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EFFECT OF THE BIO-LUBRICANT ON THE LUBRICATING PROPERTIES AND SURFACE OF THE FRICTION ZONE

WPŁYW BIO-SMARU NA WŁAŚCIWOŚCI SMARNE I POWIERZCHNIĘ STREFY TARCIA

Key words: inorganic bio-lubricant *Crambe abyssinica* seed oil, lubricating properties, coefficient of friction.

Abstract: Tests carried out on a rotational rheometer with a tribological cell enabled the simultaneous designation of lubricating and viscosity characteristics depending on temperature and load as a function of variable sliding speed. The analysis of the dependence of the coefficient of friction on the sliding speed provided the basis for evaluating the effectiveness of lubrication of steel tribosystems with bio-lubricants. Spectroscopic methods were used to monitor changes in the microstructure of the bio-lubricants after tests. The scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were used to characterise the morphology and structure of the surfaces of the tribosystem elements. The instrumental methods used confirmed that the test conditions did not destabilise the chemical structure of the lubricant and did not accelerate the oxidation process of the vegetable grease.

Słowa kluczowe: nieorganiczny bio-smar, olej z nasion Crambe abyssinica, właściwości smarne, współczynnik tarcia.

Streszczenie: Badania prowadzone na reometrze rotacyjnym z przystawką tribologiczną umożliwiły jednoczesne wyznaczenie charakterystyk smarnych i lepkościowych w zależności od temperatury i obciążenia w funkcji zmiennej prędkości poślizgu. Analiza zależności współczynnika tarcia od prędkości poślizgu stanowiła podstawę do oceny efektywności smarowania stalowych węzłów tarcia bio-smarami. Do monitorowania zmian mikrostruktury bio-smarów po testach wykorzystano metody spektroskopowe. Do scharakteryzowania morfologii i struktury zużytych powierzchni elementów węzła tarcia wykorzystano skaningową mikroskopię elektronową (SEM), spektroskopię z dyspersją energii (EDS). Zastosowane metody instrumentalne potwierdziły, że warunki testów nie wpłynęły destabilizująco na strukturę chemiczną smaru i nie przyspieszyły procesu utlenienia smaru roślinnego.

INTRODUCTION

The environmental impact of used petroleum products constitutes a major ecological threat to soil, groundwater, and organisms living in them. In such a situation, there is a real need to search for substitutes and develop new technologies of lubricant production according to the latest trends in green technologies and products with the lowest environmental impact **[L. 1–3]**. The society's growing environmental awareness and concern for the environment, as well as the policy of sustainable development are all contributing to the search for mineral oil substitutes. The use of vegetable oils to produce environmentally friendly lubricants is becoming increasingly popular **[L. 4–7]**.

It seems, the most promising to base the lubricant production technology on renewable raw materials that are widely available. The suitability of using vegetable oil substitutes as a lubricant base

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is supported by such arguments as safety of use and environmental protection. Lubricants based on renewable raw materials should have functional properties comparable to those of petroleum products, because, as their substitutes, they must provide the required durability of lubricated tribosystems of machines and devices **[L. 8–13]**.

The development of new technologies entails the development of bio-lubricants based on non-food vegetable oils, which successfully provide the functional and ecological properties required by the application. The use of the *Crambe abyssinica* seed oil, with oxidation resistance comparable to mineral oils, opens up a new perspective for environmentally friendly lubricants **[L. 14–19]**. The production of a new generation of lubricants based on renewable biocomponents contributes to the development of new lubricants, which, besides ecological criteria, meet functional criteria providing the adequate lubrication of a tribosystem **[L. 20–22]**.

This paper presents lubrication characteristics of a bio-lubricant obtained using a rotational rheometer with a tribological cell. The aim of the study was to develop a procedure for evaluating the bio-lubricant based on the dependencies obtained and the analytical methods used. For the purpose of the study, to evaluate the influence of the temperature and frictional load on the changes in the structure of the tested lubricant and in the surface area of the friction zone, spectroscopic methods were used.

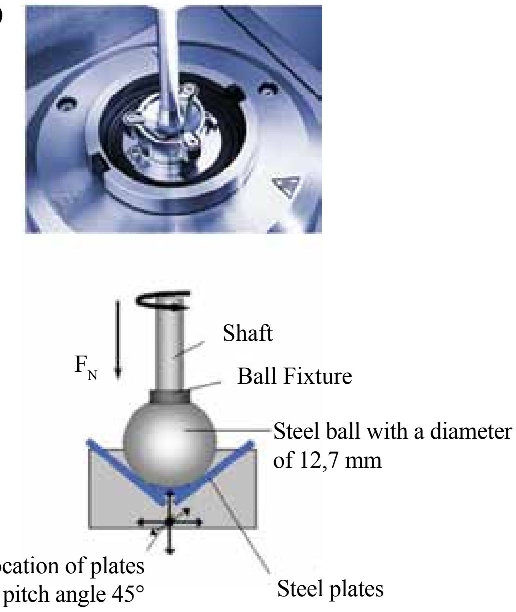


Fig. 1. Tribological cell of the MCR 102 rotational rheometer: a) image of the tribosystem, b) diagram of the tribosystem

Rys. 1. Przystawka tribologiczna reometru rotacyjnego MCR 102: a) zdjęcie węzła tarcia, b) schemat węzła tarcia

SUBJECT AND METHOD OF RESEARCH

The subject of the study was a vegetable grease (*Bio-grease A*) developed on the basis of a *Crambe Abissinica* vegetable oil and an inorganic thickener in the amount of 8% m/m.

The tribological tests were carried out using a MCR 102 rheometer (Anton Paar) equipped with a tribological cell, in which three rectangular plates, fixed in a holder, were pressed down with an appropriate force by a ball fixed in a spindle rotating at an appropriate speed **(Fig. 1)**.

The tribological cell allowed the authors to carry out tests at a given temperature and load. The tribosystem consisted of a ball with a diameter of 12.7 mm and three plates with dimensions of 15 × 5 × 2 mm. The tribosystem elements were made of bearing steel with roughness Ra = 0.3 µm and hardness 60–63 HRC. The tribological tests were carried out at tribosystem loads (1 N, 5 N, 10 N), variable speeds from 0 to 2100 rpm and at a specified temperature (20°C and 120°C). Immersion lubrication was used during the tests. The tests were carried out according to a developed procedure with a running-in phase with a load of 0.5 N, conditioning to the required temperature value and the carried out the appropriate tests, a next 6 measurements. The tests were carried out according to a developed procedure with a running-in phase with a load of 0.5 N, conditioning to the required temperature and the carrie out 6 measurements.

Bio-grease A tests were performed at two temperatures and under three different F_N loads **(Table 1)**. During the bio-lubricant tests, the viscosity and friction factor were simultaneously recorded as a function of sliding speed (0.01– 1 m/s) **(Fig. 1)**.

The infrared spectroscopy was used to monitor changes in the structure of the bio-lubricant after tests. The tests were carried out using a PE System 2000 FT-IR spectrophotometer. The spectra were recorded using the ATR-FTIR method (TT/IR6200). Lubricant samples were applied to a cell containing a ZnSe monocrystal. Spectra were recorded in the wavelength range 4000–650 cm^{-1} with a resolution of 4 cm^{-1}; 30 scans were carried out for background and measurements.

Table 1. Conditions of tests for *Bio-grease A*
Tabela 1. Warunki badań tribologicznych Bio-smaru A

Sample symbols of Bio-grease A	Test conditions				
	Load, N			Temperature, °C	
	1	5	10	20	120
A-1 N-20	x	-	-	x	-
A-1 N-120	x	-	-	-	x
A-5 N-20	-	x	-	x	-
A-5 N-120	-	x	-	-	x
A-10 N-20	-	-	x	x	-
A-10 N-120	-	-	x	-	x

A Hitachi FE-SEM SU-70 field emission scanning electron microscope equipped with a Thermo Scientific EDS energy dispersive X-ray microanalyser (SEM/EDS) was used to analyse the friction track surface after the tests. A quantitative and qualitative EDS analysis of the elemental composition from the friction track surface was performed, with the following working parameters: accelerating voltage 15 kV, working distance 30°, SE detector. SEM images were taken at a magnification of 150x.

TEST RESULTS

To investigate the impact of load and temperature on the lubrication sliding efficiency of the tribosystem with vegetable grease, the tests were carried out using a rheometer with a tribological cell. The lubrication characteristics of *Bio-grease A* were carried out as a function of the test conditions **(Table 1)**. The influence of tribosystem load and temperature on changes in the coefficient of friction as a function of sliding speed was evaluated. The total coefficient of friction is shown in **Figure 2**.

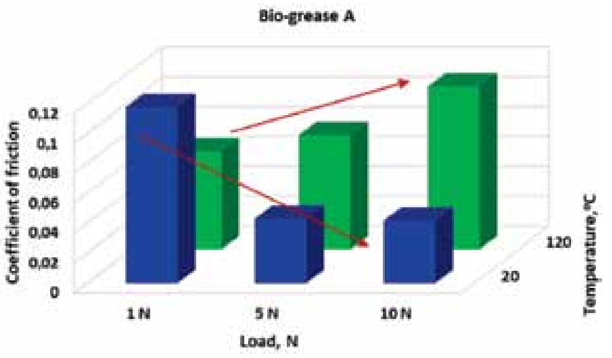


Fig. 2. Impact of tribosystem load and temperature on the coefficient of friction
Rys. 2. Wpływ obciążenia węzła tarcia i temperatury na współczynnik tarcia

The tests carried out showed that for the same tribosystem loads vegetable bio-lubricant A in sliding contacts behaves quite differently at 20°C and at 120°C. At 20°C, an increase in the tribosystem load caused a significant reduction in the coefficient of friction, which was favourable in determining the range of possibilities of lubrication of tribosystems with vegetable bio-lubricants. When vegetable lubricants are used, the temperature can change their chemical structure and, consequently, the oxidation products, which can hamper the work of lubricated tribosystems. Tests at 120°C showed that at higher temperature the coefficient of friction and load increased. It should be noted that the possibility of the formation of oxidation products at this temperature may have an impact on changes in the observed characteristics, increasing the value of the coefficient of friction and tribosystem load. Therefore, spectroscopic methods were used to analyse and explain the changes occurring on the surface of the friction elements and in the structure of the bio-lubricant.

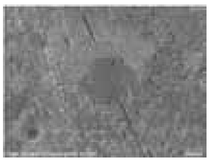
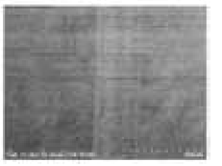
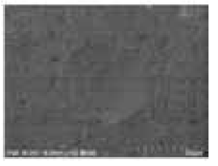
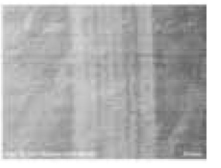
After tribological tests, the impact of the test conditions on the morphology of the wear surfaces of the tribosystem elements and the change in quality of the vegetable grease were evaluated.

Methods based on surface analysis, namely scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS), were used to characterise the surface morphology. After tests, the tribosystem elements lubricated with Bio-grease A were carried out the scars of wear on the ball and plate surfaces. The elements tribosystem were rated on the scars of wear ball and plate after test. **Table 2** shows examples of scanning images of the wear scars on the sliding elements of the tribosystem lubricated with bio-lubricant by load of 5 N, at temperatures of 20°C and 120°C. The size (i.e. the width and diameter) of the wear scars on the tribosystem elements and on the ball and plate surfaces depended on the test conditions **(Figure 3)**.

The impact of the tribological test conditions on the changes occurring on the surface of wear scars of tribosystem elements lubricated with *Bio-grease A* were evaluated by analysing of wear scars surface of tribosystem elements. The impact of the tribological test conditions on the changes occurring on the surface of wear scars of elements lubricated were evaluated by analysing of wear scars surface. It was observed, that as a result of an increase in load and in temperature, the number of wear scars on the ball grew and the diameter of

Table 2. SEM scanning images of wear scars on the surface of tribosystem elements after test carried out under a load of 5 N, temperature of 20°C and 120°C; magnification 150x

Tabela 2. Obraz skaningowy SEM zużytych powierzchni elementów węzła tarcia po testach wykonanych pod obciążeniem 5 N i w temperaturze 20°C oraz 120°C; powiększenie 150x

Operating condition of bio-lubricant A	Wear scar on the plate surface	Wear scar on the ball surface
A-5 N-20°C	 150x	 150x
A- 5 N-120°C	 150x	 150x

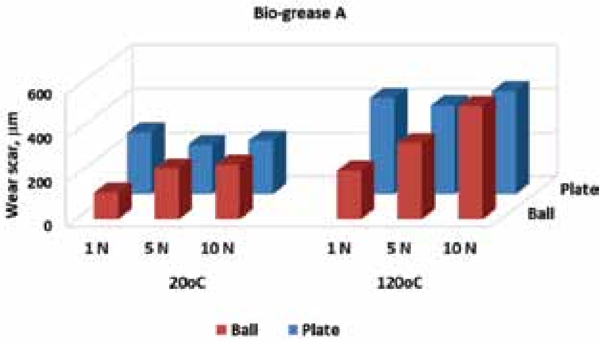


Fig. 3. Effect of tribosystem load and temperature on the width of the wear scar on the ball and the diameter of the wear scar on the plates

Rys. 3. Wpływ obciążenia węzła tarcia i temperatury na średnicę skazy na kulce oraz szerokość śladu na płytkach

wear scars on plates increased. At the same time, it was found, that the changes occurring on the surface of plates were varied of wear and showed a decreasing trend with increasing load in tests carried out at 20°C. At the same time, it was found, that the changes occurring on the surface of plates showed a decreasing trend with increasing load, in at 20°C. With the same load on the friction node, a significant effect of temperature on the wear occurring on the tribosystem elements was found, **Fig. 2**. With the same load on the friction node, a significant effect of temperature on the wear the tribosystem elements was found, **Fig. 2**.

The tests carried out confirmed the increased effect of the temperature 120°C, used in the test on the wear scar on elements of sliding tribosystem. The tests carried out confirmed the increased effect of the temperature 120°C, on the wear scar on the ball and plate. However, the trend of increasing scars, both on the ball and on the plates, with increasing load was maintained at all temperatures realized in the test. The trend of increasing scars, both on the ball and on the plates, with increasing load was observed for temperature 20°C and 120°C realized in the test. The knowledge of the behaviour of bio-lubricants at elevated temperatures has a great operational importance, as it has a significant effect on the wear of cooperating elements of machines and devices in lubrication process.

The analysis of the surface friction zone after tests carried out under variable conditions of load and temperature in the presence of *Bio-grease A* confirmed the relationship between the determined coefficient of friction and the width of the scuffing scar on the ball and the scar diameter on the plates **(Figures 2** and **3)**. The comparison of changes in the coefficient of friction occurring at 20°C and in the diameter of wear scars on the plates shows that an increase in load resulted in a decrease in the values of the parameters measured **(Figures 2** and **3)**. On the other hand, at 120°C, an increase in the coefficient of friction and in the diameter of the wear scars on the plates could be observed when the load was increased **(Figures 2 and 3)**. The occurring changes reflect the interdependence between the lubricant, the elements of tribosystem, and extortions realised during triborheometry tests. The occurring changes reflect the interdependence between the lubricant, the elements of tribosystem, and extortions realised in triborheometry tests.

The X-ray EDS analysis was used to evaluate the content of oxygen and carbon elements adsorbed on the wear scar surface of tribosystem elements lubricated with *Bio-grease A* **(Figure 4)**.

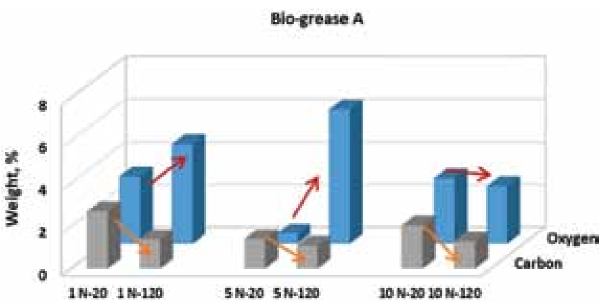


Fig. 4. Elemental content of oxygen and carbon adsorbed on the wear scars on plates determined by the EDS technique

Rys. 4. Zawartość tlenu i węgla zaadsorbowane na zużytych powierzchniach płytek, wyznaczone techniką EDS

From the comparison of the content of the adsorbed elements in the wear scars on the plates' surface it follows that an increase in the tribosystem load and in the temperature resulted in a decrease in the carbon content in the scuffing scar, which may indicate that a wear layer was created on the plate surface. The EDS tests of the scuffing scar surface confirmed that on the plates surface occurs an increased oxygen signal, which may indicate on the presence of oxidation products in the friction zone. The EDS tests confirmed that on the plates surface occurs an increased oxygen signal, which may indicate on the presence of oxidation products in the friction zone.

To explain the presence of increased oxygen value in the friction zone on the plates surface, the infrared absorption spectroscopy was used to obtain spectra of the grease of ATR-FTIR method. To explain the presence of increased oxygen value in the friction zone, the infrared absorption spectroscopy was used ATR-FTIR method. By analysing the spectra, the changes in the structure of *Bio-grease A* after tests under varied conditions of load and temperature were evaluated. An example of a spectrum of the grease after action of specified loads, i.e. 1 N, 5 N, 10 N and at 120°C are shown in **Figure 5**.

The analysis of the spectra of *Bio-grease A* obtained after the tribological tests did not found any changes in the structure of the lubricant in the range of carbonyl groups. The changes in the participation of carbonyl groups were analysed in the wavelength range 1800–1700 cm^{-1}. The analysed changes concerning the intensity of the band at wavenumber 1741 cm^{-1} and the band containing carbonyl group at wavenumber 1654 cm^{-1}. It was found that even after the tests carried out at 120°C with different loads of tribosystem, there was no decrease in the intensity of the band at wavenumber 1741 cm^{-1} associated with the hydrolysis of the ester bond occurring in vegetable grease. This indicates that oxygen identified by the EDS method on the surface of plates was not created as a result of oxidation process of the ester groups in vegetable oil.

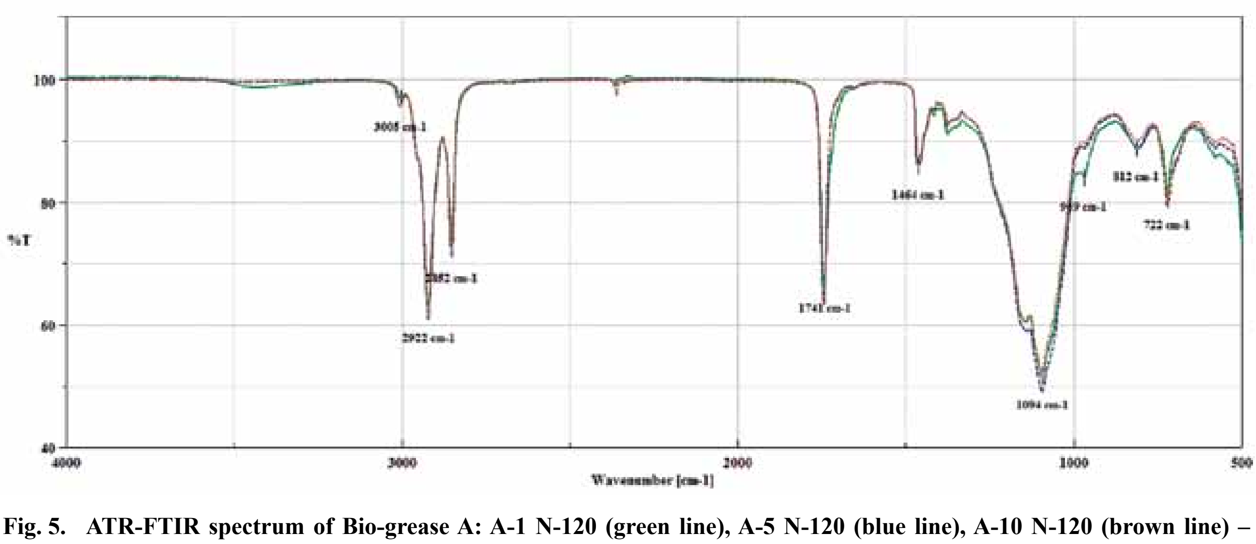


Fig. 5. ATR-FTIR spectrum of Bio-grease A: A-1 N-120 (green line), A-5 N-120 (blue line), A-10 N-120 (brown line) – wavelength range: 4000 cm^{-1} – 500 cm^{-1}

Rys. 5. Widma ATR-FTIR bio-smaru: A-1N-120 (linia zielona), A-5N-120 (niebieski), A-10N-120 (brązowy) – zakres 4000 cm^{-1} – 500 cm^{-1}

CONCLUSIONS

The accepted experimental program, based on rheometric and spectroscopic tests, allowed obtaining comprehensive information on the influence of the test conditions on change of lubricating properties, chemical structure of bio-lubricant and the morphology of the friction zone. The accepted experimental program, based on rheometric and spectroscopic tests, allowed obtaining comprehensive information of lubricating properties, chemical structure of bio-lubricant and the wear of the friction elements.

Tests carried out on a rotational rheometer with a tribological cell enabled the determination of lubricating characteristics, depending on the temperature and load as a function of sliding speed. The analysis of dependence between the coefficient of friction and the sliding speed constituted the basis for the evaluation of the effectiveness of lubrication of steel tribosystems with bio-lubricant. The experiments conducted enabled the evaluation of the impact of the test parameters on the evolution of the scuffing and wear properties.

The instruments based on the surface analysis, including scanning electron microscopy SEM and energy dispersive spectroscopy EDS, were used to characterise the surface morphology. It was found that the changes occurring on the surface of tribosystem elements were the result of interactions between the bio-lubricant and the tribosystem elements.

Changes in the structure of the bio-lubricant were evaluated by FTIR infrared spectroscopy. The small changes occurring in the chemical structure of the vegetable bio-lubricant provide of significant resistance on thermal and mechanical forces. Small changes occurring in the chemical structure of plant biolubricants prove their resistance thermal and mechanical influence

The obtained results provide the basis for the characterisation of bio-lubricants intended for use in friction associations to reduce friction, limitation of wear and maintenance necessary temperature of tribosystem. The obtained knowledge on the way to identify the key properties concerning the resistance on oxidation process of bio-lubricants is essential to ensure adequate lubrication and useful life of machines and devices elements.

The obtained results provide the basis for the characterization of bio-lubricants designed for use in friction zone to reduce friction, limitation of wear and maintenance necessary temperature of tribosystem. The obtained knowledge on the way to identify the key properties lubricating of bio-lubricants is essential to ensure adequate lubrication and useful life of machines elements.

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