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THE COMPARATIVE ANALYSIS OF PREPARED MIXTURES OF *CRAMBE ABYSSINICA* SEED OIL WITH MINERAL AND SYNTHETIC OIL

ANALIZA PORÓWNAWCZA OPRACOWANYCH MIESZANIN OLEJU Z NASION *CRAMBE ABYSSINICA* Z OLEJEM MINERALNYM I SYNTETYCZNYM

Key words:	vegetable oil from <i>Crambe Abyssinica</i> seeds, a mixture of vegetable and mineral oil, a mixture of vegetable and synthetic oil, the oxidation stability, the antiwear properties, the Raman spectrum analysis, the thermal and mechanical extortions.
Summary:	The base oils must meet the high technical and ecological requirements for the raw materials used in the production of lubricants. The relevant useful properties can be obtained by mixing the vegetable oil with mineral or synthetic oil. The paper presents the results of tests of mixtures of oil obtained from <i>Crambe Abyssinica</i> seeds with mineral and synthetic oil. A variation of tested mixtures with regard to thermo-oxidative stability and antiwear properties were found. The mixtures with the most favourable properties were recommended as a base oils during the production of ecological lubricants.
Słowa kluczowe:	olej roślinny z nasion <i>Crambe Abyssinica</i> , mieszanina oleju roślinnego z mineralnym, mieszanina oleju roślinnego z syntetycznym, stabilność oksydacyjna, właściwości przeciwzużyciowe, spektroskopia Ramana.
Streszczenie:	Oleje bazowe muszą spełniać wysokie wymagania techniczne i ekologiczne stawiane surowcom wykorzystywanym do produkcji środków smarowych. Odpowiednie właściwości użytkowe można uzyskać przez mieszanie oleju roślinnego z olejem mineralnym lub syntetycznym. W pracy przedstawiono wyniki badań mieszanin oleju pozyskanego z nasion <i>Crambe Abyssinica</i> z olejem mineralnym i syntetycznym. Stwierdzono zróżnicowanie badanych mieszanin pod względem stabilności termooksydacyjnej i właściwości przeciwzużyciowych. Mieszaniny o najkorzystniejszych właściwościach rekomendowano jako oleje bazowe podczas wytwarzania ekologicznych środków smarowych.

INTRODUCTION

The requirements of environmental protection justify the desirability of searching for new types of base oils for the production of lubricants. The changes occurring in the natural environment caused by overloading with used

petroleum products stimulate the interest of lubricants based on vegetable oils [L. 1–4]. Because of the structure, these oils are characterized by a variability of chemical and physical properties, and their chemical analysis is cumbersome, because they consist of a complex mixture of chemical compounds. However, using to test of fats

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and oils, besides analytical methods such as liquid or gas chromatography, modern instrumental methods spectral methods can be effectively used for the evaluation of changes in the quality of vegetable oils [L. 5, 6]. Recently, many publications concern the using of spectroscopic methods to evaluate the quality of vegetable oils and their identification [L. 7]. The analysis of vegetable oils may be carried out by absorption spectroscopy in the area of ultraviolet visible spectroscopy (UV-VIS) [L. 8], infrared (IR) spectroscopy [L. 9, 10–11, 15], or Raman spectroscopy [L. 12–14].

Chemically, the vegetable oils are glycerol esters of unsaturated fatty acids [L. 2–4]. The difficulty in using these oils in pure form is their low thermo-oxidative stability. Therefore, for the production of lubricants, it is preferable to use a mixture of oils, i.e. the vegetable oil and mineral or synthetic oil [L. 11, 16]. The increased of environmental pollution as well as limited crude oil resources and the growing demand for “environmentally friendly” pro-ecological lubricants to be used in bearings of machines working in a wide range of rotations and loads lead to the decision to undertake research works on the development of non-toxic and biodegradable lubricants based on renewable oil bases. This influenced the authors' interest in the possibility of using vegetable oils, in particular, inedible vegetable oils, as the base oil for lubricants [L. 4, 11, 16]. It was founded that non-

toxic agents should function properly in tribosystems, and after working, they should be characterized by high biodegradability, which will contribute to effective protection of the natural environment. In this situation, it was necessary to develop new pro-ecological lubricants based on oils characterized by a high resistance to the oxidation process.

The aim of the work was to evaluate changes in the structure of the developed oil mixtures subjected to the thermal and mechanical stresses. The research results allowed selecting a base oil with the best resistance to stresses, in the aspect of its use, as the base oil for pro-ecological certified lubricants intended for use in machines and devices in the food industry.

THE SUBJECT AND METHODOLOGY OF RESEARCH

The test samples were vegetable oil from *Crambe Abyssinica* seeds (A) and its mixtures with certified oils, i.e. paraffin oil of Fina A 360B type (AF) and polyalphaolefin oil SpectaSyn 100 (AS), with specific viscosity and temperature properties [L. 12]. The type and designation of the prepared oil bases before and after tests are summarized in **Table 1**.

Table 1. The type and designation of base oils

Tabela 1. Rodzaj i oznaczenie baz olejowych

The type of oil	The oil before the test	The oil after the PetroOxy test in temp. 80°C	The oil after the PetroOxy test in temp. 120°C	The oil after the antiwear hour test, G _{oz}
The vegetable oil A	A	A_80	A_120	A_G _{oz}
The vegetable and mineral oil AF 1.5:1	AF_60	AF_60_80	AF_60_120	AF_60_G _{oz}
The vegetable and mineral oil AF 1.7:1	AF_63	AF_63_80	AF_63_120	AF_63_G _{oz}
The vegetable and synthetic oil AS 1.5:1	AS_60	AS_60_80	AS_60_120	AS_60_G _{oz}
The vegetable and synthetic oil AS 1.7:1	AS_63	AS_63_80	AS_63_120	AS_63_G _{oz}

The oils were mixed in various proportions, AS_{1.5:1}; AS_{1.7:1} and AF_{1.5:1}; AF_{1.7:1}. The type and content of individual oils in mixture determined the viscosity class, **Tab. 2**. The mixtures of Abyssinian oil with mineral oil belonged to the VG 46 viscosity class, and Abyssinian oil with synthetic oil belonged to VG 150 class. Prepared samples of base oil were subjected to the oxidation process at the temperature of 80 and 120°C and the action of mechanical forces in an hour antiwear test. After the tests, the Raman spectra were made and

on this basis. Then, the influences of the processes on the degree of unsaturation of the base vegetable oil were evaluated.

The PetroOxy™ apparatus by Petrotest Instruments was used to evaluate the thermo-oxidative stability of the base oils. The method consists in passing oxygen through a 10 g sample of oil heated to a temperature of 80 and 120°C. The oxidative stability of tested oil bases was determined, and the criterion of evaluation was the time of pressure reduction by 10%, **Table 3**.

Table 2. The viscosity-temperature characteristics of base oils – mixtures of vegetable and mineral oil A:F and synthetic A:S

Tabela 2. Charakterystyki lepkościowo-temperaturowe baz olejowych – mieszanin oleju roślinnego z mineralnym A:F oraz z syntetycznym A:S

The test parameters	The base oils			
	The ratio of vegetable oil A to mineral Fina F A:F		The ratio of vegetable oil A to synthetic S A:S	
	1.7:1 The oil AF_63	1.5:1 The oil AF_60	1.7:1 The oil AS_63	1.5:1 The oil AS_60
The viscosity class according to ISO ¹⁷⁾	VG 46	VG 46	VG 150	VG 150
The kinematic viscosity at temp. 40°C ¹⁸⁾ , mm ² /s	51.13	50.66	171.4	140.2
The kinematic viscosity at temp. 100°C ¹⁸⁾ , mm ² /s	9.56	9.45	24.4	22.8
The viscosity index ¹⁹⁾ , WL	174	173	184	192
The pour point ²⁰⁾ , °C	-16	-17	-15	-17
The flash point ²¹⁾ , °C	248	244	258	274

The determination of antiwear properties was carried out using a T-02 tribological tester in accordance with the PN-76 /C-04147 standard with the load of the tribosystem 392 N, spindle rotational speed 500 ± 20 rpm, and for a time of 1 hour. The measure of wear resistance was the diameter of the flaws resulting on the surfaces of the balls and the limiting load of wear, **Table 3**.

The Raman spectra were obtained using a confocal Raman micro-spectrometer NRS 5100 (Jasco Corporation, Japan) equipped with an excitation laser with a wavelength of 532.12 nm and a CCD detector. The spectrometer working conditions were as follows: the diffraction grating 1800 lines/mm, the laser power 5.1 mW, numerical aperture 4000 μ m, resolution 8.4 cm^{-1} , objective magnification 20x, and exposure time 15 s.

THE RESULTS OF RESEARCH

The resistance to the oxidation process of vegetable oil and prepared oil mixtures was determined by the PetroOxy method by doing the determinations at the temperatures of 80°C and 120°C in an oxidizing atmosphere. The anti-wear properties of the tested oils were determined in an hourly wear test by evaluating the wear trace after the test. On this basis, the limiting load of wear G_{oz} was calculated according to the formula: $G_{oz} = 0.52 \cdot (392/d^2)$, where d – mean diameter of the friction trace, and 392 is the applied load. The obtained results are presented in **Table 3**.

The mixtures containing 63% Abyssinian oil and 37% mineral or synthetic oil were characterized by the

Table 3. The properties of starting oil and tested oil mixtures

Tabela 3. Właściwości oleju wyjściowego i badanych mieszanin olejowych

The type of oil mixtures	The parameters			
	The oxidation induction time, h		The antiwear properties	
	Temp. 80°C	Temp. 120°C	d, mm	G_{oz} , N/mm ²
The oil A	32.78	2.55	0.59	586.18
AS_60 S:A 1:1.5	18.89	2.56	0.62	530.82
AS_63 S:A 1:1.7	37.30	2.04	0.72	393.61
AF_60 F:A 1:1.5	31.77	2.16	0.55	674.54
AF_63 F:A 1:1.7	45.42	2.02	0.66	468.43

highest thermo-oxidative stability at the temperature of 80°C. The mixtures with 60% Abyssinian oil and 40% mineral or synthetic oil were less resistant under these conditions. At the temperature of 120°C, the thermo-oxidative stability of the oil mixture with 40% synthetic oil content was similar to the stability of Abyssinian oil. The stability of the other tested mixtures slightly decreased.

The test result indicate that the anti-wear properties of the mixture containing 40% mineral oil were better than the Abyssinian oil, while the mixtures containing 40% and 37% synthetic oil and 37% mineral oil had slightly worse of anti-wear properties.

The Raman spectra for the evaluation of changes in the structure of oil mixtures occurring during thermal and mechanical processes were made.

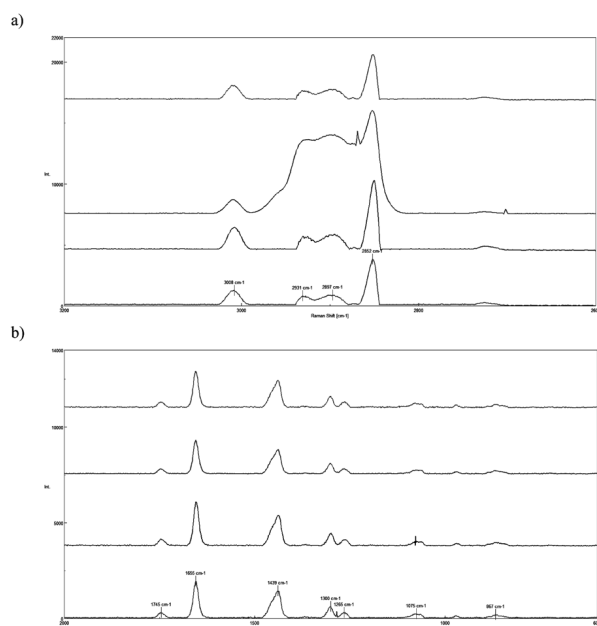


Fig. 1. The Raman spectra of vegetable oil from *Crambe Abyssinica* seeds: the starting oil A (green line), the A_80 oil after oxidation in 80°C (blue line), the A_120 oil after oxidation in 120°C (brown line), the A_G_{oz} oil after antiwear test (grey line) in the range of: a) 3200 – 2600 cm⁻¹ and b) 2000 – 600 cm⁻¹

Rys. 1. Widma Ramana oleju roślinnego z nasion *Crambe Abyssinica*: olej wyjściowy A (linia zielona), olej A_80 po utlenianiu w 80°C (linia niebieska), olej A_120 po utlenianiu w 120°C (linia czerwona), olej A_G_{oz} po teście zużycia (linia szara) w zakresie a) 3200 – 2600 cm⁻¹ oraz b) 2000 – 600 cm⁻¹

First, the structure of vegetable oil A obtained from *Crambe Abyssinica* seeds was evaluated on the basis of Raman spectra. Vegetable oil A contains fatty acids with the following molecular structure: erucic acid (C22:1), oleic acid (C18:1), linoleic acid (C18:2), and linolenic acid (C18:3). In the Raman spectra of vegetable oils from the *Crambe Abyssinica* seeds, bands were identified which were induced by stretching vibrations = C-H

and CH of CH₃ and CH₂ groups in the range of 3100 – 2800 cm⁻¹ and stretching vibrations of C = O, C = C groups, CH₂ scissor vibrations, CH₂ twisting, swinging = CH and stretching C-C occurring respectively at 1746, 1654, 1440, 1300, 1262, and 1087 cm⁻¹, **Figure 1**.

Based on Raman spectra analysis, the degree of the unsaturation of fatty acids occurring in vegetable oil, which was a component of the mixtures, was determined. For this purpose, the ratio of the intensity of the C = C bands at 1266 cm⁻¹ and the C-C bands at 1300 cm⁻¹, resulting from the vibrations of characteristic functional groups, was used, **Figures 1, 3**. The higher value of the ratio of these bands, the higher was the level of vegetable oil unsaturation. **Figure 2** shows the examples of Raman spectra obtained for the tested mixture of vegetable and mineral oil – the starting oil AF_63 and after thermal and mechanical extortions AF_63_80, AF_63_120, and AF_63_G_{oz}.

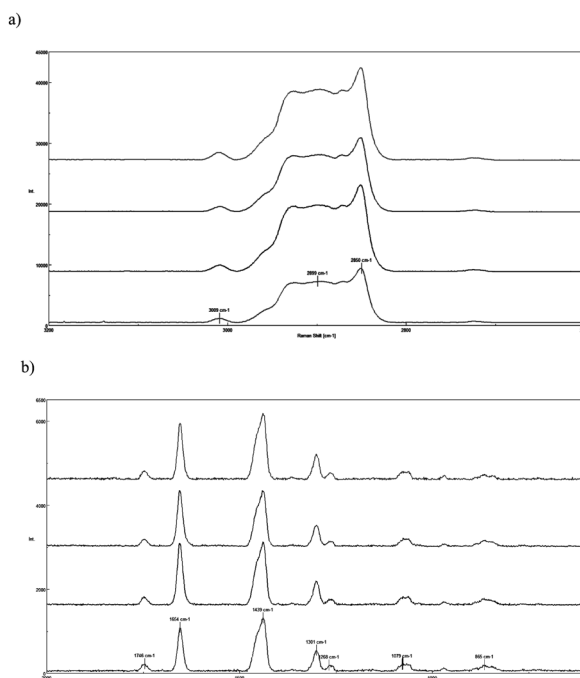


Fig. 2. The Raman spectra of the AF_63 oil mixture containing of 63% A oil and 37% F oil: the starting AF_63 oil (green line), the AF_63_80 oil after oxidation in 80°C (blue line), the AF_63_120 oil after oxidation in 120°C (brown line), the AF_63_G_{oz} oil after the antiwear test (grey line) in the range of: a) 3200 – 2600 cm⁻¹ and b) 2000 – 600 cm⁻¹

Rys. 2. Widma Ramana mieszaniny olejów AF_63 zawierającej 63% oleju A i 37% oleju F: olej wyjściowy AF_63 (linia zielona), olej AF_63_80 po utlenianiu w 80°C (linia niebieska), olej AF_63_120 po utlenianiu w 120°C (linia czerwona), olej AF_63_G_{oz} po teście zużycia (linia szara) w zakresie a) 3200 – 2600 cm⁻¹ oraz b) 2000 – 600 cm⁻¹

The spectra of mixtures of vegetable oil with mineral and synthetic oils showed the same bands as the spectra

of vegetable oil, but the intensity of these bands changed in relation to the ratio of vegetable oil A to mineral oil A:F, or synthetic oil A:S. The band intensity at 1654 cm^{-1} in the spectra of the tested mixtures generally decreased. For the mixtures of Abyssinian oil with mineral oil, this band showed a greater intensity at the lower level of vegetable oil content (60%), and lower at the higher level (63%). The spectrum of the mixture with 63% vegetable oil content was characterized by the greater intensity of the bands at 2929 and 2850 cm^{-1} from the spectrum of the vegetable oil. With a vegetable oil content of 60% in a mixture with synthetic oil, there was an increase of bands at 2902 and 2850 cm^{-1} compared to pure vegetable oil. In the case of the rest of the mixtures, the bands in the range of $3100 - 2800\text{ cm}^{-1}$ were decreased. The degree of fatty acid unsaturation was determined on the basis of the intensity ratio bands of I_{1263} / I_{1300} decreased in comparison to vegetable oil. In both mixtures, it was the lowest with the vegetable oil content of 63%, **Figure 3**.

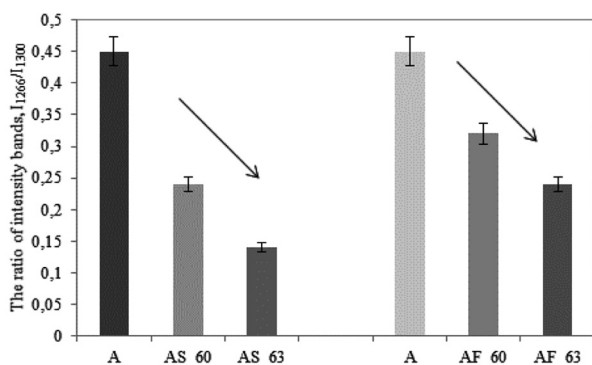


Fig. 3. The change of the ratio of intensity bands of I_{1266}/I_{1300} C=C to C-C bands of the starting base oils in compared to the oil A

Rys. 3. Zmiana stosunku intensywności pasma I_{1266}/I_{1300} wiązań C=C do C-C wyjściowych baz olejowych w porównaniu do oleju A

The different nature of the changes was observed in the spectra of the tested mixtures after the carried out tests. For a mixture containing of 60% vegetable oil and 40% synthetic oil in both spectra after oxidation test, the bands at 2929 and 2850 cm^{-1} were significantly greater than in the case of the starting oil mixture. The bands at 3010 and 1654 cm^{-1} also changed significantly under the influence of oxidation at 120°C , and the antiwear tests indicated a decrease in the intensity bands. For the mixture, after each test, there was a small decrease of the degree of the unsaturation of fatty acids. This decrease was a little greater for the mixture oxidized at 120°C , **Figure 4**.

In the case of the mixture AF_60 containing 60% vegetable oil and 40% mineral oil, the wide variety of spectra obtained after various tests was observed. The spectrum obtained for the mixture of oils after oxidation

at 120°C was the most similar to that obtained for the starting mixture. After the oxidation tests at 80°C and antiwear tests, the intensity of most of the bands were increased. In all cases, the degree of unsaturation was decreased slightly.

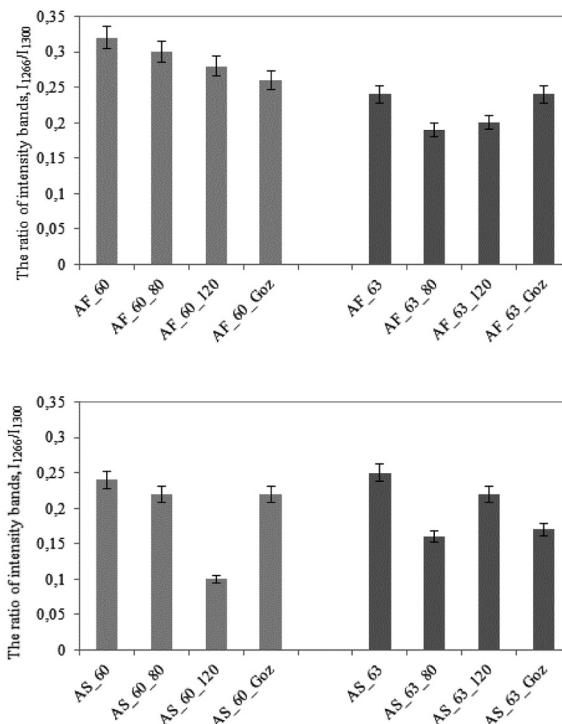


Fig. 4. The change of the ratio of intensity bands C=C (I_{1266}) to C-C (I_{1300}) of oil bases before and after PetroOxy tests in 80°C and 120°C and after an hour antiwear test G_{oz} : a) the type mixtures AF, b) the type mixtures AS

Rys. 4. Zmiana stosunku intensywności pasma wiązań C=C (I_{1266}) do C-C (I_{1300}) baz olejowych przed i po testach Petrooxy w temperaturze 80°C i 120°C oraz po godzinny teście zużyciowym G_{oz} : a) mieszaniny typu AF, b) mieszaniny typu AS

In the spectra recorded for the mixture of AS_63 containing 63% vegetable oil and 37% synthetic oil after the tests, the bands were more intense than the bands in the spectrum of the starting mixture. The band size at 2850 cm^{-1} was relatively low for the mixture after antiwear test, in other cases, it changed significantly. The tests caused a slight increase in the degree of the unsaturation of the oil mixture.

Under the influence of the tests, the intensity of the bands in the spectra of the AF mixture containing 63% vegetable oil and 37% mineral oil were increased. The band at 2850 cm^{-1} for the mixture after the oxidation test at 80°C and the antiwear test showed a greater intensity. Despite the changes of the size of bands, the tests did not significantly indicate the degree of the unsaturation of the tested oil mixture. However, the biggest change was observed after the oxidation process at 80°C . It was probably caused by the time of the action of the oxidizing

agent on the tested oils, because the determined time of oxidation induction for all mixtures were from approximately 2 to 46 hours, **Table 3**. The values of the degree of the unsaturation of the oil mixtures AF and AS before and after the tests, determined on the basis of the ratio of intensity bands I_{1266}/I_{1300} , are presented in the summary diagram, **Figure 5**.

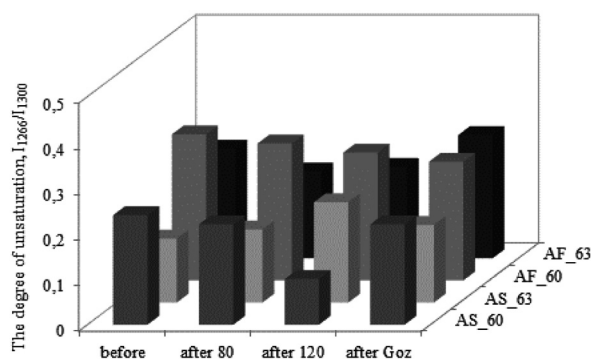


Fig. 5. The change of the ratio of intensity bands C=C (I_{1266}) to C-C (I_{1300}) of oil bases before and after PetroOxy tests in 80°C and 120°C and after an hour antiwear test

Rys. 5. Zmiana stosunku intensywności pasma wiązań C=C (I_{1266}) do C-C (I_{1300}) baz olejowych przed i po testach Petrooxy w temperaturze 80°C i 120°C oraz po godzinnym teście zużyciowym

When evaluating the influence of thermal and mechanical processes on changes in the quality of mixtures in the field of the analysed functional groups, it was found, that the temperature had a stronger influence on the occurring transformation than the effects of the antiwear test conditions. The observed diversity of the spectra obtained for the base oils after the use of various tests were associated with the conditions of these tests, i.e. the temperature, pressure, exposed to oxygen, load, and shear rate. This indicates a different direction and character of the transformation occurring in tested oil bases. The identified changes are the result of the transformation of the oil components during at the time of tests. It was found that the qualitative changes in the tested oils after tribological and thermo-oxidative tests are so significant that they can be monitored using Raman spectroscopy. This method is an excellent diagnostic instrument that allows one to monitor changes caused by the oxidation process of oil.

CONCLUSIONS

The test results of oxidative stability indicate the possibility of using vegetable oil from *Crambe Abyssinica* seeds as the main component of the base oils in mixtures with mineral or synthetic oil. The shown diversity of the tested mixtures in terms of quality was conditioned by differences in their chemical composition. The presence of mineral and synthetic oils significantly influenced the thermo-oxidative resistance and antiwear properties of the mixtures. The Raman spectroscopy was successfully used to identify changes occurring in the structure of vegetable oils, and the obtained spectra were the basis for the evaluation of the degree of the unsaturation of the tested oils. To analyse the samples, we took into account the ratio of the intensity bands identified in the spectrum of the tested mixtures coming from the fatty acids of vegetable oil, and the occurring changes were monitored on this basis. The influence of the oxidation process conditions and mechanical processes carried out in an hour antiwear test on the change of the unsaturation degree of the tested mixtures were evaluated, which proves the occurring changes in the structure of vegetable oil of the main component of the developed base oils.

Based on the carried out experiments, effects of thermal and mechanical processes on the change of the degree of unsaturation of vegetable oil contained in the mixtures were evaluated, and which of these factors had the greatest influence on the structure change, and also on the change of the quality of the base oils. This information is significant, because of the desired use of oil mixtures as a potential oil bases for the developed lubricants. The main assumption in the production of pro-ecological lubricants was the use of base oils with the highest resistance to the processes, while using above 60% vegetable oil in the composition with base oils. The obtained results have helped the selection and application of some of the tested mixtures, in particular, with synthetic oil, to prepare pro-ecological lubricating greases, which had the composition and production method covered by the patent claim [L. 22, 23].

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