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ANALYSIS OF TRIBOLOGICAL PROPERTIES OF VEGETABLE GREASES FOR APPLICATIONS IN AGRI-FOOD INDUSTRIES

ANALIZA WŁAŚCIWOŚCI TRIBOLOGICZNYCH SMARÓW NA BAZIE OLEJU ROŚLINNEGO DO ZASTOSOWAŃ W PRZEMYŚLE ROLNO-SPOŻYWCZYM

Key words: Abstract

Streszczenie

oils mixture, plant oil, greases, lubricity properties.

The article presents the results of research and development works and analyses the results in order to develop an ecological lubricant for use in food industry equipment. The research was carried out as part of a project undertaken by a scientific and industrial consortium. While the specialized lubricants were composed, particular attention was paid to the selection of components, taking into account both their functional and ecological properties. A vegetable oil and a mixture of vegetable and synthetic oil were used as the dispersion phase for the preparation of lubricants. Prior to the verification of the quality of developed lubricants in operation, oxidation stability and lubricating properties were tested in model experimental studies. The lubrication properties of the developed greases under operating conditions, namely, high dust environments, moisture, the presence of water. As a result, greases resistant to the oxidation process and with favourable anti-seizure and anti-wear properties have been developed.

Słowa kluczowe: mieszanina olejów, olej roślinny, smary plastyczne, właściwości smarne.

W artykule przedstawiono wyniki prac badawczo-rozwojowych oraz analiz przeprowadzonych w celu opracowania ekologicznego smaru do zastosowania w urządzeniach przemysłu spożywczego. Badania realizowane były w ramach projektu podjętego przez konsorcjum naukowo-przemysłowe. Podczas komponowania specjalistycznych środków smarowych zwrócono szczególną uwagę na dobór komponentów, uwzględniając oprócz ich właściwości funkcjonalnych także ekologiczne. Do wytworzenia środków smarowych jako fazę dyspersyjną zastosowano olej roślinny oraz mieszaninę oleju roślinnego z olejem syntetycznym. Przed weryfikacją jakości opracowanych środków smarowych w eksploatacji oceniono ich stabilność oksydacyjną oraz właściwości smarne w modelowych badaniach eksperymentalnych. Zweryfikowano właściwości smarne opracowanych środków w arunkach eksploatacyjnych. Właściwości funkcjonowania opracowanych środków smarowych zweryfikowano w ekstremalnych warunkach eksploatacji, a mianowicie wysokiego zapylenia, wilgoci, obecności wody. W wyniku realizacji projektu opracowano smary odporne na proces utleniania oraz o korzystnych właściwościach przeciwzatarciowych i przeciwzużyciowych.

INTRODUCTION

Special requirements for the usage the lubricants are in the food industry. The systematic increase of ecological awareness and related environmental protection requirements makes it necessary to look for new solutions that meet strict criteria in this area. These requirements include, among others, ensuring appropriate conditions for the operation of machinery and equipment in industries related to direct and indirect impact on the ecosystem [L. 1-2]. In this area, the issues related to the design of lubricants that provide desirable operating properties that meet the high environmental requirements are important [L. 3-4].

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The analysis of food production and hygiene requirements shows that the lubrication of machines in the food industry forces the use of only certified lubricants, and all components used to manufacture food items must meet the requirements of the level of certification, which results in a restriction in the use of a wide range of oil bases, thickeners, and enriching additives [L. 5-6]. The standard regarding machinery for the food industry clarifies the hazards that machinery can have for manufactured food and also what lubricants can be used in food zones [L. 7]. Food production plants adapt the production conditions to the requirements set by the EU directives on safety and hygiene in the production of food and particularly take into account the requirements for machine lubrication and the use of only certified lubricants.

The increase in the competitiveness of the food production industries and the analysis of the development trends of the machinery sector for the food industry confirmed the need to compose lubricants that meet requirements of functional properties and the precise selection of individual components for a final product that meets the stringent environmental standards.

The Regulation of the European Parliament assumes the needs of strategic research in the field of developing environment-friendly lubricants mainly by improving the methods of their production in low-waste or waste-free technologies, as well as developing the new products based on safe raw materials in accordance with legal regulations regarding environmental protection and food production safety [L. 10].

The development of technologies of pro-ecological lubricants based on non-toxic components determines the use of vegetable oils or mixtures of vegetable oils with mineral or synthetic oils as a dispersing phase of plastic lubricants, which meet the physicochemical, lubricating, rheological, and ecological requirements [L. 11-14]. The solution of the presented problems requires undertaking scientific research, the verification of the obtained results in laboratory conditions, and their confirmation in industrial applications. However, the introduction of a new generation of lubricants developed based on non-toxic ingredients into the lubrication technique will undoubtedly contribute to the increase of competitiveness of enterprises. In research related to the design of lubricants, it was very useful to apply modelling methods to provide the predictive models supporting the method of grease development [L. 15–19].

The authors of this paper, in prior publications, presented the results of research on new lubricants development with application of components that meet ecological requirements, in particular, oil bases that also meet functional requirements [L. 20–24].

In this article, the authors presented the influence of the type of oil base on the functional properties of developed lubricants, dedicated for use in the agri-food industry.

MATERIALS AND METHODS

The subjects of the research were lubricants developed on the basis of vegetable oil (1A, 1B) and lubricants prepared on a mixed base of vegetable-synthetic oils (2AS, 4AS). Apart from the dispersion and dispersed phase, the component of the evaluated lubricants was the BCH multifunctional additive containing an antioxidant, a corrosion inhibitor, and EP/AW additives. [L. 22, 23]. Lubricants 1B, 2AS, and 4AS contained a multi-functional additive. Lubricant 1B, which was based on vegetable oil contained 1.5% m/m BCH added, and 2AS and 4AS greases prepared on a mixed plantsynthetic oil base contained, respectively, 2.5% m/m BCH (2AS) and 1.5% m/m BCH (4AS).

All developed lubricants were subjected to tribological tests, determining anti-wear and anti-seizure properties and durability. Tribological tests were carried out using a four-ball tribotester, at ambient temperature, under conditions of a constant and linear load of the friction node.

Anti-wear properties of the tested lubricants were determined in an hour test, carried out under constant load conditions, P=392 N, of the friction node at constant spindle revolutions of 500 ± 20 rpm at a temperature of $20\pm5^{\circ}$ C. After tests, the diameter (*d*) of the wear scare on the stationary ball was measured, then the limit value of the wear load was calculated from the relation $G_{oz} = 0.52 \ P/d^2$, which included the load on the friction pair (P) and the average diameter of the wear trace (*d*).

Measurements of the diameter of the wear scare of the ball were made using a Nikon NM-40 optical microscope, measuring the size of the wear scare, in the direction parallel and perpendicular to the direction of friction. The determined G_{oz} parameter was adopted as a measure of the anti-wear performance of the tested lubricants. The test balls were made of 100Cr6 bearing steel. The seizure loads were determined in the conditions of the linear increase of friction node load while maintaining a constant rate of 409 N/s load increment, a spindle speed of 500 ± 20 rpm, and a slip speed of 0.19 m/s. Resistance to seizure of the friction the graph.

After one-hour of wear testing, the surface conditions of abrasive elements were evaluated and quantitative and qualitative identification of elements in the trace of wear were carried out using the SEM/EDS technique (SEM scanning electron microscopy and EDS energy dispersion spectrometry).

Due to the possibility of lubricant exposure during operation to thermal and oxidizing agents, it was necessary to assess their oxidative stability. A PetrooxyTM

apparatus from Petrotest Instruments was used to assess the thermo-oxidative stability. The principle of the method consisted of passing oxygen through a 10 g sample, heated to 80°C and 120°C. As a criterion for assessing oxidative stability, a 10% reduction of time was adopted, referred to as the oxidation induction time.

The main parameters determining the operational durability of a given lubricant are the working conditions of the bearing, namely, load, operating temperature, and rotational speed. Therefore, prior to the commencement of operational tests, the service life of lubricants was evaluated by performing tests under constant load conditions [L. 25].

RESULTS AND DISCUSSION

The oxidation stabilities of the lubricants were determined on the basis of the Petroxy test, which was carried out in the presence of oxygen at temperatures of 80°C and 120°C, which determined the oxidation induction time, **Fig. 1**.

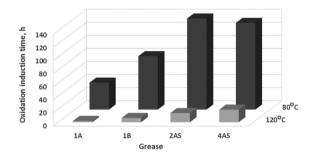


Fig. 1. Oxidative stability of lubricants determined at 80°C and 120°C – Petroxy test

The most favourable thermo-oxidative stability at 80°C and 120°C were found for 2AS and 4AS greases developed using a base mixture of vegetable and synthetic oils. It was also observed that the content of the multifunctional additive influences the increase in the oxidative stability of greases. In the case of vegetable lubricants, it was found that the multifunctional additive, in the amount of 1.5% m/m, enhances the oxidation stability of the 1B grease than the 1A grease. However, increasing the amount of the additive in 2AS plant-synthetic grease to the level of 2.5% m/m slightly changes the stability of the 4AS grease containing 1.5% m/m of this additive. Lubricants 1A and 1B produced using a base of vegetable oil were characterized by significantly lower oxidation stability compared to lubricants 2AS and 4AS based on a mixture of vegetable oil and synthetic oil.

Anti-wear properties were determined based on the diameter of the wear trace on the ball after an

hour testing, and then the G_{oz} limit load of wear was calculated. The results are shown in **Figure 2**.

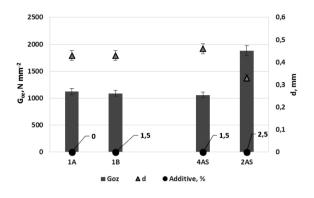
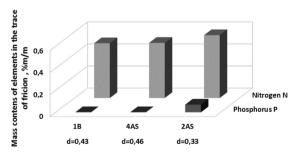


Fig. 2. Effect of content of multifunctional BCH additive on antiwear properties of lubricants (1A, 1B, 2AS, 4AS), limit load of wear G_{oz} and average diameter of wear scare, d

Rys. 2. Wpływ zawartości wielofunkcyjnego dodatku BCH na właściwości przeciwzużyciowe smarów (1A, 1B, 2AS, 4AS), graniczne obciążenie zużycia G_{oz} oraz średnia średnica śladu zużycia, d

The introduction of the same amount of additive (1.5% m/m) to lubricants 1A (vegetable) and 4AS (plant-synthetic) resulted in slight changes of anti-wear properties, expressed in the limit value of G_{oz} wear. The larger amount of additive (2.5% m/m) in the 2AS vegetable-synthetic lubricant resulted in a significant reduction in the diameter of wear scare of ball. After an hour of wear testing, the surface conditions of friction elements were evaluated, and quantitative and qualitative analysis of chemical elements in the trace of wear using SEM/EDS technique was performed, **Fig. 3**.



Phosphorus P Nitrogen N

- Fig. 3. Results of EDS X-ray analysis of the surface of the ball, after one hour wear test (*d* mean diameter of wear scare) in the environment of tested lubricants 1A, 1B, 2AS, 4AS
- Rys. 3. Wyniki analizy rentgenowskiej EDS z powierzchni kulki po godzinnym teście zużyciowym (d – średnia średnica skazy) prowadzonym w środowisku badanych smarów – 1A, 1B, 2AS, 4AS

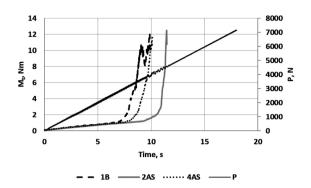
In the trace of friction after the tests carried out in the presence of 1A vegetable grease, no presence of

Rys. 1. Stabilność oksydacyjna środków smarowych wyznaczona w temperaturze 80°C i 120°C – test Petrooxy

active elements, such as phosphorus and nitrogen, were found on the surface. They appeared on the surface of the balls after tests carried out in the presence of 1B, 2AS, and 4AS lubricants, which contained a multi-purpose additive Brad-Chem 351 (BCH). A significant effect of additive on the quantities identified in the surface layer of active elements was found. They were derived from the used multifunctional additive and observed in the chemically modified surface layer as nitrogen and phosphorus. The obtained results confirmed that the content of the BCH multifunctional additive in lubricants affects the lubricant-surface interactions, which results in the increase in resistance to wear and as result the wear scare diameter reduction, **Figs. 2** and **3**.

The lubricants that were the subjects of the tests showed different courses of the frictional moment changes as a function of the linearly increasing load and were characterized by variable resistance to scuffing. **Figure 4** compares the friction torque curves obtained for vegetable grease 1B and plant-synthetic 2AS, 4AS lubricants, which contained a multifunctional additive in their composition.

Depending on the composition of the tested lubricants, there is a difference between the courses of the moment of friction, which is expressed in the delay of the initiation of scuffing as well as the subsequent seizure. When the lubricating film is interrupted, the friction torque increases dramatically until it becomes seized, under the load called "the limiting seizure load P_{or} ". Analysing the courses of the frictional moment of the composed greases, it was found that initiation of scuffing for 1B grease occurs under a lower load than for 2AS and 4AS greases. The observed faster initiation of scuffing and seizure in the case of 4AS (1.5%m/m BCH) grease in comparison with 2AS (2.5% m/m BCH) is associated with a lower content of the multifunctional additive. The boundary layers created with 2AS grease are more durable than when using 1B and 4AS greases.



- Fig. 4. The curves of Mt friction torque as a function of test time, obtained under conditions of continuous increase of load *P*, for developed lubricants: based on vegetable oil 1B and based on a mixture of vegetable oil with synthetic 2AS, 4AS
- Rys. 4. Krzywe momentu tarcia M_t w funkcji czasu badania, uzyskane w warunkach ciągłego wzrostu obciążenia *P*, dla opracowanych środków smarowych: na bazie oleju roślinnego 1B oraz na bazie mieszaniny oleju roślinnego z syntetycznym 2AS, 4AS

As it results from the presented results, the type of dispersive phase and the amount of the multifunctional additive used have a significant influence on the antiseizure properties of the composed greases.

The next stage of research was undertaken to evaluate the operational properties of the composed greases in relation to the standard [L. 25]. The resistance to wear of the tested lubricants was evaluated on the test stand with the friction contact roller - ring [L. 12]. Optical profilometry for the analysis of the geometric structure of the surface was used to assess the wear scare of rollers. The exemplary wear scares that were obtained for the 2AS grease under different loads of the friction junction are illustrated in **Figure 5**.

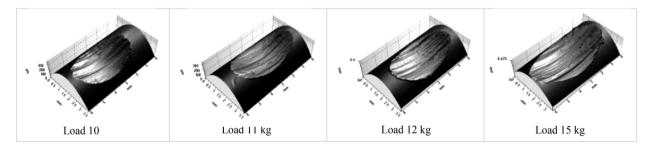


Fig. 5. The wear scare on the surface of the rollers after tests carried out under different loads, in the presence of 2AS grease

Rys. 5. Ślad wytarcia na powierzchni wałeczka po testach prowadzonych przy zmiennym obciążeniu w obecności smaru 2AS

Analysis of the acquired surface images of the friction element made it possible to determine the volume consumption at various loads of the friction contact. The tests carried out showed that, in the test conditions, the 2AS grease worked without disturbances at all given loads. At higher loads of 12 kg and 15 kg,

a slight increase in the wear volume was observed compared to the lower loads. Under the conditions of the tests, 2AS grease was characterized by a good wear resistance, so it was reasonable to verify its properties under operating conditions.

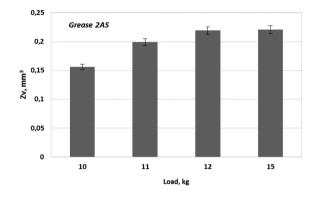


Fig. 6. Volume wear Z_y of the friction contact element lubricated with 2AS grease at different load

Rys. 6. Zużycie objętościowe Z_y elementu węzła tarcia smarowanego smarem 2AS przy różnym obciążeniu

After the tests in the model conditions adopted as the basis for the developed greases, tests were carried out in the devices selected for conducting supervised use. The correctness of the functioning of the tested devices with the application of the composed greases was monitored during the production mode. The efficiency of the functioning of the developed greases was evaluated in devices in the baking and confection industries as well as in the vegetable processing industry [L. 12, 24].

Lubricants were tested in bakery devices under highly dusty conditions. For example, 2AS grease was used in spherical roller bearings (22310) and in articulated–sliding nodes (GE45ES) in a lifting tipping system by dosing the right amount of grease in selected intermeshing elements. In addition to monitoring the functioning of the applied lubricant in the bearing, the monitoring of the correct functioning of the equipment in the production mode was carried out.

The 4AS grease was used for testing in a confection plant, where it was applied in screw conveyor bearings. During operation, the lubricants were supplemented in accordance with technical requirements. The monitoring of the bearing operation in which the grease was applied consisted of temperature control and noise level measurement. It has been observed that the replacement of the lubricant caused a drop in the temperature of the bearing housing and a reduction in the noise level. This indicates that the lubricant used functions properly in such extreme conditions with a large amount of sugar dust. Based on the previous observation, it was found that the developed grease can be a replacement for the previously used lubricant in the slide bearing of the screw conveyor.

During operation, there were no symptoms determining the need to replace grease in the bearing, and there were no changes in the conditions and disturbances in the correct operation of the bearing were observed. Verification of the developed lubricants confirmed that the lubricants fulfilled their functions very well in conditions of high levels of sugar dust, humidity, and loads.

CONCLUSIONS

As a result of the research, it was confirmed that the development of an ecological lubricant with usable properties appropriate to the operating conditions requires careful selection of high-quality components.

The research confirmed that the developed procedure for designing the lubricant should take into account the stages of both the selection of components at each stage of grease design as well as verification tests on each of component.

As a result of the work carried out in accordance with the adopted procedure, including model tests in laboratory experiments, verification of the properties of developed lubricants under extreme operating conditions, namely high levels of dust, moisture, and the presence of water, the lubricants were selected that meet the required assumptions.

The ecological lubricant developed was successfully verified at work after direct application in food industry machinery nodes.

The lubricant developed as a result of cooperation between research and development units and industrial enterprises ensures the safety of use for both people and the environment and meets all the functional requirements assumed.

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REFERENCES

- 1. El-Adly R.A., El-Sayed S.M., Ismail M.M.: J. Synthetic Lubrication, (2005), Vol. 22, pp. 211–223.
- 2. Schneider M.P.: Plant-oil-based lubricants and hydraulic fluids. J Sci Food Agric 86, (2006): 1769–1780.
- 3. Report no. 10/12: Primary biodegradation of petroleum hydrocarbons in seawater. CONCAWE Brussels December 2012.

- 4. Beran E.: Ocena biodegradowalności słabo rozpuszczalnych w wodzie związków organicznych na przykładzie olejów smarowych. (in Polish) Problemy Ekologii, 2008, R. 12, nr 3, s.153–159.
- 5. Girma Biresaw G., Bantchev G.B., Cermak S.C.: Tribological properties of vegetable oils modified by reaction with Butanethiol. Tribol Lett (2011) 43:17–32. DOI 10.1007/s11249-011-9780-z.
- 6. El-Adly R.A., Bedier A.H., Hussein M.F., Ismail E.A., EL-Emary M.M.: International Journal of Scientific & Engineering Research, Vol. 5, Issue 6, pp. 755–762, 2014.
- 7. Drabik J., Iłowska J., Gniady J., Szmatoła M., Semeniuk I., Kozupa M.: Wpływ bazy olejowej i składu zagęszczacza na właściwości użytkowe smarów plastycznych. (in Polish) Przem. Chem. 2012, 9, nr 10: 1922–1926.
- Muphy W.R., Blain D.A., Galiano-Roth A.S.: Synthetic bases benefits of synthetic lubricants in industrial applications. J. Synth. Lur. 2002, 18 (4), 301–325.
- 9. Norma PN-EN 1672-2+A1:2009. Maszyny dla przemysłu spożywczego Pojęcia podstawowe Część 2: Wymagania z zakresu higieny. (in Polish).
- Rozporządzenie (WE) nr 178/2002 Parlamentu Europejskiego i Rady z dnia 28 stycznia 2002r. ustalające ogólne zasady i wymagania prawa żywnościowego, ustanawiające Europejski Urząd ds. Bezpieczeństwa Żywności oraz ustanawiające procedury w sprawie bezpieczeństwa żywności" (ze zm.) (in Polish).
- 11. Drabik J., Kozdrach R., Wolszczak M., Wrona M.: Przemysł Chemiczny, 97/9 (2018) s. 1538-1541. DOI:10.15199/62.2018.9.30.
- Drabik J., Rogoś E., Trzos M., Kozdrach R., Wolszczak M., Michalski J., Janecki J., Piątkowski M.: Specjalistyczne środki smarowe gwarantujące bezpieczeństwo produkcji żywności.Rozdział w monografii pt: "Innowacje w Przemyśle Chemicznym", (in Polish) ISBN 978-83-916361-1-4, 2018, 80.
- Drabik J., Wrona M., Kozdrach R.: Możliwość wykorzystania metod spektroskopowych do oceny zmian jakości olejów smarowych po wymuszeniach cieplnych i mechanicznych. (in Polish) Artykuł w monografii pt: Nauka i przemysł – metody spektroskopowe w praktyce, nowe wyzwania i możliwości. ISBN 978-83-945225-4-4. Lublin, 2018s. 374-379.
- Cousseau T., Graca B.M, Campos A.V, Seabra J.H.O: Influence of grease rheology on thrust ball bearings friction torque. Tribology International 46 (2012) 106–113.
- 15. Nagendramma P., Kaul S.: Development of ecofriendly/biodegradable lubricants: An overview. Renewable and Sustainable Energy Reviews, Volume 16, Issue 1, January 2012, Pages 764–774.
- Iłowska J., Chrobak J., Grabowski R., Szmatoła M., Woch J., Szwach I., Drabik J., Trzos M., Kozdrach R., Wrona M., Molecules 2018, 23, 2025; DOI: 10.3390/molecules23082025.
- Trzos M., Drabik J., Iłowska J., Grabowski R.: Design of conditions for the modification of oil in the process of designing ecological lubricants. Tribologia, 2/2018, 133–138.
- Drabik J., Trzos M., Janecki J.T.: Effect of modelling of the grease content in the metal-polymer composite on the tribological resistance. Modelowanie wpływu zawartości smaru plastycznego w kompozycie metalopolimerowym na odporność na zużycie. (in Polish) Przemysł Chemiczny 92/3, 2013, 369–373.
- 19. Drabik J., Trzos M.: Improvement of the resistance to oxidation of the ecological greases by the additives. Journal of Thermal Analysis and Calorimetry 113 (1) 2013, 357-363.DOI: 10.1007/s10973-013-3090-7.
- 20. Studnik H., Iłowska J., Chrobak J., Szmatoła M., Szwach I., Grabowski R., Drabik J.: Badania porównawcze podatności modyfikowanych olejów roślinnych na biodegradację (in Polish), Przem. Chem., 97/12, 2018.
- Szmatoła M., Chrobak J., Grabowski R., Iłowska J., Woch J., Szwach I., Drabik J., Wrona M., Kozdrach R., Orlińska B., Grymel M.: Spectroscopic methods in evaluation of modified vegatable base oils from Crambeabyssinica. Molecules 2018.
- 22. Zgłoszenie patentowe P.427319.
- 23. Zgłoszenie patentowe P.427324.
- Drabik J., Trzos M., Wrona M., Kozdrach R., Wolszczak M., Duszyński G., Piątkowski M.:Modelowanie i ocena właściwości środków smarowych stosowanych w przemyśle spożywczym. (in Polish) Przem. Chem. 97/12, 2018.
- 25. Norma ASTM D 2981 94(2014): Standard Test Method for Wear Life of Solid Film Lubricants in Oscillating Motion.