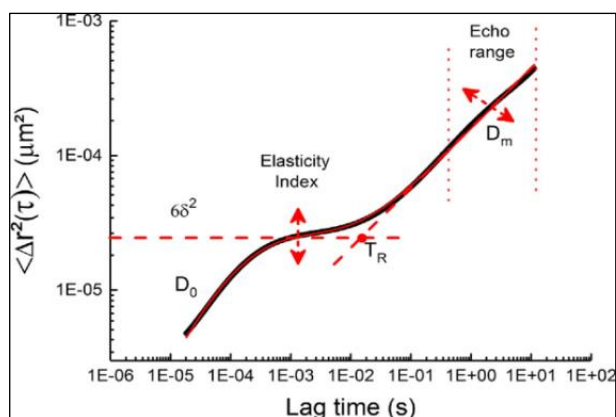
b) The rules of rheology measurements of optical rheometer DWS



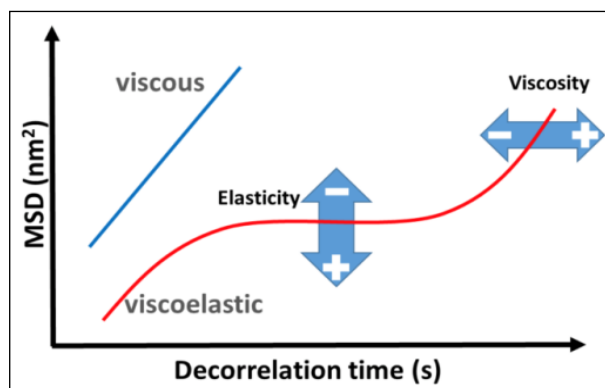
c) The rules of rheology measurements of optical rheometer DWS



d) The rules of rheology measurements of optical rheometer DWS



e) The and interpretation of MSD function [57-60]



f) The interpretation of MSD function [57-60]

Fig. 2. The view of optical rheometer and rules of rheology measurements



In each of the rheological tests, the condition was fulfilled, that the ratio of the cuvette thickness to the mean free path must be greater than 7 ( $\frac{L}{l^*} > 7$ ). The based on the analysis of the rheological parameters determined, the  $D_0$  and  $D_m$  diffusion coefficients were calculated at short and long delays of the MSD correlation function, the elasticity index EI and the slope of the MSD correlation function  $n$ .

The correlation function called the mean square displacement of the molecule MSD is determined on the basis of measurements of the dynamics of the displacement of the marker molecule in the tested fluid at specified intervals and is determined from the formula (7) [60,62,64,66]:

$$\langle \Delta r^2(t) \rangle = \langle [x(t + \theta) - x(t)]^2 + [y(t + \theta) - y(t)]^2 \rangle \quad (7)$$

where:

$x, y$  – the location coordinates of marker molecules,

$t$  – time,

$\theta$  – time between following marker positions.

The slope of the MSD function determines the nature of the sample under investigation. This parameter is determined from the formula (8) [63,64,67]:

$$\langle \Delta r^2(\omega) \rangle \sim \omega^n \quad (8)$$

where:

$\omega$  – frequency.

The slope of the MSD function is in the range of 0-1 for viscoelastic fluids, and 1 for Newtonian fluid, and 0 for a perfectly elastic fluid.

The modulus of elasticity EI is a measure of the elasticity of the tested sample and is calculated from the formula (9) [61,62,65]:

$$EI = \frac{1}{\langle \Delta r^2(\tau) \rangle_{max}} \quad (9)$$

This parameter is determined from the MSD function. This is the point at which the plateau characteristic of this function ends and the values of the correlation function start to increase.

The complex viscosity is the viscosity of the viscoelastic fluid depending on the complex module, temperature and frequency and is determined from the formula (10) [60,62,64,66]:

$$\eta^*(\omega) = \frac{G^*(\omega)}{\omega} \quad (10)$$

where:

$G^*$  – complex modulus,

$\omega$  – frequency.

The loss modulus  $G''(\omega)$  is a measure of the viscosity forces, because represents sinusoidal deformations proportional to frequency. This parameter can be described by the following formula (11) [60,62,65,66]:

$$G''(\omega) = G^*(\omega) \sin\left[\pi \frac{\alpha(\omega)}{2}\right] \quad (11)$$

where:

$G^*$  – complex modulus,

$\omega$  – frequency,

$\alpha(\omega)$  – the logarithmic derivative of the MSD function after time.

The storage modulus  $G'(\omega)$  is a measure of the elastic forces during cosinusoidal deformations proportional to frequency. This parameter can be described by the following formula (12) [60,61,64,65]:

$$G'(\omega) = G^*(\omega) \cos\left[\pi \frac{\alpha(\omega)}{2}\right] \quad (12)$$

where:

$G^*$  – complex modulus,

$\omega$  – frequency,

$\alpha(\omega)$  – the logarithmic derivative of the MSD function after time.

The  $D_0$  and  $D_m$  diffusion coefficients characterizing the behavior of suspended particles in liquids under the influence of variable temperature and concentration at short and long delay times of the correlation function MSD were determined from the Bellour equation (13) [67]:

$$\langle \Delta r^2(\tau) \rangle = 6\delta^2 \left(1 - \exp\left(-\frac{D_0}{\delta^2} \tau\right)\right)^{\frac{1}{\alpha}} - \frac{1}{\alpha} \left(1 + \frac{D_m}{\delta^2} \tau\right)^{-\beta} \quad (13)$$

where:

$\tau$  – lag time,

$\delta$  – the amplitude of particle movement,

$\alpha$  – the parameter characterizing a wide spectrum of relaxation times in a plateau with short delay times for MSD functions,

$D_0$  – the diffusion coefficient at short delay times of MSD function,

$D_m$  – the diffusion coefficient at long delay times of MSD function,

$\beta$  – the parameter characterizing changes in the slope of the MSD correlation function with long delay times describing the diffusion area of the tested sample.

### 3. RESULTS OF RESEARCH

#### 3.1 The rheological research

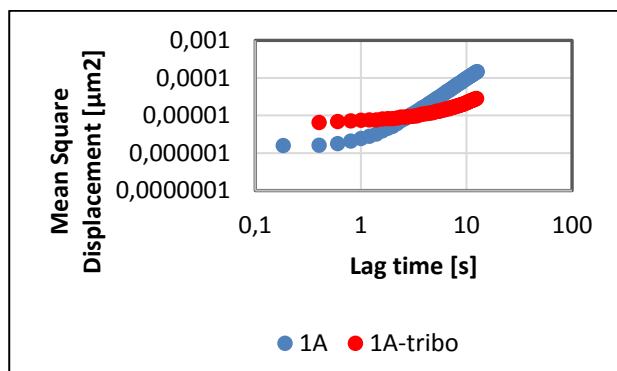
For tested vegetable lubricating greases were carried out the rheological properties before and after tribological tests, and then the evaluation what influence has a tribological tests on the change of rheological parameters. The obtained test results are shown in Fig. 3, and the calculated of rheological parameters were shown in Table 1.

**Table 1.** The calculated rheological parameters of tested lubricating greases.

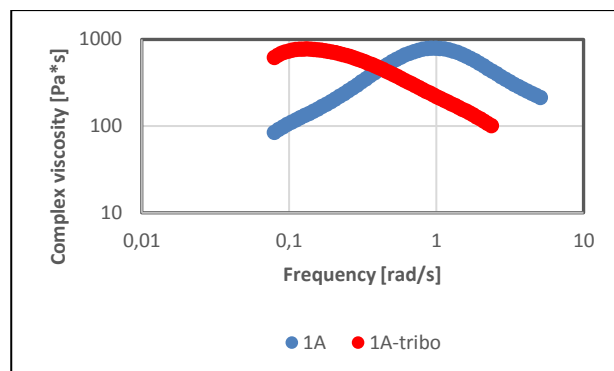
Tested lubricating grease	The storage modulus $G'$ [Pa]	The loss modulus $G''$ [Pa]	The complex modulus $G^*$ [Pa]
Lubricating grease	45,62	196,34	211,98
Lubricating grease after tribological test	754,11	262,78	802,33

Tested lubricating grease	The complex viscosity [Pa*s]	The slope of MSD function [-]	The elasticity index [ $\mu\text{m}^2$ ]
Lubricating grease	224,95	0,93	$2,51 \cdot 10^{-6}$
Lubricating grease after tribological test	811,67	0,92	$3,47 \cdot 10^{-6}$

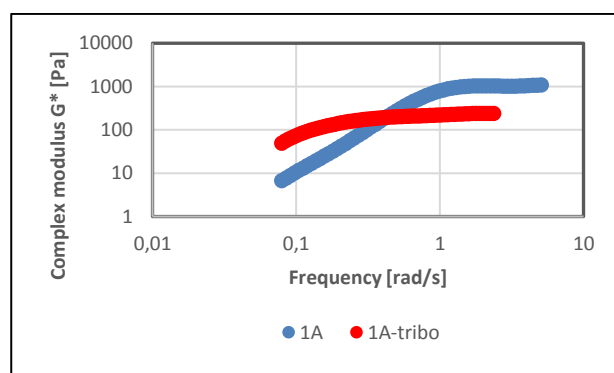
Tested lubricating grease	The diffusion coefficient $D_0$ [ $\text{m}^2/\text{s}$ ]	The diffusion coefficient $D_m$ [ $\text{m}^2/\text{s}$ ]
Lubricating grease	$5,56 \cdot 10^{-2}$	$7,23 \cdot 10^{-5}$
Lubricating grease after tribological test	$3,11 \cdot 10^{-2}$	$2,89 \cdot 10^{-5}$



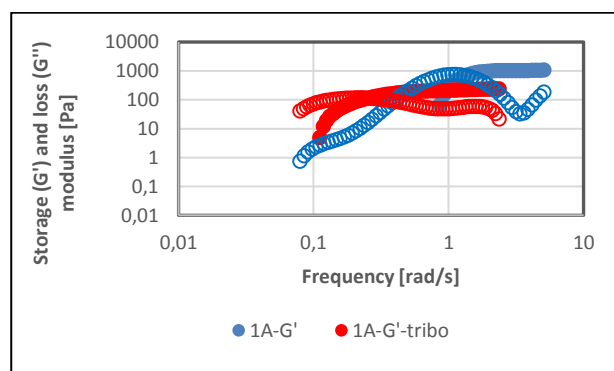
a) The results of dependence of MSD function from lag time for vegetable lubricating grease (blue – vegetable lubricating grease before the tribological tests, red- vegetable lubricating grease after the tribological tests).



b) The results of dependence of complex viscosity from frequency for vegetable lubricating grease (blue – vegetable lubricating grease before the tribological tests, red- vegetable lubricating grease after the tribological tests).



c) The results of dependence of complex modulus  $G^*$  from frequency for vegetable lubricating grease (blue – vegetable lubricating grease before the tribological tests, red- vegetable lubricating grease after the tribological tests).



d) The results of dependence of storage and loss modulus from frequency for vegetable lubricating grease (blue – vegetable lubricating grease before the tribological tests, red- vegetable lubricating grease after the tribological tests (● -  $G'$ , ○ -  $G''$ )).

**Fig. 3.** The results of rheology measurements.

For the tested grease, after the tribological tests was observed a change of rheological properties, which in consequence could lead to change of the structure of the tested grease. The base of



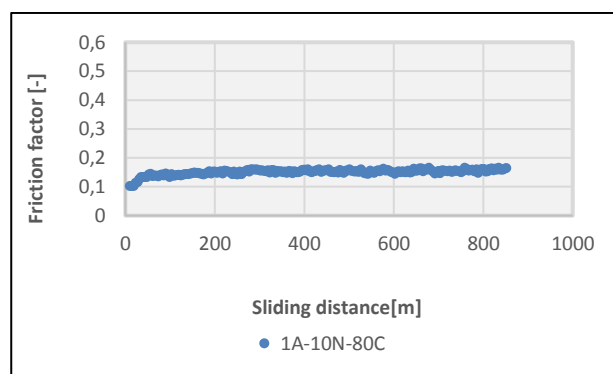
the evaluation were calculated rheological parameters for the grease before and after tribological tests. Analyzing the correlation function of MSD was observed, a decrease in the value of the slope coefficient of the discussed function for the composition after tribological tests in compared to the grease that wasn't subjected of tribological tests. The value of the discussed parameter provides that the tested grease is a viscoelastic fluid of low viscosity. The increase of slope coefficient of MSD function for the composition after tribological tests provides an increase of sample viscosity, as evidenced by the obtained test results. Such behavior of the tested grease could have been caused by changes occurring in its microstructure during tribological tests, which can carry on to destabilization of the structure and later loss of its functional properties by the grease. The results of the elastic, viscous and complex modulus tests prove, that after the tribological tests was occurred a change in the microstructure of the tested composition. A significant change of the value of the discussed modulus characterizing the structural stability of the lubricating grease, especially  $G'$  and  $G''$  after tribological tests, provides a change of the functional properties of the tested grease. For the tested grease from the MSD correlation function, the elasticity index EI was carried out, which the value was changed after tribological tests. This parameter characterizes the elastic forces in viscoelastic fluids. The smaller elasticity index value, the more stable the microstructure and the higher viscosity of tested sample. The decrease value of the elasticity index after tribological tests provides an improvement of stability and an increase the viscosity of the tested grease sample. The cause of this behavior of the analyzed grease was a oxidation processes occurring under the influence of increased temperature during the tribological test.

The rising oxidation products caused the increase of elastic forces and change the viscosity of tested grease, which carried out to changes in its microstructure. For the tested grease, the diffusion coefficient  $D_0$  was also calculated with very small values of the delay time of the mean square displacement function MSD before and after tribological tests. The obtained results allow to ascertainment, that the values of this parameter was subjected

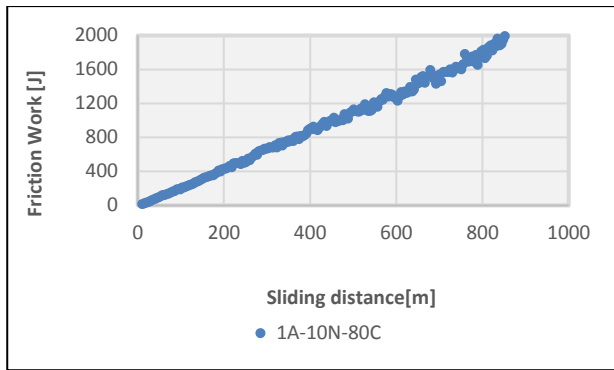
significantly change after tribological tests. The higher values of the discussed parameter were obtained for the grease after the tribological test, which provides a change in the nature of the tested grease sample. The higher value of the diffusion coefficient  $D_0$ , the slower moves of the particles suspended in the tested fluid, and the slower the dynamics of the motion of the particles can be connected with the increase of viscosity. The changes taking in the structure were caused a change of viscosity and also of operational properties of grease. The cause of this behaviour of tested sample may be the low resistance of the vegetable oil base on temperature changes. The products, which were creating can caused continuous progressive degradation of the oil base, which carried out to a deterioration of the quality of grease and losing their protective properties. The values of the diffusion coefficient  $D_m$  at high values of the delay time of the MSD correlation function were calculated by Bellour's equations. The obtained results allow to ascertainment, that the values of this parameter were subjected change after the tribological test. An increase of the value of discussed parameter after the tribological test provides a change of the structure, an increase of viscosity and stronger diffusion properties of the tested sample. As a result of structural changes, they change its properties, which decide about the possible application areas of the tested lubricating grease.

### 3.2 The tribological research

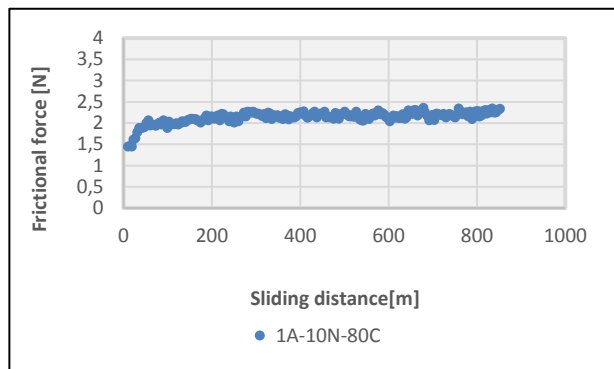
For tested vegetable lubricating grease was carried out the tribological properties, and then the evaluation tribological parameters of tested lubricating composition. The obtained test results are shown in Fig. 4 and Table 2.



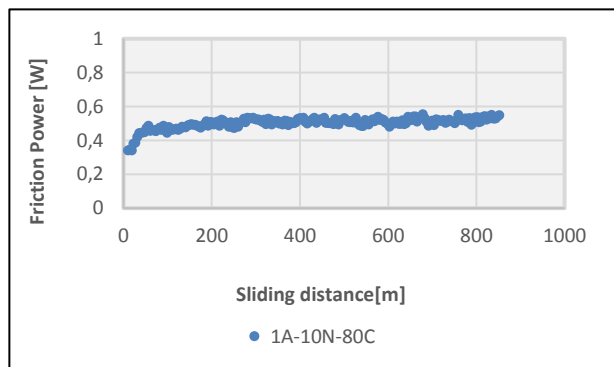
a) The results of dependence of friction coefficient from sliding distance of vegetable lubricating grease



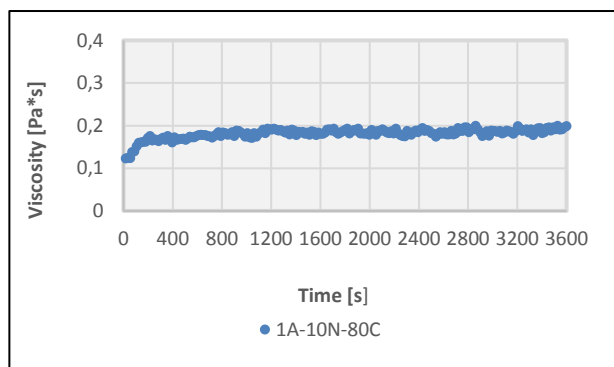
b) The results of dependence of friction work from sliding distance of vegetable lubricating grease.



c) The results of dependence of frictional force from sliding distance of vegetable lubricating grease.



d) The results of dependence of frictional power from sliding distance of vegetable lubricating grease.



e) The results of dependence of viscosity from time of vegetable lubricating grease.

**Fig. 4.** The results of tribology measurements.

**Table 2.** The calculated tribological parameters of tested lubricating grease.

Tested lubricating grease	The limiting load of wear $G_{02}$ [N/mm <sup>2</sup> ]	The average friction coefficient [-]	The average friction work [J]
Lubricating grease	494,8	0,13	0,47

Tested lubricating grease	The average frictional force [N]	The average friction power [W]	The average viscosity [Pa*s]
Lubricating grease	2,12	0,48	0,17

The methodology of tribological tests was discussed earlier provides a comprehensive evaluation of the tribological properties of the tested lubricating greases. The tribological tests carried out on the T-PTD 200 tribological adapter of MCR 302 rheometer are innovative, because they provides of measurements at small values of tribosystem (up to 50 N) with temperature control, which cannot be done using generally available tribological testers. Except of measurements of the friction coefficient depending on the sliding distance, sliding speed or time, it is possible to carried out the tribological parameters such as: friction work, friction power, friction force and dynamic viscosity of the tested medium in a wide range of shear rate or strain. The assignation of the above-mentioned tribological parameters, e.g. in the long time, provides an answer how lubricants behave under strictly defined conditions and how long they can work without losing their protective properties.

#### 4. CONCLUSION

The rheological properties of vegetable grease were carried out before and after tribological tests using DWS technique in a wide frequency range. It was observed a change of carried out rheological parameters after the tribological test, which provides a change of the microstructure of the tested grease sample. The designated values of rheological parameters obtained from the analysis of data of the MSD function, complex viscosity and modulus  $G'$ ,  $G''$  and  $G^*$  revealed correlations with the corresponding parameters calculated from the Bellour equation and will allow the evaluation of changes of the structure of the tested lubricating grease. A modified mathematical model was used, which showed flexibility in testing a wide

range of data, thus providing important view into the microstructure of the dispersion subject of tribological tests. It is particularly important, that DWS is a quick measuring technique, which is non-invasive and saves the tested material. This method can be used as a instrument to monitoring the quality of greases. Unlike other methods, the data from the MSD function not only provide knowledge about grease stability, but also carried on to a deeper understanding of the dispersion microstructure.

The tribological tests of the vegetable lubricating composition were also carried out on the MCR rheometer with the T-PTD 200 tribological cell in a wide range of sliding distance. The appointed values of tribological parameters such as: friction coefficient, friction work, friction power, friction force and dynamic viscosity provides a comprehensive tribological evaluation of the tested grease. The discussed method of testing tribological properties provides of testing of greases, oils, etc. at smaller loads of tribosystem in a wide temperature range. The assignation of above-mentioned of the tribological parameters provides the obtaining information about the behaviour of lubricants in various operating conditions and evaluation of the quality of the tested grease in long period of time.

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