

The Tribological, Physico-Chemical and Rheological Properties of Lubricating Grease Produced on Epoxy Resin Base

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ABSTRACT

The article presents the results of physicochemical, tribological, rheological, anticorrosive and oxidation tests of lubricating grease produced on the base of epoxy resin and thickened of amorphous silica Aerosil®. The tests for lubricating grease were carried out and then compared with the test results obtained for lubricants prepared on the mineral, vegetable and synthetic oil base.

The application of epoxy resin as a dispersion phase affects the increase in relation to compositions prepared on the mineral, vegetable and synthetic oil base of resistance of the top layer on scuffing and wear for tested lubricating compositions.

The application of the epoxy resin as a dispersion phase of lubricating greases demonstrated a synergistic interaction with the used thickener and a significant improvement of the dropping point in relation to the compositions produced on conventional oil bases.

The lubricating grease produced on the base of epoxy resin has a much longer oxidation induction time and better anticorrosive properties than lubricating compositions produced on other base oils.

The application of epoxy resin as a dispersion phase of lubricating greases caused a significant increase of dynamic viscosity from shear rate and in the range of low temperatures in comparison to compositions produced on conventional base oils.

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1. INTRODUCTION

The properties of lubricating greases are shaped by their structure, which results from the percentage of dispersion phase, thickener and modifying additives. The dispersion phase (base oil) usually determined 70-90 % of the volume of the final product and it is used together with the

thickening substance to produce the lubricating grease [1-3]. The base oils are of mineral origin (crude oil processing products), synthetic origin (chemical synthesis products of hydrocarbon and non-hydrocarbon oils) and vegetable origin [4,5]. As the main component of the lubricating grease, the base oil decides a series of useful properties of the obtained lubricating compositions. Its task

is to assure that greases have good lubricating properties, resistance to oxidation and temperature, appropriate rheological properties and minimal chemical reactivity (they should not react with other components of the grease) [6,7]. The growing requirements for the quality of base oils caused that it started the introduction of refining additives to improve their properties [8-10]. The production of lubricants with appropriate properties requires the use of components, in particular oil bases, which will provide the final product of very good useful properties, therefore it was decided to use a high-viscous epoxy resin as the dispersion phase of the tested lubricating composition.

The epoxy resins are chemically hardenable compounds, which molecules contain at least two epoxy groups. They are obtained by direct polycondensation of 1,2-epoxy-3-chloropropane (epichlorohydrin) with dihydroxy phenols or polyglycols in an alkaline medium. The epoxy resins are characterized by high viscosity (25 000 mPas), which is in many cases a factor hindering their use and processing, and molecular weight equal 430 u [11,12]. They are widely used in the automotive, aerospace, electronics and construction industries. They are used for the production of gliders, car bodies and yachts. They are also used as electro-isolating elements in electrical engineering and electronics. Epoxy powder paints are commonly used in the shipbuilding and machine industry. Another group of epoxy products are epoxy adhesives characterized by high adhesion. In the automotive, electrotechnical and electronic industries, products made of epoxy resins are used. They are a great material for insulation and encapsulation of current transformers, voltage transformers, rectifiers and capacitors. The epoxy resins are also used in architecture as sealing and flooring materials [13-15]. The Epidian 5 resin used as a dispersion phase of the tested greases is a low-molecular weight viscous liquid, that is a pure, dian epoxy resin, which doesn't contain solvents, diluents, fillers and other additives. The epoxy resins are characterized by good resistance on water and weather conditions. The compositions containing the epoxy resin have very good physical properties, such as: hardness, elasticity and abrasion resistance, good temperature resistance, excellent chemical resistance, very good rheological and tribological properties and very good adhesive properties [16-18].

The aim of the work was to examine the influence of epoxy resin as a dispersion phase of lubricating grease and to compare mineral, vegetable and synthetic oil base with the same quantity and type of thickener, on change of basic tribological, rheological, anti-corrosion and oxidation parameters for lubricating compositions developed at the Research Network LUKASIEWICZ - Institute of Sustainable Technologies in Radom.

2. EXPERIMENTAL DETAILS

2.1 Materials

A lubricating grease produced on the base of epoxy resin was used for the tests (grease A) and for comparison of lubricating greases based on high quality hydrotreated mineral oil (grease B) [19], based on refined vegetable oil (grease C) [20-25] and on the base of high-viscous synthetic oil (grease D) [26]. The information concerning the use of materials is included in Table 1. All lubricating compositions were thickened with 5 % of amorphous silica Aerosil®. The lubricating greases prepared in this way were tested of tribological, physico-chemical, rheological, anticorrosion and oxidation tests, and obtained results were compared with each other. For all obtained test results, statistical analysis was performed using t-Student.

Table 1. The composition of tested lubricating greases.

The symbol of lubricating greases	The type of base oil in lubricating greases	The type of used thickener in lubricating greases
A	Epoxy resin Epidian®5	Amorphous silica Aerosil®
B	Hydrotreated mineral oil FINAVESTAN A360B	Amorphous silica Aerosil®
C	Refined rapeseed oil	Amorphous silica Aerosil®
D	high-viscous synthetic oil SILOL 350F	Amorphous silica Aerosil®

2.2 Methodology of research

The tribological properties of the lubricating compositions were evaluated on a T-02 of four-ball machine. These properties were determined by measuring the limiting load of wear ($G_{oz/40}$)

and limiting pressure of seizure (p_{oz}) on a four-ball machine [27-31]. The friction pair to be tested on elements were the steel balls of diameter 12.7 mm, applied in steel bearing type ŁH 15. The roughness of ball surface was $R_a = 0.32 \mu\text{m}$ and its hardness was 60-65HRC [32-35]. The measurement of the limiting load of wear ($G_{oz/40}$) was carried out by tribosystem load of 392.4 N for entire test run lasting 3600 s. The spindle speed of rotating ball was set up at 500 rpm according to the WTWT-94/MPS-025 test method [36]. The measurements of lubricating properties in scuffing conditions (i.e. in the constantly increasing load during the tests) were carried out according to the methodology developed by ITeE – PIB. The test was performed in a linearly increasing load from 0 to 7200 N (ramp 409 N/s) within the time of 18 s at 500-rpm spindle speed. The moment of a sudden increase of the friction point is called the scuffing load P_t . The measurement was carried out until the point of friction reached the point of 10 Nm or the maximum load of device set up at 7200 N. This point was defined as the limiting load of scuffing P_{oz} [27-33,35,37-38].

The measurement of dropping point was carried out according to the PN-ISO 2176:2011 standard. The rule of determination was to designate the temperature at which the first drop of grease flows out of the test vessel during its uniform heating [31].

The oil separation from the grease test was carried out according to the PN-V-04047:2002 standard. The determination method consisted of determining the quantity of oil under static conditions, that separated from the grease in a nickel grid cone at 100 °C for 30 hours. The quantity of separated oil was given as a mass fraction expressed in percentages [31].

The lubricating grease penetration test was carried out according to the PN-ISO 2137: 2011 standard. The determination method consisted of measuring the gravitational depth of the immersion, the normalized cone in the test grease, at 25 °C, falling within 5 s. The penetration was expressed in the so-called penetration units (non-counted number corresponding to 0.1 mm of the cone immersion in the tested grease) [31,39-42].

For lubricating greases, the test of the corrosive effect on metals was carried out on copper plates

with dimensions of 75x12.5x1.5 mm (PN-85/C-04093- method A). At least 3 samples with different weights must be taken from places away from the walls of the grease vessel. The plates should be grinded before using. The rule of the method consisted on immersing the copper plate in a glass vessel with a diameter 70 mm and with a height 130 mm, in which the tested product (lubricating grease) was placed, so that its level after immersion there in the plate was 15-20 mm below the upper edge of the vessel. Then the vessel with the product was heated to the temperature of 70 +/-2 °C and left for 5 hours. The plates were observed in daylight at first perpendicularly, then at an angle of 45°. The evaluation was subjected to the change created on the surface of plate and were compared with the corrosion standard.

The test of anticorrosion properties of the dynamic method was carried out in accordance with the PN-ISO 11007: 2010 standard. The test was carried out on the EMCOR machine for testing lubricants of company SKF. The test used self-aligning ball bearings that were filled with the tested grease (after approx. 10 g for each bearing) and rotating at 80 rpm in the presence of deionized water (after 10 cm³ for each frame, in which the bearing with the grease was located). The test was carried out for one week for consecutive periods of working and stopping of test bearings: 8 h of work and 16 h stop, 8 h work and 16 h stop, 8 h work and 108 h stoppage.

The thermooxidative stability test was carried out using the PetroOxy apparatus. The sample of grease in the quantity of 5 ml was introduced into the test chamber of the device and oxidized with oxygen at a constant temperature of 120 °C. The filling pressure was 700 kPa and the oxygen pressure was 8 bar (800 kPa). The final result was the time necessary to obtain a 10 % decrease in the maximum pressure in the measuring chamber [43-46].

The tests of rheological properties were carried out using a Physica MCR 101 rotational rheometer (manufactured by Anton Paar), equipped with a diffusion air bearing, connected to pneumatic power supply - an oil-free Jun-Air compressor and an air-drying block. The device is equipped with Peltier temperature control system in the range of -40-200 °C and in the external thermostatic system VISCOTHERM V2,

working in the temperature range -20-200 °C. The rheometer control and analysis of measurement data were carried out using the Rheoplus software [46-47].

3. THE RESULTS OF RESEARCH

3.1 Results and discussion of tribological properties of the tested greases

The results of tribological tests (antiscuffing and antiwear properties) of lubricating compositions produced on epoxy resin and for comparison on mineral, vegetable and synthetic oil thickened with a modified silica Aerosil® are presented below. Three tribological tests were carried out for each lubricating grease and the error bar for the obtained tests was added.

For selected lubricating compositions, the antiscuffing properties under scuffing conditions, i.e. the limiting pressure of seizure p_{oz} were determined. The obtained results of test this parameter are presented in Fig.1.

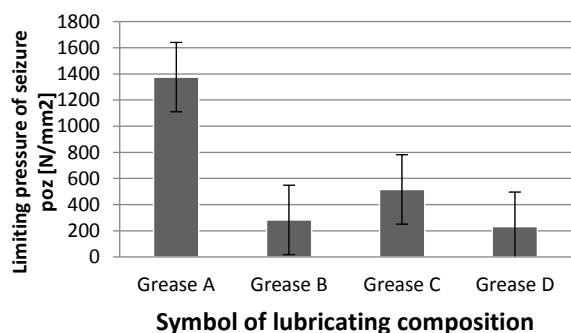


Fig. 1. The limiting pressure of seizure of tribosystem lubricated compositions produced on epoxy resin, mineral, vegetable and synthetic oils.

The determined values of the limiting pressure of seizure showed a varied level of antiscuffing properties of the tested lubricating compositions. The average friction coefficient for the tested lubricating greases was within 0.10 [-].

Depending on the type of dispersion phase used in the tested lubricating greases, the value of the parameter determining the level of antiscuffing properties was subject to significant changes. The highest value of the p_{oz} parameter was characterized by composition A produced with the participation of epoxy resin. In this case, the value of the parameter determining the level of antiscuffing properties was more than twice the

value of this parameter for the composition produced on the base of vegetable oil. The lowest value of this parameter was observed for grease D, which was produced on a synthetic oil base. The composition B produced on a mineral oil base characterized a similar value of limiting pressure of seizure p_{oz} as composition D. The value of the p_{oz} parameter for composition B produced on a mineral oil base and composition D produced on a synthetic oil base is, respectively, about 385.2 % and 494.6 % lower, and for composition C produced on a vegetable oil base is 166.3 % lower than the value of this parameter for composition A produced on the base of epoxy resin.

The lubricating composition produced on the base of epoxy resin and thickened with amorphous silica Aerosil® showed a much better level of antiscuffing properties in compared to the compositions produced with the participation of mineral, vegetable and synthetic oil base (Fig. 1). The determined values of limiting pressure of seizure showed that the use of epoxy resin as a the dispersion phase, has affected the increase in comparison to the composition produced on mineral, vegetable and synthetic base oil the resistance of the top layer on scuffing for the tested lubricating compositions used in the experiment. The p_{oz} parameter provides the information about the pressure prevailing in the friction zone at the moment of seizing. On the base of the obtained test results, it can be concluded that the use of epoxy resin as a dispersion phase of greases affected on the formation of highly resistant on scuffing of the surface layers. The higher p_{oz} parameter for the composition A, where the epoxy resin was used as the dispersion phase shows that the character of the created film favours a significant reduction of wear [19,20,24,30,34].

The antiwear properties of the tested lubricating greases were verified by determining of the limiting load of wear $G_{oz/40}$ of tribosystem lubricated with the evaluated compositions. The average friction coefficient for the tested lubricating greases was within 0.07 [-]. The obtained results are shown in Fig. 2.

The tests of tribological properties of the produced lubricating compositions have shown that the type of oil base used changes the ability of the tested lubricating greases to the antiwear

protection of the tribosystem. The durability of the boundary layer is provided by the value of the limiting load of wear $G_{oz/40}$. The higher the index, the bigger the durability of the boundary layer and the reduction of wear. The highest value of the limiting load of wear was characterized by a lubricating composition, which was produced on the base of epoxy resin (grease A). The value of the $G_{oz/40}$ parameter for grease A was about 30.6 % higher than the value of this parameter for grease C produced on a vegetable base and, respectively, about 97.3 % and 154.5 % higher, than the value of the limiting load of wear for grease B (based on mineral oil) and grease D produced on synthetic oil. The lowest value of this parameter was observed for the composition D, which was produced on the base of synthetic oil. The type of dispersion phase used of lubricating grease has an influence on changing their antiwear properties. The lubricating composition produced on the base of epoxy resin and thickened with the amorphous silica Aerosil® showed a much more favourable level of antiwear properties in relation to the compositions produced on the base of mineral, vegetable and synthetic oil (Fig.2).

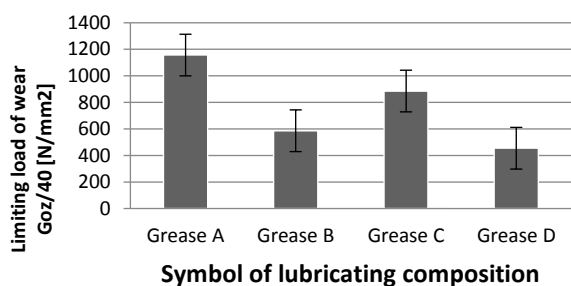


Fig. 2. Limiting load of wear of tribosystem lubricated compositions produced on epoxy resin, mineral, vegetable and synthetic oils.

The analysis shows that the most effective antiwear protection under constant load conditions of the tribosystem was represented by the lubricating composition, in which the epoxy resin was used as the dispersion phase, and the lowest effective antiwear effect was observed in the case of the composition produced on a synthetic oil base.

The type of the dispersion phase used in the lubricating compositions, and in particular its viscosity, has a large influence on the value of the basic tribological parameters of the tested lubricating greases. The higher viscosity of the

dispersion phase, the lower the tendency for tribological wear. This can be explained by increasing the thickness of the oil film, which allows reduction of the contact frequency of surface irregularities [24,25,28,30,34,37].

The quality criteria of lubricants designed for the food industry are established individually by the producers of particular devices. On the basis of market research, the lubricating compositions are classified as follows: those of $G_{oz/40} > 600$ N/mm² have very good antiwear properties; those in the range of 400-600 N/mm² are regarded as having effective properties; whereas, the compositions of $G_{oz/40}$ with the value less than 400 N/mm² are considered unsatisfactory.

The results shown in this paper doubtlessly classify all tested compositions as effective in terms of antiwear properties under constant load conditions.

3.2 Results and discussion of physico-chemical properties of the tested greases

The comparison of the test results of the effect of the type of dispersed phase on the dropping point of the lubricating compositions are presented in Fig. 3, while the evaluation of the influence of the oil base on the value of emission oil from grease are shown in Fig. 4.

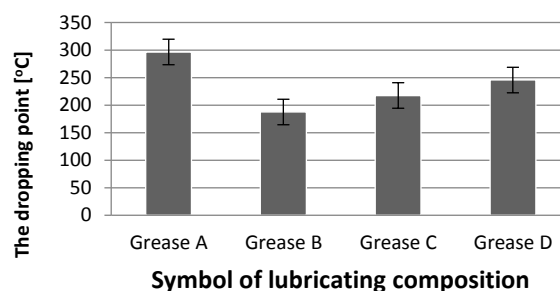


Fig. 3. The influence of type of dispersion phase on change of dropping point for researched lubricating greases.

Based on the analysis of the executed tests, it was ascertained that the use of epoxy resin as a dispersion phase of lubricating greases caused an increase of the dropping point of the grease subjected of the experiment compared to the compositions produced on a mineral, vegetable or synthetic oil base, which proves the improved structural stability of the tested lubricant (Fig. 3). The use of epoxy resin as a dispersion phase of

lubricating greases significantly improved the dropping point of the tested lubricating composition in compared with other lubricating compositions, which were confirmed by the tests. The dropping point of grease A produced on epoxy resin was higher by 58.1 % than the value of this parameter for a composition prepared on a mineral oil base. In case of a lubricating composition produced on a vegetable oil base, a decrease of dropping point about 26.6 % was observed, and for a grease produced on synthetic oil, decrease of the dropping point about 17.2 % was observed, in relation to the value of this parameter for grease A. Using an epoxy resin as the dispersion phase of lubricating greases showed a synergistic interaction with used thickener and a significant improvement of the dropping point. Thus, the use of epoxy resin as the "oil" base of lubricating greases affected the improvement of the chemical stability of the lubricating composition.

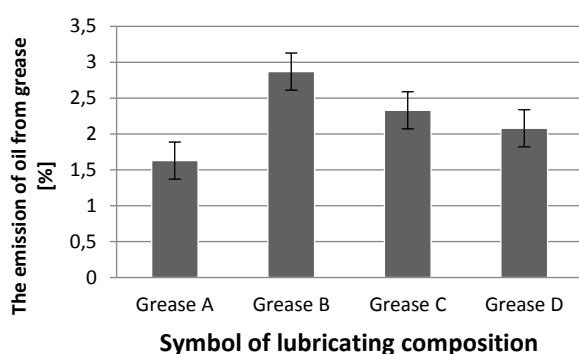


Fig. 4. The influence of type of dispersion phase on change of parameter emission of oil from grease for researched lubricating greases.

The emission oil from a grease test is a measure of the durability of the dispersion phase (base oil) - the thickener and tendency of the grease to separate this phase. It was observed that the most stable structure was characterized by composition A produced on the base of epoxy resin, while the least stable was grease B produced on the base of mineral oil. The use of the epoxy resin as a dispersion phase of lubricating greases in relation to the results obtained for lubricating compositions prepared on different oil bases causes an increase of the structural stability of the lubricating grease, which is evident by reducing the quantity of the dispersion phase (oil).

It should be stressed that the standard for bearings greases LT specifies the quantity of emission oil

from lubricating greases without losing the lubricating abilities. The quantity of oil that can be separated from the grease should not transcend of 10 %. The best properties characterized the lubricating composition produced on the base of epoxy resin, in which the quantity of separated under these conditions of dispersion phase was 1.93 %, i.e. much lower than the limit value, i.e. 10 %. It is observed that the use of epoxy resin as the dispersion phase together with an inorganic thickener in the form of amorphous silica Aerosil® has a very advantageous effect on the quantity of the separated dispersion phase from the grease. The lubricating compositions based on mineral, vegetable and synthetic oil were characterized by a small quantity of separated oil (less than 4 %), i.e. fulfilled the requirements of the standard for bearing greases (under 10 %), while the use of epoxy resin during the composing of the grease significantly reduces the value of the discussed parameter. So that they don't lose their lubricating properties, each grease should emission a small quantity of the dispersion phase (oil), which provides between the lubricated surfaces the existence of a film of appropriate thickness.

For selected lubricating compositions, an oxidation tests on the PetroOxy apparatus were carried out. The influence of type of dispersion phase on the oxidizability of the tested greases at 80 °C and 120 °C are shown in Fig. 5.

The test of oxidation of lubricating compositions was carried out on the PetroOxy apparatus at 80 °C and 120 °C. The carried out test was to determine the effect of the type of dispersion phase of lubricating greases on the value of oxidation stability of the tested lubricating compositions. It was ascertained that the oxidation induction times of the tested lubricating compositions determined with the PetroOxy test differ from each other significantly. The lubricating composition based on epoxy resin was characterized by a higher oxidation at 80 °C about 172.6 % on the value of this parameter for the composition B, which was produced on a mineral oil base. In the case of composition C based on vegetable oil and composition D produced on a synthetic base, whose oxidative stability were, respectively, 37.48 h and 48.55 h, and were lower than the oxidative stability of composition A about, respectively, 221.10 % and 147.88 %. Whereas at 120 °C, the oxidation stability for grease A was 15.44 h and

was 5-8 times higher than the oxidation stability of lubricating compositions based on the mineral, vegetable or synthetic oil. The lubricating grease produced on the base of epoxy resin has a much longer oxidation induction time than lubricating compositions produced on the base of mineral, vegetable or synthetic oil.

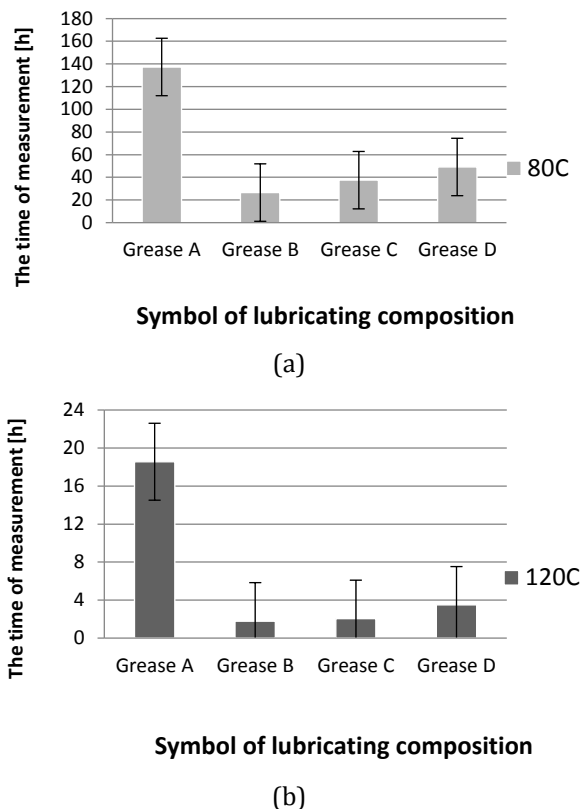


Fig. 5. The influence of type of dispersion phase on change of oxidation stability of researched lubricating greases in (a) 80 °C and (b) 120 °C.

The elongation of the oxidation time of the lubricating composition prepared on the base of epoxy resin can be caused by the presence of resin molecule of epoxy groups, hydroxyls derived from phenols and polyglycols, which interact synergistically with the thickener molecules causing an increase of the resistance of the tested composition on oxidation.

For selected lubricating compositions, the anticorrosive properties test using the dynamic method on the SKF EMCOR apparatus were carried out. The effect of the type of dispersion phase on the corrosion of the greases tested under dynamic conditions were carried out, as shown in Fig. 6, and the results of tests on the effect of the type of dispersion phase on corrosion of the tested lubricating compositions on copper plates are presented in Table 2.

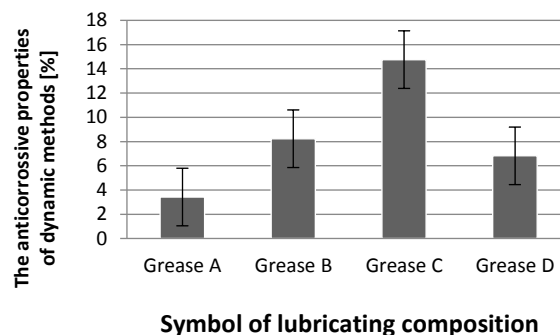


Fig. 6. The influence of type of dispersion phase on anticorrosive properties of dynamic methods of researched lubricating greases.

Table 2. The results of corrosion tests on copper plates for the tested lubricating compositions.

The parameter	The test method	The unit	The symbol of grease			
			A	B	C	D
The corrosion on copper plates [degree of corrosion]	PN-85/C-04093 – method A	-	1a	1a	1a	1a

The performance of the test was meant to determine the effect of the type of dispersion phase on the anticorrosive properties of the tested lubricating compositions determined by the dynamic method. It was ascertained that the anticorrosive properties of the tested lubricating compositions determined with the EMCOR test differ from each other significantly. The lubricating composition based on epoxy resin (grease A) was characterized by the lowest value of anticorrosive properties (corroded 3.42 % of the bearing surface), which indicates a high corrosion resistance for this composition. The compositions B, C and D, which were prepared on mineral, vegetable and synthetic base oil, were characterized by lower values of this parameter. Grease B, which has been prepared on a mineral oil base, was characterized by more than twice worse anticorrosion properties (more than 8 % of the bearing surface was corroded). In the case of composition C based on vegetable oils and composition D produced on the base of synthetic oil, the anticorrosion properties were more than four times worse (grease C) and two times worse (grease D) compared to the anticorrosive properties of grease A. The lubricating grease produced on the base of epoxy resin shows much better anticorrosive properties than compositions

produced on the base of mineral, vegetable or synthetic oil. This situation may be caused by the presence of hydroxyl groups derived from phenols in the resin molecule, which interact synergistically with the thickener molecules causing an increase in the resistance of the tested composition to corrosion under dynamic conditions. It did not allow to determine the presence of corrosion on the copper plates for all tested lubricating compositions, which may provide that the chemical composition of the lubricants subjected to the experiment shows high resistance on copper.

The area of application of the lubricating greases depends, among others, on the consistency class, which was determined on the base of the penetration measurement. It is the base for evaluation of the rheological properties of lubricating greases. The obtained results of testing this parameter are presented in Fig. 7. However, the results of mechanical stability tests, i.e. the difference between penetration and penetration after prolonged kneading, are shown in Fig. 8.

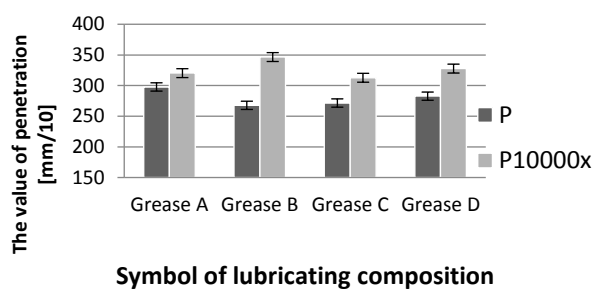


Fig. 7. The influence of type of dispersion phase on change of value of penetration of researched lubricating greases.

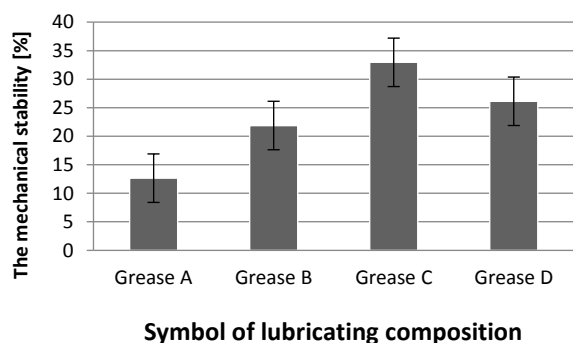


Fig. 8. The influence of type of dispersion phase on change of mechanical stability of researched lubricating greases.

It was ascertained that the type of the dispersion phase used influences the change of penetration of

the tested lubricating compositions. The highest penetration was characterized by a composition produced on epoxy resin, and the lowest the composition based on synthetic oil. All lubricating compositions were in the second consistency class. However, after prolonged kneading (10^4x), the highest penetration was characterized by composition B produced on mineral oil, the lower has a compositions on epoxy resin and synthetic oil, and the lowest the composition C was produce with vegetable oil. However, after prolonged kneading the consistency class for all tested compositions declined by one class (from 2 to 1). The penetration changes indicated on change of the mechanical stability of the tested lubricating compositions. The analysing of the tested lubricating compositions, it was ascertained that on penetration was significantly affected by a type of dispersion phase used. Analysing the mechanical stability of the tested lubricating compositions, i.e. the % change of penetration before and after prolonged kneading, it should be ascertained that the composition based on epoxy resin (change of penetration about 7.72 %) looks preferably. The worse mechanical stability showed the compositions produced on the base of vegetable and synthetic oil (change of penetration about respectively 15.19 % and 15.92 %). While the weakest mechanical stability showed the composition produced on the base of mineral oil, where the penetration after prolonged kneading increased about 29.41 %. A small decrease or increase of the penetration of the tested lubricating compositions caused by the presence of different dispersion phase doesn't cause deterioration of their functional properties. In the industry, lubricating products with different consistencies (in a wide range of penetration values) are used, so the use of the obtained lubricating greases (except the composition B based on mineral oil) was most reasonable, especially that their lubricating properties aren't deteriorated. According to the ISO 2137: 2011 standard on the measurement of the penetration of lubricating greases, it is allowed to change of the penetration after prolonged kneading (10^4x) to 50 units of penetration [28-30,34,37,39,40,46,47].

3.3 Results and discussion of rheological properties of the tested greases

For selected lubricating compositions, the tests of rheological properties on the rotational rheometer were carried out. The influence of

type of dispersion phase on change the rheological properties of lubricating greases were presented on Figs. 9-11.

The measurements were carried out using a cone-plate measuring system in the shear rate range $0.01 \div 100 \text{ s}^{-1}$ at $20 \text{ }^\circ\text{C}$ (flow curves), at a shear rate of 100 s^{-1} in the temperature range $-30 \text{ }^\circ\text{C} \text{ } -180 \text{ }^\circ\text{C}$ (viscosity curves) and in the range shear rates $0\text{-}1000 \text{ s}^{-1}$ at $20 \text{ }^\circ\text{C}$ (viscosity curves).

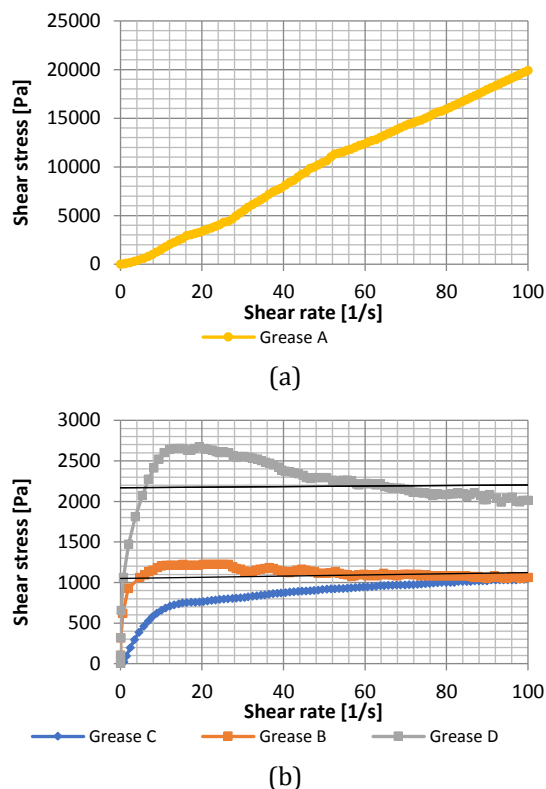


Fig. 9. (a) The influence of type of dispersion phase on change of flow curves for lubricating grease A, and (b) The influence of type of dispersion phase on change of flow curves for lubricating greases B-D.

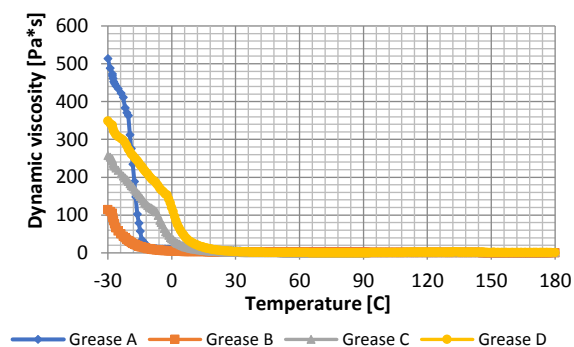


Fig. 10. The influence of type of dispersion phase on change of dependence of dynamic viscosity from temperature for researched lubricating compositions.

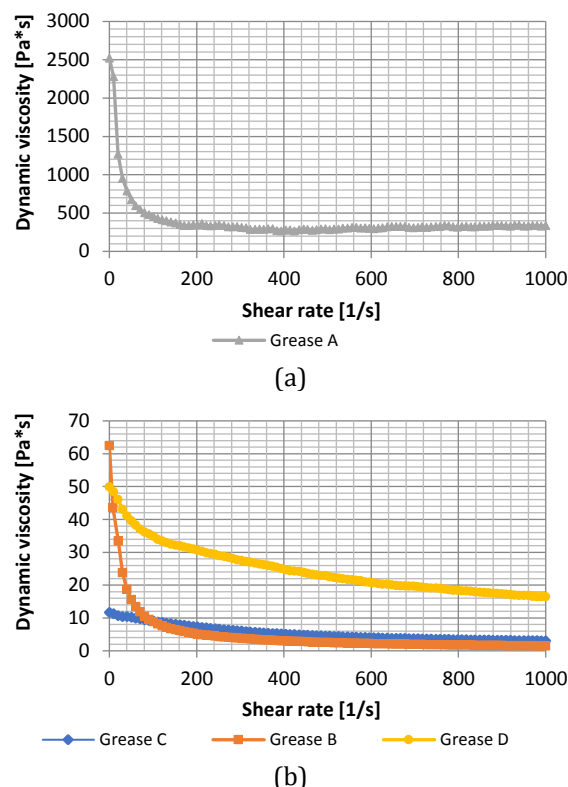


Fig. 11. (a) The influence of type of dispersion phase on change of dependence of dynamic viscosity from shear rate for lubricating grease A, and (b) The influence of type of dispersion phase on change of dependence of dynamic viscosity from shear rate for lubricating greases B-D.

The use of epoxy resin as a dispersion phase of the lubricating greases has caused a significant increase of dynamic viscosity from shear rate in compared with compositions produced on conventional base oils. At lower shear rate values, the dynamic viscosity of the lubricating composition A based on epoxy resin was more than 10 times higher than the dynamic viscosity of lubricating compositions produced on mineral, vegetable and synthetic oil (greases B, C, D). However, at higher shear rate values, the dynamic viscosity of the composition A was higher several times from the compositions prepared on conventional base oils. Such a high viscosity of the obtained lubricating composition in compared with compositions prepared with conventional oils can have a consequence of extension the possibility of using such a grease in many industries, where until now there have been a big difficulties with lubrication of parts of machines and devices with traditional lubricants of much lower viscosity than the discussed composition.

The evaluation of changes of dynamic viscosity and temperature for the tested lubricating compositions, it was ascertained that the use of epoxy resin, the mineral oil, vegetable oil and synthetic oil as the dispersion phase of tested lubricating greases has caused of significant changes of viscosity in the low temperature range. The value of the dynamic viscosity of the tested lubricating compositions has changed depending on the type of dispersion phase. The lowest value of dynamic viscosity in the range of negative temperatures was characterized by a composition based on vegetable oil (grease C). The composition produced on a mineral base oil (grease B) has more than twice time higher of dynamic viscosity. The composition produced on the base of synthetic oil (grease D) has even more viscosity in the range of negative temperatures, but the maximum dynamic viscosity was characterized by the composition based on epoxy resin (grease A), whose viscosity was twice times higher from viscosity of composition D and seven times higher than viscosity of composition C. In the temperature range 30-180 °C, it wasn't observe a significant changes in dynamic viscosity. The high viscosity of composition A in the range of negative temperatures compared to the viscosity of lubricating compositions prepared on conventional oils may be of interest for using the tested composition for the lubrication of machines and devices in industries, where temperatures are low.

The shear stress value for a lubricating composition produced on the base of epoxy resin undergoes a continuous increase along with the increase of the shear rate. At a shear rate of 100 [1/s], it acquired value of 8400 [Pa]. However the compositions prepared on a mineral, vegetable and synthetic oil base has a different shape from the discussed composition. In the case of greases B and D, the stress value increases to 1000-1500 [Pa] at a shear rate of 10 [1/s] achieving the maximum, after the stress value shall not be a significantly changes. For composition C produced on the base of vegetable oil, the maximum stress value was 2800 [Pa] and was achieved at a shear rate of about 12 [1/s], then the stress value systematically decreases to 2000 [Pa] at 100 [1/s]. The calculated tangential stress (yield stress) for the tested lubricating compositions was the highest for composition A and ranged 3000 [Pa], for composition C ranged 2800 [Pa], and for compositions B and D,

respectively 1000 [Pa] and 1350 [Pa]. This proves that the use of epoxy resin as a dispersion phase of greases reinforces the spatial structure of such grease and reduces the possibility of occurrence of unfavourable phenomena that may affect the change of properties of the receiving composition. It has an important meaning during the selection of construction parameters of machines and devices in various branches of industries, for example when designing of central lubrication systems.

All lubricating greases used in the article are non-Newtonian fluids, whose dynamic viscosity decreases with the temperature and shear rate, i.e. they are thinned with shear [30, 34,37,38,39,40,46,47].

The rheological properties of the lubricating compositions have affected alike the type of dispersion phase, the dispersed phase, the grease production technology and the conditions under which the grease will be used. The interaction between the dispersed phase and the particles of dispersion phase increases with increasing of percentage content and type of the oil base until to achievements of certain optimum. With increasing this participation and the type of dispersion phase, the value of dynamic viscosity changes from the temperature and the shear rate and the yield stress of the grease. The conditions in which the grease was produced has a significant impact on the stability of its structure and on the shaping of limit values of tangential stresses. To consolidate this structure and make it more homogeneous and resistant to external factors are used such oil bases, which in the required range affecting on the size of free interfacial energy between the dispersion phase and the dispersed phase.

4. CONCLUSIONS

Based on the results of the carried out tests, it was ascertained that the properties of the tested lubricating compositions were significantly changed depending on the type of the dispersion phase used.

The designated values of the limiting pressure of seizure have shown that the use of epoxy resin as a dispersion phase affect on increasing in relation to the lubricating compositions produced on the

mineral, vegetable and synthetic oil base of the resistance of the top on scuffing for tested lubricating compositions. As a result of improving the properties of the surface layer, the start of scuffing occurs at a higher load of tribosystem, which results in an increase of the durability and efficiency of many sliding associations.

The use of epoxy resin as a base oil of a lubricating greases caused an increase of the value of the G_{oz} parameter in relation to the compositions produced on a mineral, vegetable and synthetic oil base, which proves about the high resistance of the discussed lubricating composition on the interrupting of the surface layer.

The use of the epoxy resin as a dispersion phase of the lubricating greases showed a synergistic interaction with the thickener used and a significant improvement of the dropping point in relation to the compositions produced on conventional oil bases, which affected on improvement of the chemical stability of the tested lubricating composition. The use of the epoxy resin as a dispersion phase of lubricating greases in relation to the results obtained for lubricating compositions prepared on oil bases cause of an increase of the structural stability of the lubricating grease, which was visible by reducing the quantity of the dispersion phase (oil).

The lubricating grease produced on the base of epoxy resin shall show a much longer of oxidation induction time than lubricating compositions produced on the base of mineral, vegetable or synthetic oil. The extending of the oxidation time of the lubricating composition prepared on the base of epoxy resin can be caused by the presence in resin molecule of epoxy groups, hydroxyls originating from phenols and polyglycols, which interacting synergistically with the thickener molecules causing an increase of the resistance of the tested composition on the oxidation process.

The lubricating grease produced on the base of epoxy resin shows much better anticorrosive properties than compositions produced on the base of mineral, vegetable or synthetic oil. This situation may be caused by the presence of hydroxyl groups originating from phenols in the resin molecule, which interacting synergistically with the thickener molecules causing an increase of the resistance of the tested composition on

corrosion under dynamic conditions. The presence of corrosion on the copper plates for all tested lubricating compositions was not ascertained, which may provide that the chemical composition of the greases subjected to the experiment shows a high resistance on the copper.

It was ascertained that the type of the dispersion phase used influenced the changes of the penetration of the tested lubricating compositions, and changes of the penetration indicated the change of mechanical stability. The best mechanical stability was characterized by the composition based on epoxy resin, and the weakest was composition based on mineral oil. A small decrease or increase of the penetration of the tested lubricating compositions caused by the presence of different dispersion phase does not cause deterioration of their functional properties.

The use of epoxy resin as a dispersion phase of the lubricating greases caused a significant increase of dynamic viscosity from shear rate in compared with the compositions produced on conventional base oils.

The value of the dynamic viscosity of the tested lubricating compositions has been changing depending on the type of dispersion phase. The lowest value of the dynamic viscosity in the range of negative temperatures was characterized by a composition based on vegetable oil, the composition on mineral oil has a twice time higher of viscosity, but the highest value of the dynamic viscosity was characterized by the composition based on epoxy resin, whose viscosity was seven times higher than the viscosity of vegetable grease. In the temperature range 30-180 °C, significant changes of the dynamic viscosity for the tested lubricating compositions weren't observed.

The shear stress value for a lubricating composition produced on the base of epoxy resin undergoes a continuous increase with the shear rate increases. In the case of greases on mineral and vegetable base, the stress value at first increases achieving the maximum, and then shall not be a significant changes. For compositions based on vegetable oil, the stress value at first increases to optimum and then decreases. The calculated values of the yield stress showed that the highest value of this parameter was characterized by the composition based on epoxy

resin, and the lowest value was characterized by the composition based on mineral oil. This proves that the use of epoxy resin as a dispersion phase of lubricating greases strengthens the spatial structure of such grease and reduces the possibility of unfavourable phenomena that may affect the change of properties of the received composition.

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