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RESEARCH

The Application of Chitosan as a Modifier for Lubricating Greases Based on Vegetable Oil

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A B S T R A C T

The paper focuses on the influence of novel chemical additive on the physicochemical and rheological properties of novel lubricating grease compositions. The mentioned physicochemical tests revealed that the additive influenced positively the dropping point and oxidation stability of the tested lubricating greases as well as on the reduction of oil release from the grease. The introduction of chitosan caused also an increase of dynamic viscosity at lower shear rates and significant changes of viscosity in the low temperatures. The introduction of modifier led to the increase of yield point value of the tested lubricating compositions. Spectral analyses of lubricating compositions after tribological tests showed that during mechanical and thermal excesses, some components of the lubricant undergo chemical reactions with the surface of the friction pair, creating compounds effectively protecting the tribosystem from damage.

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1. INTRODUCTION

The properties of lubricating greases depend on its composition and production technology and are shaped, among others, by appropriately selected enriching additives [1-4]. Typical packages of additives improving lubricating greases include, among others: antioxidants (increasing grease resistance to oxidation), anti-wear and anti-seizure additives (improving the tribological properties of grease), anti-corrosion (reducing the aggressiveness of lubricants against metals), adhesives (improving adhesion of grease to machine construction elements) and rheological (improving the rheological properties of the grease) [1,5-9]. Not only the presence of the additive decides about the usable properties of the lubricant, but also the way it is incorporated into the lubricating grease structure. The introduction of additives to lubricating greases causes many technological difficulties, because the additive particles adsorb on the surface of the thickener, which in turn may lead to a reduction of the effectiveness of such a component, and even to a decrease in the stability of the grease [1,3,5,7,10-11].

For lubricating greases, appropriate, specially selected additives should be used in an amount that determines the improvement of their performance. Lubricating greases mix very well with solid lubricating additives, which reduce the friction force and increase the resistance of the friction pair to loads and seizing [12-17]. In harsh operation conditions, these additives increase the efficiency of the lubricant due to resistance to chemical agents and better resistance to high temperatures. The most common among this type of additives are graphite, molybdenum disulphide, polytetrafluoroethylene, copper and chloroparaffins [5-6,8-9,18-22].

The introduction of modifiers to the lubricant structure significantly improves anti-seizure and anti-wear properties, as well as the physicochemical and rheological properties on which the lubricating composition's performance depends [12-17,20-23]. These features explain the great interest in innovative additives, such as chitosan. The share of additives introduced into the lubricant compositions at the level of 0.1-5 % is sufficient to achieve specific high demands placed on lubricating greases [5,8-17].

Chitosan is a naturally occurring water-soluble polycation, mainly manufactured by alkalinede-acetylation of chitin. It should be noted that chitosan is not a common structural polymer in nature. This concerns the initial molecule of chitin, which is found in the outer skeletons of, e.g., crustaceans and insects. Chemically it is a linear polysaccharide of β–(1-4) linked 2-acetamide-2-deoxy-D-glucose and 2-amino-2-deoxy-D-glucose [24]. Chitosan possess amphiphilic nature, the D-units of the molecule are strongly hydrophilic while A-units are responsible for the hydrophobic properties of the polysaccharide influencing the surface activity. The non-polar acetyl groups will associate with the oil phase, while the segments containing polar and (at acidic pH-values) charged D-units will extend into the aqueous phase [25]. As chitosan is the only abundant low-toxic, biodegradable, and (at acidic pH-values) positively charged biopolymer, it has become a popular device or excipient in many applications involving biological systems [26].

The aim of the work was to investigate the effect of various amounts of chitosan as a modifier on the change of physicochemical and rheological parameters for lubricating compositions applicable in the food industry and developed at the Institute of Sustainable Technologies - NRI in Radom.

2. THE MATERIALS AND METHODOLOGY OF RESEARCH

A group of model lubricating compositions was developed using non-toxic ingredients that are a dispersion and dispersed phase. Linseed oil was used as the dispersion phase [27-29] and a modified silica type Aerosil® was used as the dispersed phase [23,27,30]. Such prepared lubricating composition was then modified with various amounts of chitosan. Using the selected components, the second-class of consistency lubricating greases were produced and for the use in the food industry.

The modifier was added to the structure of each lubricating grease in the amount of 0,2; 0.6; 1; 3 and 5% m/m. The lubricating compositions prepared this way were marked as follows: A (base grease), B (modified of 0.2 % chitosan), C (modified of 0.6 % chitosan), D (modified of 1% chitosan), E (modified of 3 % chitosan) and F (modified of 5 % chitosan). In the early phase of the experiment, research was carried out on the amount of additive that should be added into the lubricant composition. Tests were carried out for concentrations from 0.1 to 7 % of the modifying additive. The lubricating compositions prepared this way were subjected to the physico-chemical and rheological tests.

The dropping point measurement was made according to the PN-ISO 2176:2011 standard. The principle of determination was to determine the temperature at which the first drop of grease flows out of the test vessel during its even heating. The arithmetic mean of the two thermometers was taken as the result of the test: from the test tube with the test grease and the oil bath [17].

The oil separation test was performed according to PN-V-04047:2002. The determination method is based on determination- under static conditions- of the amount of oil separated from the grease in a nickel grid cone at 100 °C for 30 hours. The amount of separated oil was given as a mass fraction expressed as a percentage [17].

The thermo oxidative stability test was carried out using the PetroOxy apparatus. A sample of 5 ml of lubricant was introduced into the test chamber of the device and subjected to 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 % reduction in the maximum pressure in the measuring chamber [31-33].

The research on rheological properties was performed 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 was equipped with Peltier temperature control system in the range -40-200 °C and in the external VISCOTHERM V2 thermostatting system, operating in the temperature range -20-200 °C. Rheometer control and analysis of data were carried out using the Rheoplus software. The measurements were made using a cone-plate measuring system in the shear rate range 0.01÷100 s^{-1} at 20 °C (flow curves), at a shear rate of 100 s^{-1} in the temperature range -30 -180 °C (viscosity curves) and in the shear rates range 0-1000 s^{-1} at 20 °C (viscosity curves) [34].

A confocal dispersive Raman NRS 5100 microspectometer (Jasco Corporation, Japan) equipped with an excitation laser with a wavelength of 532.12 nm and a CCD detector was used to test the chemical composition of lubricating greases. The parameters of the spectrometer were as follows: diffraction grating 1800 lines/mm, laser power 5.2 mW, numerical aperture 4000 µm, spectral range 4000÷400 cm^{-1}, resolution 4.2 cm^{-1}, magnification of the lens 100x, exposure time 20 s. The material samples were placed onto glass plates [35].

Before the spectral tests of the grease the tribological (antiwear) test was carried out. The tribological tests were performed using the T-02 testing machine made by the Institute for Sustainable Technologies - NRI. The measured parameter called the limiting load of wear ($G_{oz/40}$) [12-17,36-39]. The friction pair was composed of 12,7 mm balls made of LH 15 bearing steel, surface roughness Ra=0,32µm and hardness of 60-65HRC. The measurement of limiting load of wear ($G_{oz/40}$) was performed by 392,4 N of load for 3600 s and rotation of 500 rpm according to the test standard WTWT-94/MPS-025 [12-17,40]. The final result was the arithmetic mean based on at least three independent tests, that did not differ from one another by more than 10 %. The statistics was based on Q-Dixon test at 95 % level of confidence [12-17,36-39,41-43]. The limiting load of wear ($G_{oz/40}$) is the measure of antiwear properties of the lubricants. This parameter was evaluated by the following calculation: $G_{oz}=0,52*392,4N/d_{oz}^2$, where 392,4 N- the constant load of wear test, and d_{oz}- the diameter of the trace measured on the test balls used in the test. The mentioned measurements were performed using the microscope equipped with the adequate measuring kit. The results were used in determination of $G_{oz/40}$ - the evaluation of the antiwear properties of the test lubricants [12-17,41-43].

3. THE RESULTS OF RESEARCH AND DISCUSSION

The results of the impact of the additive used on the dropping point of lubricating compositions is shown in Fig. 1, whereas the results on oil separation from the lubricant is shown in Fig. 2.

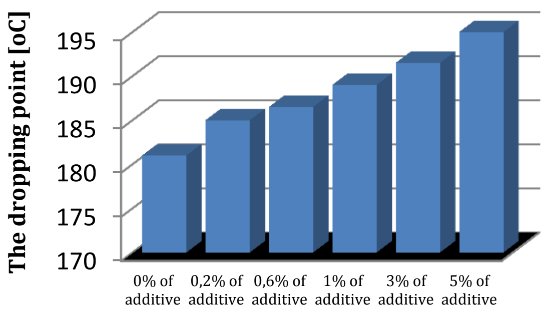

Fig. 1. The influence of modified additive for a change of dropping point for researched lubricating greases.

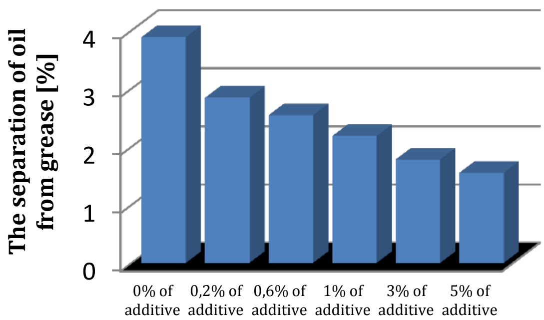

Fig. 2. The influence of modified additive for a change of parameter separation of oil from grease for researched lubricating greases.

On the basis on the performed tests it was found that the introduction of chitosan increased the dropping point of the tested lubricants when compared to the control, which means improved structural stability of the samples (Fig. 1). The introduction of increasing amounts of additive into the base grease resulted in increasing dropping point. The grease modified with 5 % of chitosan revealed greater dropping point by 7.7 %. The chitosan as a modifier showed a synergistic interaction with the thickener and the base oil. Thus, the additive used improved the chemical stability of the lubricating compositions evaluated.

The oil release test is a measure of the stability of the base oil-thickener system. The most stable composition was F and the less stable composition was A. The modifier introduced into the initial grease composition increases the structural stability of the greases at any concentration, visible in reduction of the released oil. It should be noted that the standard for bearing greases specifies the amount of oil released from greases without losing their lubricating properties. The amount of oil that can be released should not exceed 10 %. The most advantageous properties for grease with 5 % chitosan, for which the amount of oil was 1.55 %, i.e. much lower than the limit value, i.e. 10 %. The lubricating composition before chitosan modification is characterized by a small amount of separated oil is (does not exceed 4 %), while the introduction of a modifying additive significantly reduces the value of the discussed parameter.

For selected lubricating compositions were examined for the oxidation resistance on the PetroOxy apparatus. The influence of various amounts of the modifier on this parameter were presented on Fig. 3.

The oxidation stability of modified greases was carried out using PetroOxy apparatus at 80 and 120 °C. The purpose of this task was to determine the effect of the additive on the oxidation stability of the tested lubricant compositions. It was found that the oxidation induction times of the tested lubricating compositions determined with the PetroOxy test differ. A linear relationship was observed along with the additive content and in some cases also for the oxidation induction of the tested lubricant compositions. The greater the quantity of lubricant added, the longer it takes for its oxidation. Modified greases with chitosan showed a much longer induction time of oxidation than the base composition lacking the modifying additive. The prolongation of the oxidation induction time may occur due to the presence of amine and hydroxyl groups in the chitosan molecule, which incorporate into the hydrocarbon chain of linseed oil and interact synergistically with the thickener molecules causing an increase in resistance of the test compositions to oxidation.

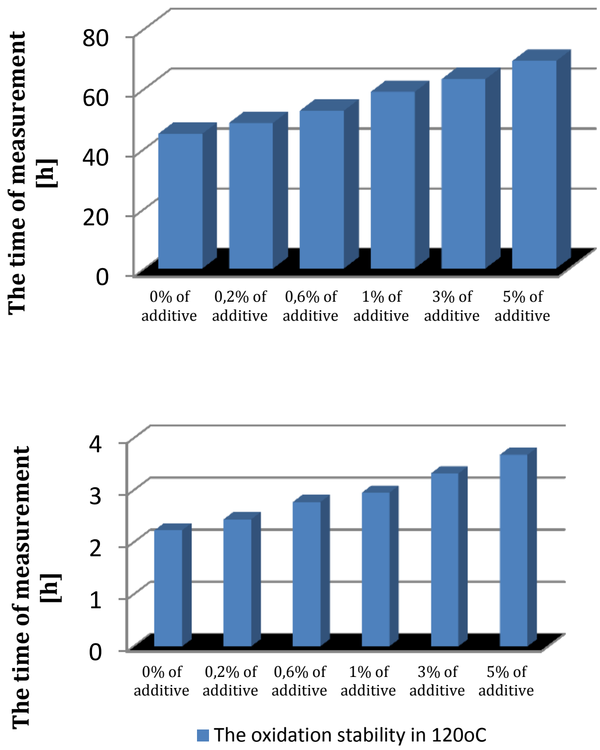

Fig. 3. The influence of different quantity modifying additive for a change of oxidation stability of researched lubricating greases in 80 °C and 120 °C.

For selected lubricating compositions were carried out the tests of rheological properties on the rotational rheometer. The influence of various amounts of the modified additive on the rheological properties of lubricating greases were presented on Figs. 4-6.

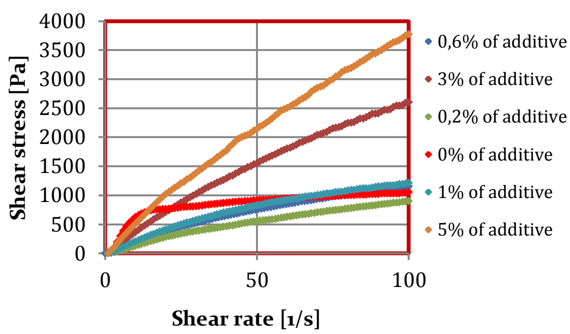

Fig. 4. The flow curves of researched lubricating compositions modified of different quantity of modified additive.

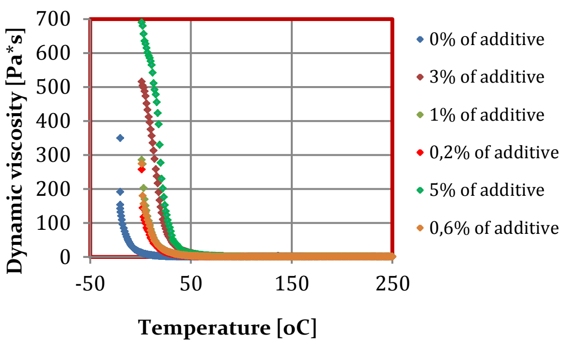

Fig. 5. The dependence of dynamic viscosity from temperature for researched lubricating compositions modified of different quantity of modified additive.

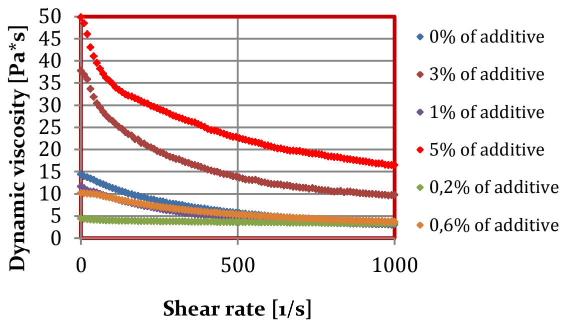

Fig. 6. The dependence of dynamic viscosity from shear rate for researched lubricating compositions modified of different quantity of modified additive.

The introduction of increasing amounts of the modifier into the grease resulted in increased dynamic viscosity at lower shear rate values. On the other hand, the incorporation of 3 % of chitosan into the lubricating composition revealed a significant increase in dynamic viscosity at higher shear rates in comparison to the control and greases containing up to 1% of chitosan. Assessing changes in dynamic viscosity and temperature for the tested lubricating compositions it should be noted, that the introduction of chitosan into the structure of greases tested resulted in significant changes in viscosity in low temperature range. The value of dynamic viscosity of the tested lubricating compositions increased along with the amount of additive. The highest viscosity was observed for the grease modified with 5 % of chitosan, and the lowest for 0.2 %. In the temperature range 50-250 ºC, no significant changes in dynamic viscosity were observed.

The tangential stress value (yield point) first decreases for compositions modified with max. 1 % of chitosan, and increases in the case when at least 3 % modifier was added. This proves that the introduction of the modifier into lubricating compositions strengthened the spatial structure. This is important for designing central lubrication systems. The rheological properties of greases are influenced by the type of base oil, thickener and additive content incorporated to the grease as well as the production technology and the conditions under which the lubricant is used. The interaction between the thickener and the additive increases along with the percentage content of the additive to a certain optimum. With the increase of this share, the relationship between temperature and dynamic viscosity changes as well as the yield strength of the lubricant. The production technology has a significant impact on the stability of grease structure and on the development of limit values of tangential stresses. To consolidate this structure and make it more homogeneous and resistant to external factors, additives are used that modify the free interfacial energy between the base oil and individual thickener molecules within the required range. Chitosan as an additive behaved this way.

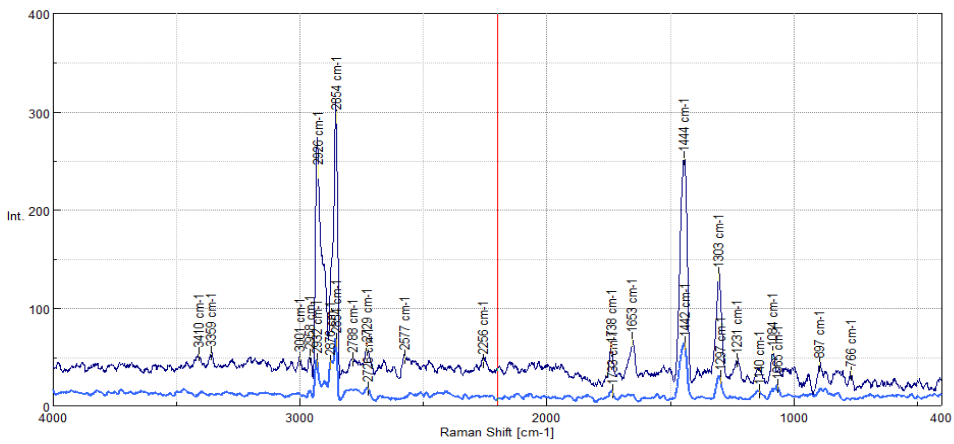

Fig. 7. The influence of additive content to the change of the structure of a base grease (blue) and grease modified with chitosan (dark blue).

For selected greases a Raman spectroscopy analyses were performed. The influence of the used modifier on the chemical structure of lubricants were presented in Fig. 7.

The spectra reveal that on the surface of a steel element after tribological tests lubricated with grease containing chitosan, the presence of organometallic compounds in the top layer was recorded. The band at approx. 3410 and 3359 cm^{-1} was assigned to the -OH group involved in the formation of intermolecular hydrogen bonds. The bands at 2926 and 2854 cm^{-1} were created as a result of overlapping bands characteristic for symmetrical and asymmetric vibrations of the methyl and methylene groups ($-CH_3$, $-CH_2-$) derived from the carbon chain of the oil base of the test lubricant. The bands at 2729 and 2778 cm^{-1} are derived from the stretching vibrations of the $-CH_2$ and $-CH_3$ groups of the carbon chain. In contrast, the 2256 cm^{-1} band is most likely to group stretching vibrations - C = O - characteristic of aerobic oxidation products. The band appearing at 1738 cm^{-1} may be attributed to oscillations of the C = O group characteristic for esters. The band at 1653 cm^{-1} is assigned to the C = O group present in the chitosan amide bonds. On the other hand, the bands at 1440 cm^{-1} can be attributed to the -COO- vibrations or the symmetrical and asymmetric deformations of the -C-H group or groups occurring in this range -C = C-H present in the carbon chain of the oil base and the used additive. The band at 1598 cm^{-1} is characteristic of the -NH group of amide bonds present in chitosan. The band at 1300 and 1140 cm^{-1} present for both the initial composition and the modified chitosan is characteristic of both the C-C- skeletal vibrations as well as the asymmetric C-O- stretching tensions in the polysaccharides. The band at 1231 cm^{-1} is characteristic of the deformation vibrations of the -CH group in chitosan, and at 897 cm^{-1} can be attributed to the deformation vibrations of the -CH group in chitosan. The oxidation products can be explained by the very good anti-wear properties of lubricating compositions modified with chitosan. Performed tests showed that during mechanical and thermal excitations some of the components undergo oxidation, which results in organo-oxygen compounds forming an organic layer on the metal surface, counteracting the wear of the lubricated friction pair. Some compounds come to a close contact with the surface layer, increasing its resistance to wear and shearing.

The analysis of the results of tribological tests along with the analysis of the change in the structure of lubricants after tribological tests allows to state that the lubricating properties are not only the effect of the additive but also oxidation products and tribochemical reaction products with the friction pair working surface. The activity of the used additive is based on the production (during friction) a thin film strongly chemically bonded to the substrate, characterized by low shear strength and high plasticity and heat resistance. As a result of the thermal decay of chitosan, chemical reactions take place between components of the substrate material and grease. The complex compounds penetrate the working surfaces of the friction pair forming layers resistant to wear processes.

4. CONCLUSIONS

The proposed modifier is effective, improving the physico-chemical properties of the tested grease base. Its presence is revealed by an increase in dropping point, which determines the applicability of the product and a decrease in oil separation, responsible for the lubrication efficiency. The prolongation of the oxidation induction time of the tested lubricating compositions may be caused by the presence of amine and hydroxyl groups in the chitosan molecule, which incorporate into the hydrocarbon chain of linseed oil and interact synergistically with the thickener causing an increase in resistance to oxidation. A linear relationship was observed between the additive content. Such modified greases showed a much longer induction time of oxidation than the base composition.

The introduction of the chitosan to the lubricating composition resulted in the increase of dynamic viscosity in the low temperature range and at lower values of shear rate. It was also observed that the viscosity increases at higher shear rates by at least 3% addition of modifier. An increase in the yield point of the tested lubricating compositions was observed also when more than 3% of chitosan was added.

The spectral analyses of tested greases after tribological tests showed that during mechanical and thermal stresses, some components of the lubricant undergo chemical reactions with the surface of the friction pair, creating compounds effectively protecting it from damage.

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