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Lubricants based on synthesised emulsifier-stabilizer

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Hydroxylated fatty acids were synthesized by epoxidation of unsaturated fatty acids of waste food oil followed by hydrolysis by acylglycerol grouping and oxirane rings. The fatty acid aminoamides of the oils were synthesized by epoxidation of used fooding oil, followed by opening of the oxirane cycle and transamidation of acylglycerols with diethanolamine. The use of lithium soaps of hydroxyacids from waste food oil as an emulsifier-stabilizer, which acts as a lubricant thickener, and the introduction of fatty acid aminoamides of oils as an antioxidant additive into the composition of lubricants made it possible to obtain a plastic (lithium) lubricant. The physicochemical properties of lubricant were investigated and their quality indicators were compared with lubricant based on 12-hydroxystearic acid. The developed lithium lubricant is characterized by improved protective and tribological characteristics, increased stability to oxidation and mechanical stress, does not cause corrosion of non-ferrous metals, and is not inferior to lithium lubricant based on an industrial analog of 12-hydroxystearic acid. The lubricant is intended for friction units of machines and mechanisms. The properties of the resulting lubricant make it possible to predict its long service life in components and mechanisms and the prospects for using the components used in lubricant formulations. On the one hand, these studies make it possible to replace imported components for the production of lubricant thickeners, and on the other hand, to solve the problem of utilization of by-products of oil and fat production.

Keywords: recycled fatty materials, emulsifier-stabilizer, antioxidant additive, lithium lubricants

Introduction

The wear and tear of moving parts of machinery and structural materials, as well as the duration and efficiency of their operation, depend on the load and intensity of friction processes. To protect surfaces from premature wear, lubricants are used to significantly increase the durability of various machines and mechanisms. In general, lubricant is a complex dispersed system containing a base oil – a dispersion medium (65-95 %), emulsifiers-stabilizers that act as a thickener – a dispersed phase (3-30 %), and additives (0-10 %) [1-3]. It should be noted that the concentration of thickener in lubricants is relatively low, but it is the thickener that primarily determines their performance characteristics. The thickener forms the structure that gives the lubricant its plasticity, strength, colloidal stability, and other volumetric and mechanical properties. The dispersed phase of lubricants can be salts of higher carboxylic (fatty) acids – soaps, as well as solid high-melting hydrocarbons, inorganic and organic materials. The dispersion medium of lubricants can be petroleum, synthetic or mineral oils, *etc*. The classification of lubricants by type of thickener is important not only for assessing their quality level, proper use, marketing, and other technical and economic studies but also for determining the technological mode of production of certain lubricants.

Today, simple and complex lithium lubricants based on soap thickeners hold a key position in the global market. According to the National Lubricating Lubricant Institute, in 2022, they accounted for more than 62 % of total lubricant production in the United States, Western Europe, China and India [4]. The lithium lubricants market is expected to reach USD 3.5 billion by 2027, up from USD 2.6 billion in

2021, at a CAGR of 4.9 % over the forecast period. According to Arizona Advisory & Intelligence, 1.2 million tonnes of lithium lubricants are expected to be sold in 2027 [5].

Lithium lubricants have found a multifunctional application and are used to lubricate rolling and plain bearings, joints, and other friction units of machines and mechanisms. Until now, general-purpose lubricant CIATIM-201, made with petroleum oil thickened with lithium stearic acid soap and an antioxidant additive, has been widely used in Ukraine for these purposes. The lubricant is characterized by its performance at low temperatures, but does not meet current EU requirements due to its low mechanical and colloidal stability, insufficient dripping point and upper temperature limit, and a tendency to seal during long-term storage. These negative properties are associated with the stearic acid soap thickener.

However, 12-hydroxystearic acid is mainly used as a thickener for the highest-quality lithium lubricants. In Ukraine, a wide range of lubricants, such as Phiol-1, Phiol-2, Lithol-24, etc. are industrially produced using this thickener. These lubricants are characterized by high thermochemical, colloidal, and mechanical stability, low water washout, water resistance, and a wide temperature range. Lithium soaps are formed due to the reaction of 12-hydroxystearic acid based on vegetable oil (castor oil) with hydroxides of alkali or alkaline earth metals (lithium hydroxide). But 12-hydroxystearic acid is imported from India, Brazil or China.

All this encourages scientists and lubricant manufacturers to search for effective alternatives to raw materials. The use of vegetable oils or by-products from oil and fat production as components of high-temperature lubricant compositions is limited due to low thermal oxidation stability [2, 6], which is due to the presence of triglyceride groups and double bonds in the acyl residues of unsaturated higher fatty acids. Therefore, the use of these products in high-temperature lubricants requires additional chemical modification.

It should be noted that most modern oleochemical technologies are based on refined oils, the use of which for technical needs is problematic due to food shortages. In contrast to the generally accepted approach of using refined oils (rapeseed, sunflower, corn, *etc.*), it has been shown in [6-7] that the use of oil mill wastes and secondary fatty resources constitute the largest group of higher fatty acids for the synthesis of surfactants, emulsifiers, additives, etc. with the subsequent development of lubricants based on them.

We propose waste food oils (WFO) as a potential raw material. These studies will allow, on the one hand, to replace imported components for the production of lubricant thickener, on the other hand, to solve the problem of utilization of by-products of oil and fat production, and taking into account the economic factor: the cost of used edible oil is 2-3 times less than fresh oil [8-10]. WFO is produced geographically everywhere, mainly in households and hotel and restaurant businesses, and its current global production is estimated at 20 % to 32 % of total oil consumption (41-67 million tonnes per year) [11]. WFO are considered to be waste that is hazardous to the environment [12]. As the world’s population grows, the volume of these by-products will only increase.

During the frying of foods, oxidation, hydrolysis, isomerization, and polymerization of oils occur. WFO are mainly composed of triglycerides, monoglycerides, diglycerides, and free fatty acids (5-20 % by weight). Therefore, WFO can be considered a source for the production of environmentally friendly emulsifiers-stabilizers for lubricants [13-16].

Thus, this work aims to synthesize an emulsifier-stabilizer as a thickener for a lubricant composition and to develop a multipurpose lithium lubricant for friction units of machines and mechanisms that would combine high antioxidant and volumetric-mechanical properties with improved protective and tribological characteristics.

Experiment

Materials

Waste food oils (WFO), a sample provided by a local catering establishment, were used as an alternative source of secondary fatty acids. 99.5 % potassium iodide, 35 % hydrogen peroxide, 99.8 % sodium hydroxide, 99 % lithium hydroxide, 13 % hydrochloric acid, 12-hydroxystearic acid, 99.8 %, 99.5 % diethanolamine, 99.8 % formic acid were purchased from Chemlaborreactiv (Ukraine).

The main components of the fatty acid composition of the WFO sample were as follows, wt. %: palmitic – 6.5; stearic – 3.4; oleic – 32.6; linoleic – 47.5; linolenic – 5.3; the remaining 3.4 accounted for other fatty acids; water content – 1.3 wt. %. Kinematic viscosity at 40 °C is 4.7 mm^2/s; density is 0.93 g/cm^3; acid number is 3.2 mg KOH/g; iodine number is 83 g I_2/100 g; saponification number is 184.2 KOH/g, melting point is 211 °C. The food products remaining in the WFO were removed by filtration.

Testing methods

The IR spectra of products were recorded on the surface of the diamond prism of the IR-spectrometer with Fourier transform Shimadzu IRAffinity-1Sn (Japan) with ATR-console Speacac GS 10801-B. The structure of the synthesised substances was determined by NMR spectroscopy. One-dimensional (^{1}H, ^{13}C) NMR spectra were recorded on a Bruker AVANCE DRX-500 using DMSO-d6. Differential thermal analysis (DTA) and thermal gravimetry (TG) were performed employing Derivatograph Q-1500D in a temperature range 20-800 °C and heating rate 10 °C min^{-1}.

The volumetric and mechanical characteristics of the synthesized lubricants were evaluated by penetration, dropping point and effective viscosity. The number of penetration was determined according to ISO 2137, which is expressed by the depth of immersion in a mixed 60 double stroke lubricant of a cone weighing 150 g for 5 s at a temperature 25 °C. This indicator characterizes the consistency of the lubricant and is the basis of the NLGI classification. The dropping point was set according to ISO 2176, it is the maximum temperature at which a drop of lubricant falls from the cup of the Ubbelode thermometer, which was heated at a rate 1.0-1.5 °C per minute. Since lubricants are multicomponent systems that consist of components with different melting points, this method conditionally characterizes the melting point of lubricants. The dropping point characterizes the upper temperature limit for the use of lubricants. At dropping point of the lubricant of up to 150 °C, the maximum working temperature is 25 °C lower, at dropping point from 150 °C to 205 °C, working temperature is 40 °C lower, and at dropping point above 205 °C, the working temperature will be 70°°C lower [17].

Resistance to oxidation of lubricants was analyzed by the change of acid number after treatment at 150 °C for 10 h on a copper plate according to GOST 5734. The viscosity of the lubricant samples was determined by GOST 7163 using an automatic capillary viscometer AKV-2 at a temperature of 0 °C and an average strain rate gradient of 10 s^{-1}.

Anti-corrosionproperties of lubricants were evaluated according to ASTM D 4048. The essence of the method consists in keeping a copper strip in the samples of lubricants for 24 hours at a temperature 100 °C and visual comparisonof the change of the strip color with corrosion standards.

Tribological characteristics of lubricants were determined on a four-ball friction machine at a constant temperature, with a rotation frequency of 1460±70 min^{-1} for 10 s and a stepwise increase in the load according to the critical load (Pc) indicators according to GOST 9490.

General methods

The synthesis of hydroxyacids of WFO was carried out in two stages. In the first stage, the mild oxidation of the double bonds of WFO with hydrogen peroxide (35 wt. %) was carried out with the participation of formic acid (99.8 wt. %). For this purpose, in a round-bottomed flask equipped with a thermometer, stirrer, and reflux condenser, we mixed WFO (1 mol) and formic acid (5 mol) and cooled the mixture to a temperature of 5-10 °C. Under these conditions, a hydrogen peroxide solution (2 mol) was gradually (1–2 drops/min) added to the reaction mixture, the temperature was raised to 50-55 °C, and the system was kept under constant stirring for 2.5 h. After that, the aqueous phase was poured off, and the upper organic layer was washed successively with aqueous solutions of sodium bicarbonate $NaHCO_3$ (5 wt. %) and sodium chloride NaCl (10 wt. %). The separated epoxidized WFO was subjected to alkaline hydrolysis with an aqueous solution of sodium hydroxide NaOH (3M) at boiling for 2 h. At the last stage, an equimolar amount of hydrochloric acid was added to the obtained concentrated solution of salts of hydroxylated fatty acids, and the resulting product was separated by filtration. The resulting white precipitate was washed with water and dried in a vacuum drying oven to a constant weight to obtain the finished hydroxy acids of the used edible oil. Fatty acid aminoamides of oil were synthesized by epoxidation of oils and transamidation of acylglycerols with diethanolamine [6].

Results and Discussion

The use of WFO as a component of lubricant compositions is limited due to their low thermal oxidation stability. This is due to the presence of triglyceride groups and double bonds in the acyl residues of unsaturated higher fatty acids. To eliminate these active centers, chemical modification of WFO was carried out to obtain an emulsifier-stabilizer as a thickener for lubricants – saponified hydroxylated fatty acids of waste food oils (WFO_{-OH}) and an additive for lubricants – fatty acid aminoamide (WFO_{-AA}).

At the first stage of this work, WFO hydroxyacids were synthesized by epoxidation of unsaturated fatty acids (oleic, linoleic, linolenic, erucic) or their mixture, which is part of used food oils, followed by hydrolysis by acylglycerol groups and oxirane rings. Fatty acid aminoamides of waste food oils as an antioxidant additive to lubricants were synthesized by epoxidation of oils followed by the opening of the oxirane cycle and transamidation of acylglycerols with diethanolamine. The general scheme of the reaction is shown in Fig. 1. In an alkaline environment, the epoxy ring opens with the addition of a hydroxyl to the hydrocarbon chain, followed by the stabilisation of the hydroxyl group. At the same time, a reaction involving the carbonyl of the ester group takes place to form salts of hydroxylated fatty acids. The neutralization of sodium hydroxide was carried out with a hydrochloric acid solution. Simultaneously with the neutralization of sodium hydroxide in an acidic environment, the ester groups of hydroxylated WFO acylglycerides are hydrolyzed. The opening of epoxy rings and the formation of free hydroxylated fatty acids were carried out in one reaction volume. Excess hydrochloric acid was washed off with H_2O to a neutral medium.

WFO hydroxyacids are a white crystalline powder with a saponification number of 182.7 mg KOH/g, a melting point of 74 °C, an iodine number of 0.9 g I_2/100 g, and an acid number of 178.1 mg KOH/g. It is well miscible with almost all traditional petroleum and synthetic base oils.

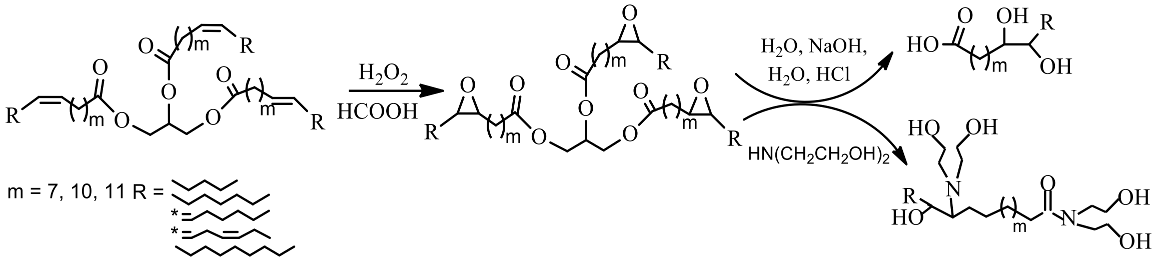


Fig. 1. Preparation of WFO hydroxyacids and WFO fatty acid aminoamide

WFO fatty acid aminoamides are brown lubricant-like substances with an acid number not exceeding 22-25 mg KOH/g and a flow point of up to 30 °C. They are highly soluble in petroleum oils, aliphatic and aromatic hydrocarbons, and mixtures.

IR-spectroscopy

The presence of -OH groups in the structure of WFO$_{\text{-OH}}$ was revealed by the results of IR spectra. The reaction of carbonyl fragments confirms the disappearance of bands 1745 cm^{-1} and 1160 cm^{-1}, C=O groups of esters (Fig. 2, curve 1) and the appearance of bands in the region of 1700 cm^{-1}, 1420 cm^{-1} and 1290 cm^{-1} (Fig. 2, curve 2), corresponding to the formed acid groups.

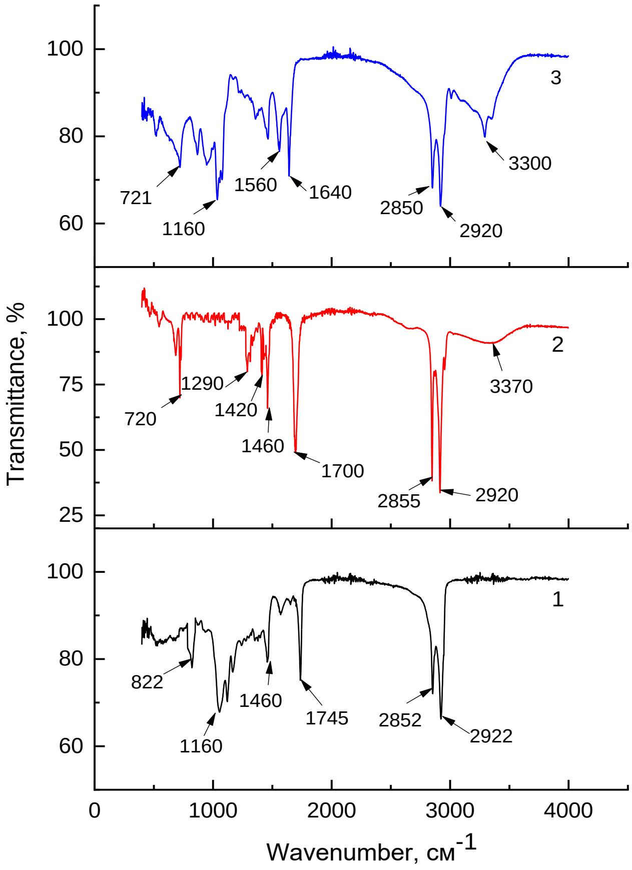


Fig. 2. IR spectra: 1 – curve WFO, 2 – curve WFO$_{\text{-OH}}$, 3 – curve WFO$_{\text{-AA}}$

In addition, the obtained hydroxylated products are confirmed by the disappearance of the epoxide peak at 822 cm^{-1} (Fig. 2, curve 1) and the appearance of a broad, flat band in the range 3250-3550 cm^{-1} with a maximum of 3370 cm^{-1} (Fig. 2, curve 2), corresponding to -OH groups formed as a result of the opening of the epoxy ring. On the IR spectra, the bands at 2920 cm^{-1}, 2855 cm^{-1} correspond to the valence vibrations of the -CH$_2$, -CH$_3$ groups, and the bands at 1460 cm^{-1} and 720 cm^{-1} to the strain vibrations of -CH$_2$- (Fig. 2, curve 2).

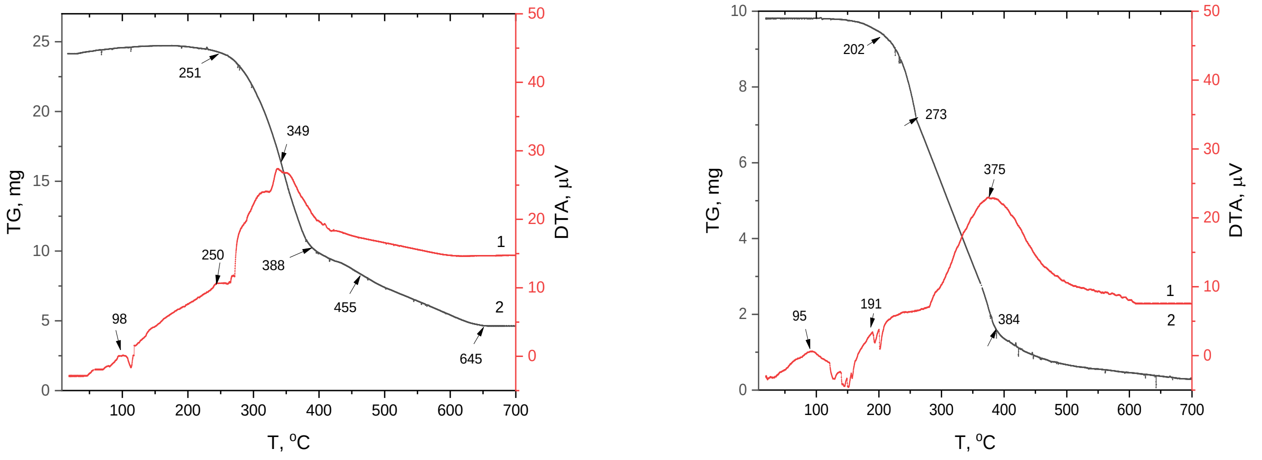
In structure of the WFO$_{\text{-AA}}$ (Fig. 2, curve 3), there appears the band at 3300 cm^{-1} of valence oscillations of N-H group, besides, bands of 1745 and 1160 cm^{-1}, C=O groups of esters disappear during amidation with the occurrence of corresponding 1640 cm^{-1} – valence and 1560 cm^{-1} and 1055 cm^{-1} – deformation oscillations of the groups of formed amides.

NMR spectroscopy

The NMR spectra of WFO$_{\text{-OH}}$ were interpreted as follows: ^{1}H NMR (DMSO-d6, 400 MHz): δ = 11.94 (extended singlet, 1H, COOH), 4.18 (extended singlet, 1H, OH), 2.17 (triplet, 2H, CH$_2$), 1.47 (multiplet, 2H, CH$_2$), 1.40 – 1.10 (multiplet, 28H, CH$_2$), 0.85 (triplet, 3H, CH$_3$). ^{13}C NMR (DMSO-d6, 100 MHz): δ = 174.33; 37.21; 33.63; 31.38; 29.26; 29.11; 29.07; 29.02; 28.93; 28.74; 28.58; 25.26; 25.23; 24.48; 22.08; 13.87. According to the ^{1}H and ^{13}C NMR spectra, the sample of WFO$_{\text{-OH}}$ is most likely an individual compound with a small amount of impurities, not more than 5 % (signal in the region of 4.75 ppm).

Differential thermal and thermogravimetric analyses of synthesised products

DTA and TG analyses revealed the of the thermal stability of WFO$_{\text{-OH}}$ (Fig. 3 a). DTA curve fixed three exothermic transitions at 98 °C, 250 °C and 349 °C, which are accompanied by the loss of weight of WFO$_{\text{-OH}}$. Specifically, the weight loss started at 111 ºC (Fig. 3 a, curve TG) that can be considered a dehydration process, for example, through the removal of water from product. The dehydration is accounted for 5.2 % of the total weight losses. Further mass losses occurred at 251 °C, 388 °C and 455 °C with full decomposition at 645 °C. DTA and TG analyses revealed the of the thermal stability of WFO$_{\text{-AA}}$ obtained from waste food oils (Fig. 3 a). DTA curve fixed exothermic transitions at 95 °C, 191 °C and 375 °C, which are accompanied by the loss of weight of WFO$_{\text{-AA}}$. The differential thermal analyses data are in good agreement with the results of the thermogravimetric analyses of WFO$_{\text{-AA}}$. The weight loss started at 109 °C (Fig. 3 b, curve TG), further mass losses occurred at 202 °C and 384 °C with full decomposition at 625 °C.



a b

Fig. 3. Derivatogram of WFO$_{\text{-OH}}$ (a): differential thermal analysis – curve 1 and thermogravimetric analysis – curve 2; derivatogram of WFO$_{\text{-AA}}$ (b): differential thermal analysis – curve 1 and thermogravimetric analysis – curve 2

Thus, according to the results of derivatographic analysis, the synthesised WFO$_{\text{-OH}}$ and WFO$_{\text{-AA}}$ is suitable for use in technical processes at temperatures up to 190-210 °C.

Production of lithium lubricant samples

In the second stage of this work, samples of lithium lubricant (LiWFO) based on API (American Petroleum Institute) group II oil with a viscosity of 22.0 mm^2/s at 100 °C with a pour point of -18 °C and a flash point of 260 °C were made.

The prototypes of lithium lubricant were produced according to the traditional technology for the production of lubricants containing lithium fatty acid soaps, which includes the following steps synthesis of the WFO$_{\text{-OH}}$ and WFO$_{\text{-AA}}$; saponification of the WFO$_{\text{-OH}}$ with lithium hydroxide solution in the base oil environment; dehydration of the resulting oil-soap mixture; thermomechanical structuring of the mixture with its subsequent cooling; introduction of the antioxidant additive (WFO$_{\text{-AA}}$); homogenization of the lubricant on a laboratory three-roll grinder. The introduction of the above components into the lubricant in a complex, as well as their specially selected quantitative composition, made it possible to obtain an optimal technical result, namely, to expand the fatty acid raw material base for the manufacture of high-quality multipurpose lithium lubricants and improve their protective and tribological characteristics.

By this technology, samples (1, 2, 3) of lithium lubricant LiWFO with the following content, % wt.: lithium soaps of hydroxyacid of oil – 24.0, aminoamide of fatty acids WFO – 3.0, petroleum oil – the rest (sample 1); lithium soaps of hydroxyacid of oil – 22.0, aminoamide of fatty acids WFO – 2.0, petroleum oil – the rest (sample 2); lithium soaps of hydroxyacid of oil – 20.0, aminoamide of fatty acids WFO – 1.5, petroleum oil – the rest (sample 3). Also, a lithium lubricant was prepared using 12-hydroxystearic acid (Li12oSt).

The table presents a comparative analysis of the quality indicators of the manufactured samples of lithium lubricants – Li12oSt and LiWFO.

Mechanical stability is one of the most important performance characteristics of lubricants, which shows a change in its consistency during operation as a result of loads. The samples of lithium lubricant were made of NLGI (National Lubricating Lubricant Institute) grade 3 consistency. The NLGI lubricant classification system is based on lubricant consistency, which is determined by the value of the «worked» penetration (with stirring by 60 double cycles). NLGI class 2 is the most common among industrially produced lubricants, and their working penetration value is in the range of 220 mm$\cdot 10^{-4}$ to 250 mm$\cdot 10^{-4}$. For the lubricant samples (1, 2, 3), this indicator meets these boundary conditions (Table).

According to the results of studies of the mechanical stability of samples (1, 2, 3) of LiWFO, the change in the penetration index after prolonged mechanical destruction (ΔP) decreased compared to Li12oSt sample (Table). This makes it possible to predict the long-term operation of the new lubricant in friction units without destruction and leakage. He criterion for mechanical stability is the change in the penetration index of the lubricant after prolonged mechanical destruction (P_2-$P_1 = \Delta P$). According to ISO 2137, the lubricants were subjected to destruction in a standard penetrometer mixer. P_1 was determined after 60, and P_2 after 100,000 double strokes. The change in the state of the lubricant (ΔP) characterizes the stability of its structural framework: for $\Delta P < 30$ – mechanical stability is excellent, for $\Delta P = 30 \div 60$ – good, for $\Delta P = 61 \div 100$ – satisfactory, for $\Delta P > 100$ – unsatisfactory [17]. For samples (1, 2, 3) of lithium lubricant, the mechanical stability is excellent.

Viscosity is the most important operational characteristic that determines the conditions for filling lubricant into friction units at low temperatures and affects the starting torque of bearings. The temperature at which the value of this indicator reaches 2000 Pa·s per 10 s^{-1} characterizes the lower temperature limit of the lubricant application. For samples (1, 2, 3) of LiWFO lithium lubricant, the viscosity at a temperature of 0 °C and strain rates 10 s^{-1} is 213 Pa·s, 203 Pa·s, 207 Pa·s, respectively (Table). Thus, the lower temperature limit of application will be at the level of 0 °C.

According to the results (Table), the obtained samples of lubricating compositions (1, 2, 3) of LiWFO are characterized by improved tribological characteristics (critical load – 735 N, 696 N, 696 N, respectively) and withstand corrosion effects on copper (Table).

Table. Comparative properties of lithium lubricants based on different fatty acids

Name of indicator, unit of measure	Values of the indicators				Testing method
	Li12oSt	LiWFO			
		Sample 1	Sample 2	Sample 3	
Dropping point, °C	198	207	204	200	ISO 2176
Penetration at 25 °C, m·10^{-4}: – after 60 double strokes (P_1) – after 100,000 doubles strokes (P_2) – mechanical stability, change (ΔP)	245 270 25	225 235 10	240 254 14	245 263 18	ISO 2137
Copper strip corrosion	1a	1a	1a	1a	ASTM D 4048
Tribological characteristics on a four-ball machine at (20±5) °C: – critical load (Pc), N	657	735	696	696	GOST 9490
Viscosity at 0 °C and strain-rates 10 s^{-1}, Pa·s	207	213	207	200	GOST 7163
Resistance to oxidation: increase in acid number (150 °C, 10 hours), mg KOH/g	0.26	0.20	0.22	0.24	GOST 5734
Protective properties under dynamic conditions. Corrosion rate, mm·10^{-4}/year	2.6	1.8	1.9	2.3	«Dynacorotest»

The upper limit for the use of high-temperature lubricants is more than 150 °C, which often leads to intensification of oxidative transformations of the dispersion medium and, as a result, deterioration of the lubricant quality and reduction of the friction unit service life [14]. The experience of operating lubricants at high temperatures indicates the need to use them in their composition, in addition to a stable dispersion medium, and effective antioxidant additives. Hydrocarbon oxidation occurs by a radical mechanism, and its termination depends on the presence of agents that can interrupt chain reactions. This role can be played by amide nitrogen, which is capable of donating a proton and an unpaired pair of electrons, thus becoming a radical with significantly lower mobility and activity, thereby inhibiting such processes. A synthesized additive, fatty acid aminoamides (WFO_{-AA}), is used as an antioxidant in the lubricant.

The antioxidant properties of the lubricant samples were analyzed by GOST 5734 by changing the acid number before and after its oxidation. Since acidic substances are formed during the oxidation of lubricants, their amount can be used to assess the protective properties of an antioxidant additive. The best are the samples (1, 2, 3), in which the increase in the acid number is less (0.20 mg KOH/g, 0.22 mg KOH/g, 0.24 mg KOH/g, respectively) (Table).

As can be seen from the results of the study (Fig. 4), the highest antioxidant activity of fatty acid aminoamides as additives is observed in the concentration range of 1.8-2.0 %.

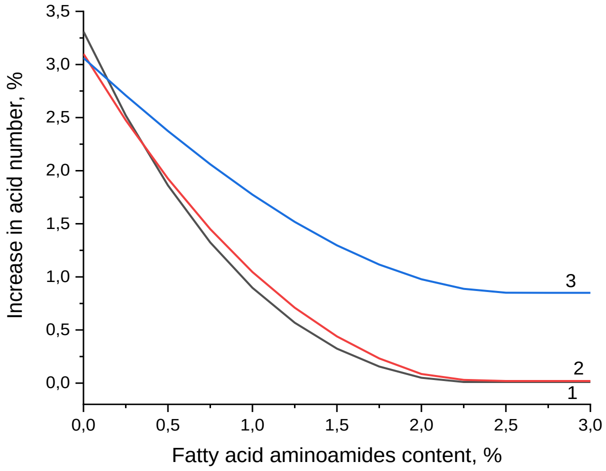


Fig. 4. Antioxidant properties of the samples lubricants with the use of derivatives:
1 - monoethanolamine, 2 - diethanolamine,
3 - ethylenediamine

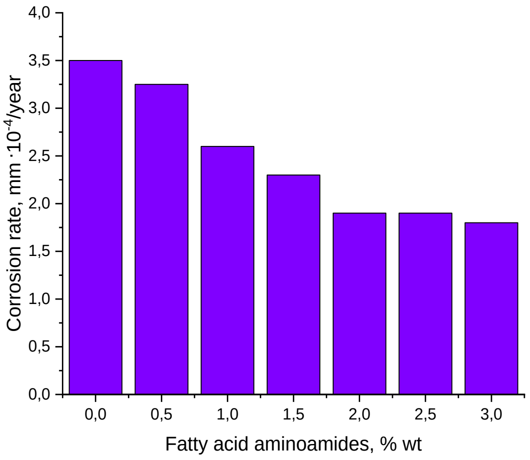


Fig. 5. Dependence of protective properties of of lubricants samples on the amount of fatty acid aminoamides

The protective properties of the resulting thixotropic systems were tested on a special “Dynacorotest” bench. The device allows the evaluate the protective properties of lubricants under dynamic conditions by the corrosion rate of rolling bearings. The corrosion rate of metal-bearing surfaces is calculated based on experimental values of the polarisation resistance, which is determined by a universal corrosion rate meter. The “Dynacorotest” device provides a mode for determining the corrosion rate at a bearing rotation speed of 500 min^{-1}. In contrast to the DIN 51802:2017 and IP 220:015 methods, which require an experiment duration of 164 hours, the Dynacorotest test duration does not exceed 3 hours. The corrosion rate is expressed in Å/cm^2, and for greater clarity is converted to mm/year. To test the protective properties, fatty acids aminoamide in the amount of 0.5-3.0 % by weight were added to the obtained LiWFO lubricant samples before the homogenization stage. The results are shown in Fig. 5 and Table.

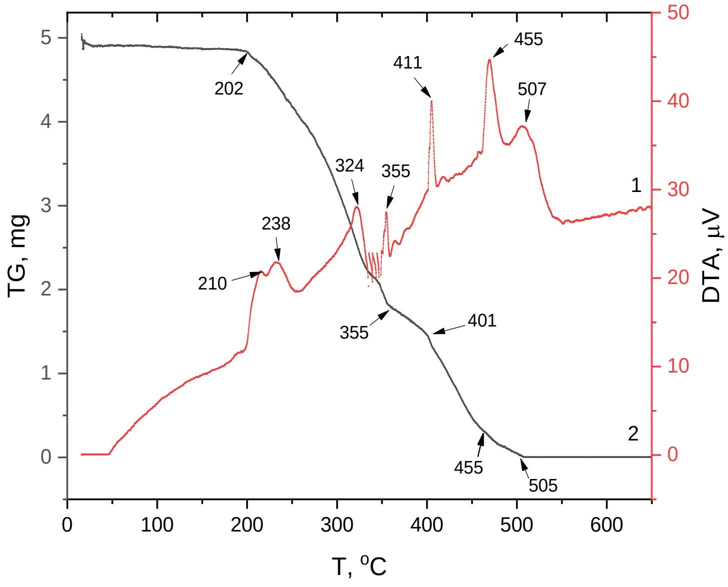


Fig. 6. Derivatogram of a lithium lubricant sample: 1 - curve of differential thermal analysis, 2 - thermogravimetry curve

As shown in Fig. 5, the corrosion rate of the developed thixotropic compositions decreases with an increase in the amount of fatty acid aminoamide in their composition. Still, when the concentration reaches 2.0 % by weight, this value practically does not change. Therefore, introducing fatty fatty acid aminoamide in larger quantities does not significantly change the antioxidant properties of the finished lubricant.

Differential thermal and thermogravimetric analyses of lubricant

The derivatographic analyses showed the degree of thermal stability of the lithium lubricant sample 1. The curve of differential thermal analysis (DTA) (Fig. 6, curve 1) shows exothermic peaks at 324 °C, 411 °C, 455 °C, and 507 °C, which characterize the thermal oxidation destruction of the dispersion medium and the dispersed phase of lithium lubricant. The thermogravimetric analysis (TGA) curve (Fig. 6, curve 2) shows that the lubricant sample remains thermally stable up to 195 °C, with almost no mass loss, which indicates high resistance of the lubricant composition to thermal transformations. When the temperature rises to 200-202 °C, intensive mass loss begins due to chemical decomposition and evaporation of the dispersion medium. Upon reaching a temperature of 512-550 °C, any energy effects disappear due to the complete thermal decomposition of the lubricant sample.

The dripping point at which the lubricant changes from a plastic solid to a liquid state is 207 °C, 204 °C, and 200 °C, respectively (Table). Thus, the upper-temperature limit for the developed lithium lubricant LiWFO according to derivatographic studies, dripping point, and calculations is 150 °C.

Summarising the results obtained, it can be stated that the objective has been achieved. The proposed lubricant composition LiWFO is superior in some respects to the prototype Li12oSt lubricant, and the optimal ratio of the new lubricant compositions ensures the manufacturability of production and their high technical and economic characteristics during operation.

Conclusion

The use of lithium soaps of hydroxoacids of waste food oil as a lubricant thickener and the introduction of fatty acid aminoamides of oils as an antioxidant additive into the composition of lubricants made it possible to obtain a plastic (lithium) lubricant characterized by improved protective and tribological characteristics, increased stability to oxidation and mechanical action and not inferior to lithium lubricant based on an industrial analog of 12-hydroxystearic acid.

The properties of the resulting lubricant make it possible to predict its long service life in components and mechanisms and the prospects for using the components used in lubricant formulations.

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Мастильні матеріали на основі синтезованого емульгатора-стабілізатора

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Синтезовано гідроксильовані жирні кислоти шляхом проведення епоксидування ненасичених жирних кислот відпрацьованої харчової олії з наступним гідролізом за ацилгліцериновим угрупуванням та оксирановими кільцями. Аміноаміди жирних кислот олій синтезували епоксидуванням відпрацьованої харчової олії з наступним розкриттям оксиранового циклу і трансамідуванням ацилгліцеринів діетаноламіном. Застосування літієвих мил гідроксокислот відпрацьованої харчової олії як емульгатора-стабілізатора, який виконує функцію загусника мастил та введення до складу мастил аміноаміду жирних кислот олій як антиокиснювального додатка дозволило одержати пластичне (літієве) мастило. Досліджено фізико-хімічні властивості мастила та проведено порівняльний аналіз їх показників якості з мастилом на основі 12-гідроксистеаринової кислоти. Розроблене літієве мастило характеризується покращеними захисними та трибологічними характеристиками, підвищеною стабільністю до окиснення та механічної дії, не викликає корозію кольорових металів й не поступається літієвому мастилу на основі промислового аналога 12-гідроксистеаринової кислоти. Пластичне мастило призначене для вузлів тертя машин і механізмів. Властивості одержаного мастила дають змогу прогнозувати тривалий строк його експлуатації в вузлах і механізмах та перспективність застосування використаних компонентів в рецептурах мастильних композицій. Дані дослідження дозволяють з одного боку замінити імпортовані компоненти для виробництва загусника мастил, а з іншого – розв’язати проблему утилізації побічних продуктів олієжирового виробництва.

Ключові слова: вторинна жирова сировина, емульгатор-стабілізатор, антиокиснювальний додаток, літієве мастило