

WASTE FOOD OILS AS COMPONENTS OF ECO-FRIENDLY GREASE

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Abstract. The possibility of obtaining eco-safety surfactants and high-temperature dispersed phase of thixotropic plastic systems based on waste food oils were demonstrated. The structure of the synthesized fatty acid alkanolamides was confirmed by IR and ¹H NMR spectroscopy. The upper temperature limit (above 463 K) for the use of synthesized fatty acids alkanolamides and complex calcium grease was determined by thermogravimetric analysis. The introduction of fatty acid alkanolamides to the high-temperature composition of grease improves its tribological, anti-oxidant and environmental characteristics.

Keywords: waste food oils, fatty acids alkanolamides, high-temperature grease

1. Introduction

Modern technical progress has led to a constant increase in the number and variety of machines and mechanisms, which inevitably leads to an increase in consumption and an expansion of the range of lubricating materials and surfactants for their effective operation and maintenance. At the same time, these are mainly petrochemical products with low biodegradability, which are one of the sources of deterioration of the ecological state of the natural environment. The world lubricating materials market in 2017 was estimated at around 35.7 Mt.¹ Lubricating materials are characterized by a certain service life, after which they need to be replaced. During the operation of lubricating materials there are inevitable losses. Only about 50 %^{2,3} of the total amount of fresh lubricating materials are collected, therefore, a large amount of used lubricating materials are accumulated every year.

Therefore, the solution to this ecological problem is the expansion of the use of environmentally safe lubricat-

ing materials. Biodegradable lubricating materials and surfactants are especially necessary to be used in industries where there is a probability of their getting into the environment, in particular, in water, agricultural and forestry machinery, technological systems for oil and gas production.⁴ The vast majority of these eco-friendly products are made on the basis of oils and fats raw materials, in particular vegetable oils, animal fats, *etc.*

Although oilseed crops are renewable raw materials, the production of bioproducts from vegetable oils on an industrial scale is irrational. This is due to the high cost of raw materials, deforestation, need for large areas of arable land and direct competition with food crops for land use. These problems prompt the need to find alternative resources that would not cause controversy regarding food security.

Currently, the cost of waste food oil (WFO) is 2-3 times less than fresh vegetable oil,⁵⁻⁷ therefore it is considered as a promising source for oleochemical products production. WFOs are geographically generated everywhere, mainly in households, hotel and catering sector, and their current global production is estimated at 20–32 % of the total vegetable oils consumption (41–67 Mt/y).⁸ WFOs are considered dangerous waste for the environment.⁹ As the world's population grows, so the volume of these by-products will increase.

Oxidation, hydrolysis, isomerization and polymerization of vegetable oils occur during frying. WFOs are mainly composed of triglycerides, monoglycerides, diglycerides, and free fatty acids (5–20 wt. %). Therefore, WFOs can be considered as raw materials for the production of environmentally friendly lubricating materials and surfactants. However, the vast majority of researches are focused on the use of WFOs for the production of biodiesel.¹⁰⁻¹⁴

This paper proposes chemical modifications of WFO to produce environmentally friendly surfactants and high-temperature grease thickeners.

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2. Experimental

2.1. Materials and Reagents

WFO consists of 16 % phospholipids, 78 % tri-, di- and monoacylglycerides, 4 % free fatty acid, with 1 % residual moisture content, and 1 % ester non-soluble substances. Fat and acid composition of the WFO was determined chromatographically in combination with chemical functional analysis of acid and ester numbers made up, % by weight: palmitic $C_{16:0}$ – 2.4, stearic $C_{18:0}$ – 2.9, oleic $C_{18:1}$ – 23.8, linoleic $C_{18:2}$ – 54.0, linolenic $C_{18:3}$ – 13.5, others – 3.4. Kinematic viscosity at 298 K – 114 mm²/s, acid value – 3.2 mg KOH/g, iodine number – 81 g I₂/100 g, saponification value – 205 mg KOH/g.

IR spectra were recorded on the IR spectrometer Shimadzu IRAffinity-1Sn with ATR-console Specac GS 10801-B. ¹H NMR-spectra were recorded in dimethylsulfoxide (DMSO-*d*₆) with internal standard tetramethylsilane on a Bruker AVANCE DRX-500. Colloid-chemical studies were performed by well-known methods, in particular the surface tension of surfactant solutions at the interface with air, the method of separation of the plate (Wilhelmi method). Differential thermal analysis (DTA), differential thermogravimetry (DTG), and thermal gravimetry (TG) were performed employing derivatograph Q-1500D in a temperature range of 293–1273 K and heating rate of 10 K·min⁻¹.

2.2. Synthesis of Biobased Surfactants

The synthesis of biobased surface-active agent (bioSAA) was conducted employing base-catalyzed amidation of WFO by amines of different basicity. The syntheses were conducted by reactions of known (mol) amounts of WFO and alkanolamines under the action of calcium hydroxide catalyst (1:3:0.4). The reaction systems were heated under constant agitation at 353–373 K for 0.5 h and at 393–398 K for 1.5 h. Reaction progress was monitored by a potentiometric titration of amines, and by IR analyses of the reaction media during the course of the processes. After syntheses, the unreacted amines were removed at elevated temperature (333–353 K) under reduced pressure (5–10 mbar) in the nitrogen flow. Calcium hydroxide forms complexes with phosphatides; therefore, any recovery of the catalyst or further purification wasn't considered.

Surface active fatty acids alkanolamides (FAAA) produced from WFO are lubricant-like substances of brown color with a melting point of 325–336 K with improved solubility in organic non-polar solvents.

2.3. Sample Preparation of Biobased High-Temperature Greases

The components of the dispersed phase of the environmentally safe high-temperature lubricant are selected from the Lubricant Substance Classification list (LuSC list),¹⁵ such as calcium fatty acid soaps and calcium acetate as complexing agent.

Possible dispersion media are biodegradable and non-toxic base oils, such as vegetable oils, synthetic ethers, polyalphaolefins, and glycols.¹⁶ The vegetable oil cannot be used for high-temperature greases due to their low resistance to oxidation. Readily biodegradable polyalphaolefins are characterized by low viscosity, which also makes it impossible to use them for the production of high-temperature greases.¹⁶ Dioctyl sebacate with a viscosity of 3.8 mm²/s at 373 K, a pour point of 208 K and an open cup flash point of 488 K was used as base oil.

The upper limit of the high-temperature greases use is more than 423 K, which often leads to the intensification of oxidizing transformations of the dispersion medium and, as a result, the quality of the grease deteriorates and the service life of the friction unit decreases. The experience of operating greases in conditions of high temperatures indicates the need to use effective antioxidant additives in their composition, in addition to a stable dispersion medium. Oxidation of hydrocarbons occurs by a radical mechanism; its termination depends on the presence of agents capable of terminating chain reactions. This role can be played by amide nitrogen, which is capable of donating a proton and an unshared pair of electrons, thus becoming a radical one with much lower mobility and activity, thereby inhibiting similar processes. Synthesized bioSAA fatty acids alkanolamides of WFO were used as an antioxidant in the composition of high-temperature grease.

The produced samples of eco-friendly complex calcium greases had NLGI (National Lubricating Grease Institute) grade 2 consistency. The NLGI greases classification system is based on their consistency, which is determined by the magnitude of «worked» penetration (with stirring by 60 double cycles). NLGI grade 2 is the most widespread among industrially manufactured greases and the value of their worked penetration is in the range of 265 to 295 mm·10⁻¹.

The mass fraction of the dispersed phase was 18 % at a molar ratio of WFO and acetic acid equal to 1:6. The complex soap was synthesized in 70 % of the total amount of dioctyl sebacate by saponification of WFO fatty acids and neutralization of the complexing acid with an aqueous solution of calcium hydroxide at the temperatures of 368–373 K. Then dehydration to 413–433 K was carried out followed by heat treatment at 498–503 K for 15 minutes.

After that, the remaining dioctyl sebacate was added; the grease was cooled to room temperature and homogenized using a laboratory three-roll mill. A sample of base complex calcium (CaX) grease was obtained, the dispersed phase of which was formed by calcium soaps of WFO fatty acids and calcium acetate. To test the antioxidant properties of FAAA of WFO, a sample of CaX grease was prepared according to the above technology with the following difference, namely: 2 wt.% of bioSAA was introduced at the cooling stage at the temperatures of 353–343 K.

2.4. Testing Methods

The volumetric and mechanical characteristics of the synthesized greases were evaluated by penetration, dropping point and effective viscosity. Penetration was determined according to ISO 2137, which is expressed by the depth of immersion in a mixed 60 double stroke grease of a cone weighing 150 g for 5 s at a temperature of 298 K. This indicator characterizes the consistency of the grease, which is the basis for the NLGI classification. The dropping point was set according to ISO 2176, it is the maximum temperature at which a drop of grease falls from the cup of the Ubbelohde thermometer, which was heated at a rate of 1.0–1.5 K per minute. Since greases are multicomponent systems that consist of components with different melting points, this method conditionally characterizes the melting point of greases. The dropping point characterizes the upper temperature limit for the greases use. At the dropping point of the grease of up to 423 K, the maximum working temperature is 25 K lower, at the dropping point from 423 to 478 K, working temperature is 40 K lower, and at the dropping point above 478 K, the working temperature will be 70 K lower.¹⁷ The effective viscosity of greases was determined using an automatic capillary viscometer AKV-2 at a temperature of 243 K and strain-rates of 10 s^{-1} .¹⁸ This indicator determines the resistance of greases at negative temperatures when they are pumped to friction nodes, starting bearings. The effective viscosity at the lower temperature limit of the grease use should not exceed $2000 \text{ Pa}\cdot\text{s}$ per 10 s^{-1} .

The stability of the greases samples was characterized by a colloidal stability, resistance to oxidation and water resistance. Colloidal stability was determined by the amount of oil, which was pressed out of the grease on the KSA apparatus for a load of $1000 \pm 10 \text{ g}$.¹⁸ Resistance to oxidation of greases was analyzed by the change in acid number after treatment at 423 K for 10 hours on a copper plate.¹⁸ Water resistance was determined according to ASTM D 1264 – the ability of the grease to remain in the bearing under the influence of a water jet heated to 352 K for 1 h and expressed as the amount of washed off grease in percentage.

Anti-corrosion properties of greases were evaluated according to ASTM D 4048. The essence of the method consists in keeping a copper strip in the samples of greases for 24 hours at a temperature of 373 K and visual comparison of the strip color change with corrosion standards.

Tribological characteristics of greases were determined on a four-ball friction machine at a constant temperature, with a rotation frequency of $1450\text{--}1500 \text{ min}^{-1}$ for 10 s and a stepwise increase in the load according to the critical load (P_c) and welding loading (P_w) indicators according to ISO 20623.

Environmental characteristics were assessed by the indicator biodegradation according to CECL 33-A-93. The essence of the method is to compare IR spectra of greases, aged in a mineralized aqueous solution with an inoculant for 21 days in aerobic conditions, and to compare the degree of biodegradation relative to control material RL 130 (diisotridecyl adipate).

3. Results and Discussion

Phospholipids are some of the best known natural self-assembly surfactants. They are distributed in nature and are the main structural components of the lipid matrix of biological membranes. Research has accumulated extensive material on their structural organization in synthesis environments. These are monomolecular layers, bimolecular films, vesicles, liquid crystals, organogels, emulsions, and microemulsions. The main factors of self-assembly surfactants are the presence of two parts of different polarity: the non-polar component is represented by hydrocarbon chains and polar components are the remnants of glycerol, acid of phosphoric, choline or colamine.

The minimum surface tension of bioSAA and waste food oils is respectively 35–36 and 42–43 mN/m at $T = 303 \text{ K}$. As the temperature increases, the surface tension of bioSAA decreases to 33–34 mN/m at $T = 333 \text{ K}$, because the solubility of the phases increases, thermal motion increases, the intermolecular distance increases and the forces of attraction of phosphatide molecules decrease.

The results of ^1H NMR analysis are consistent with the IR spectroscopy data. The formation of bioSAA was proven by ^1H NMR spectrum (Fig. 1).

^1H NMR (500 MHz, CDCl_3/TMS , ppm): 6.01 (wide band, N-H), 5.33 (m, HC=CH), 4.21(q, $\text{CH}_2\text{-N}$), 3.81 (t, $\text{CH}_2\text{-O}$), 3.72-3.43 (q, $\text{CH}_2\text{-N}$), 2.76 (t, =CHCH₂CH=), 2.20 (t, $-\text{CH}_2\text{CH}_2\text{C=O}$), 2.04 (m, =CHCH₂-), 1.62 (m, $-\text{CH}_2\text{CH}_2\text{C=O}$), 1.51-1.26 (m, $-\text{CH}_2\text{CH}_2\text{CH}_2-$ and $-\text{CH}_2\text{CH}_3$), $\delta = 0.88$ (m, CH_2CH_3).

The IR analysis identified the formation of bioSAA as well (Fig. 2).

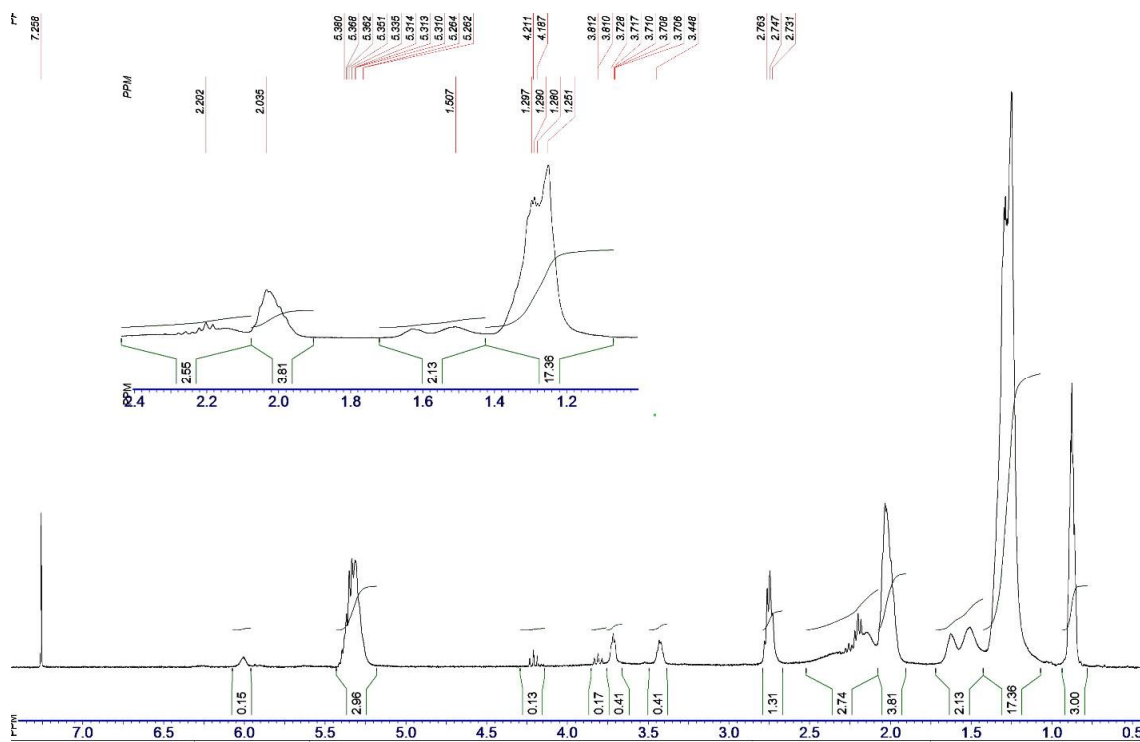


Fig. 1. NMR ^1H spectrum of alkanolamides

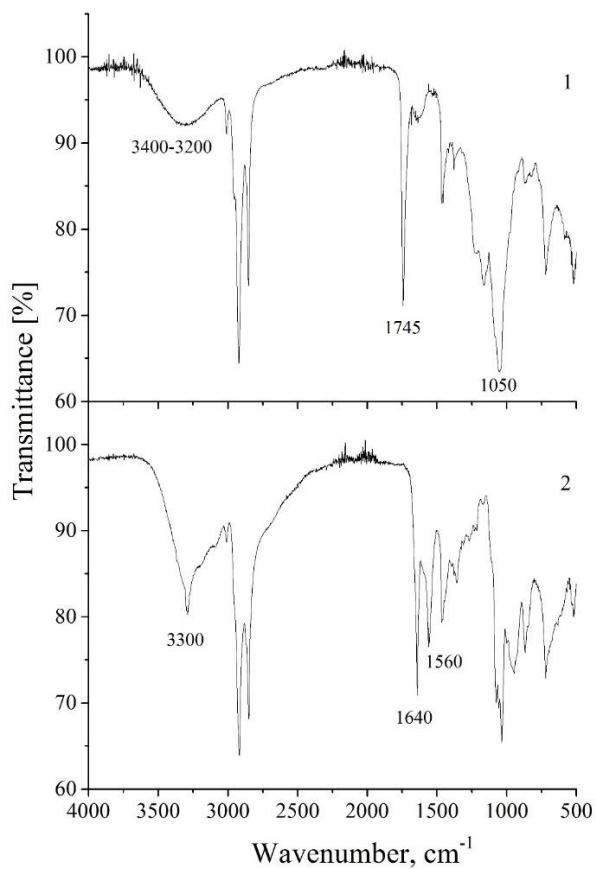


Fig. 2. Infrared spectra: 1 – waste food oils, 2 – alkanolamides

In IR spectra of the products, new characteristic bands at 3300 cm^{-1} (N–H stretching vibrations), 1640 cm^{-1} (C=O amide stretching vibrations), and 1560 cm^{-1} (N–H amide bending vibrations) appeared after reactions of WFO and alkanolamides. At the same time, the characteristic vibrations of ester bonds at 1745 cm^{-1} (C=O ester stretching vibrations) disappeared after syntheses, additionally providing evidence in favour of the generation of bioFAA. Changes have also been noted at $3400\text{--}3200\text{ cm}^{-1}$ (O–H stretching vibrations) and 1050 cm^{-1} (P–O stretching vibrations), they are likely associated with the interaction of phosphatides and calcium hydroxide.¹⁹

DTA and DTG analyses revealed the thermal stability of bioSAA obtained from waste food oils (Fig. 3a, 3b).

DTA curve fixed three exothermic transitions at 339, 464 and 502 K, which are accompanied by the loss of weight of bioSAA. Specifically, the weight loss started at 377 K that can be considered a dehydration process, for example, through the removal of water from the surface-active amide groups. The dehydration is accounted for 7 % of the total weight losses. Further mass losses occurred at 492, 639, and 693 K (93 % of total weight losses) and are likely attributed to the thermal decomposition of the product in several steps, which mechanisms are not yet clear. From these results, it is deemed that synthesized amides can be exploited in technical processes at the temperatures up to

463 K; such high temperatures are an upper reasonable borderline in the surfactant applications. A lubricating material is considered to be a high-temperature one if at least one of the requirements is met: its dropping point is above 503 K or the temperature limit of application is 423 K and more.²⁰ Thus, according to the results of derivatographic analysis, the synthesized bioSAA is suitable for use in high-temperature lubricating compositions.

Fig. 4 shows the derivatographic analysis of the sample of base CaX grease. On the DTA curve (Fig. 4a), starting from a temperature of 615 K, several exothermic effects are observed, which characterize the thermo-oxidative destruction of the dispersion medium and the dispersed phase. Upon reaching a temperature of 1228 K, any energy effects disappear due to the complete thermal decomposition of the sample. On the TG curves (Fig. 4b), the initial horizontal section up to 539 K indicates the high stability of the lubricating composition to thermal transformations. When the temperature rises to 573 K, the sharp weight loss of the grease is observed due to the intensive chemical decomposition and evaporation of its synthetic dispersion medium. The weight loss of the grease sample was 97.7 %. The obtained results make it possible to determine the upper temperature limit of using the synthesized grease at the level of 523 K.

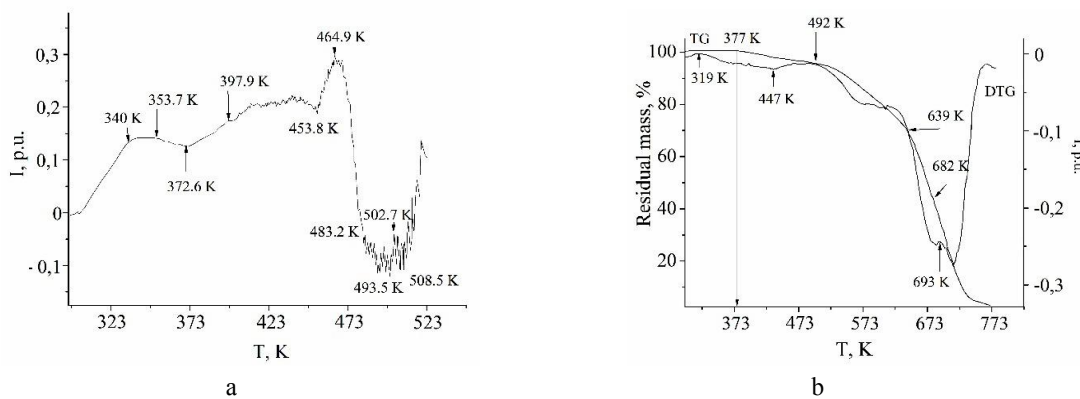


Fig. 3. DTA (a) and TG, DTG (b) curves of bioSAA

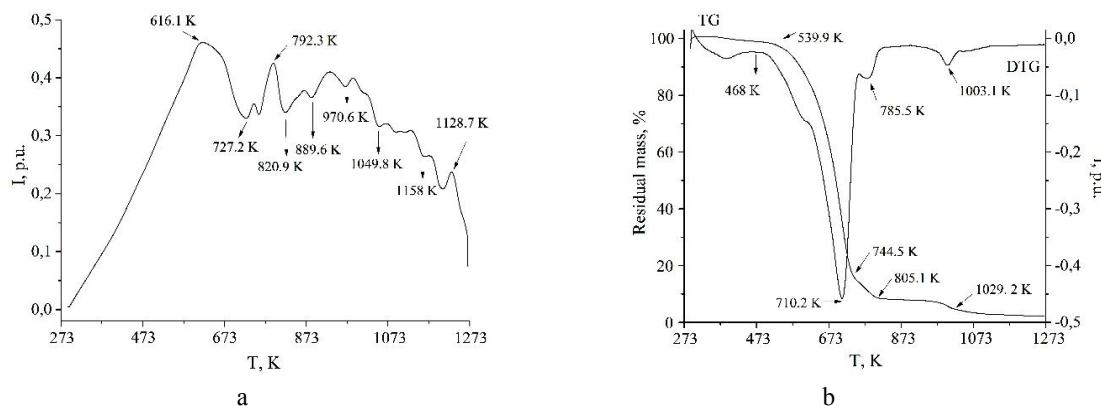


Fig. 4. DTA (a) and TG, DTG (b) curves of biogrease

Table. Physicochemical properties of developed complex calcium greases based on waste food oils

Name of indicator, unit of measure	Values of indicators	
	Base CaX grease	CaX grease with bioSAA
Penetration after 60 double strokes at 298 K, mm·10 ⁻¹	283	279
Dropping point, K	> 523	> 523
Effective viscosity at 243 K and strain-rates 10 s ⁻¹ , Pa·s	1039	1039
Colloidal stability, % of extracted oil	3.7	3.6
Resistance to oxidation: increase in acid number (423 K, 10 hours), mg KOH/g	7.5	1.8
Water washout at temperature 352 K, %	2.5	2.6
Copper strip corrosion	1a	1a
Tribological characteristics on four ball machine at (293±5) K:		
– critical load (Pc), N	1039	1039
– welding loading (Pw), N	2764	3087
Biodegradation, %	91	94

The results of laboratory and bench tests of developed CaX greases based on waste food oils are presented in Table.

As shown in Table, synthesized CaX greases are characterized by a high dropping point (above 523 K), the lack of corrosion activity and water-wash resistance. The calcium glycerolphosphatides present in these greases improve anti-wear properties by the critical load (Pc) and anti-seizure characteristics by the welding load (Pw) compared to basic commercial CaX greases based on the dispersed phase, which form calcium soaps of stearic and acetic acids.²¹

However, the presence of double bonds in unsaturated higher fatty acids in the structural frame worsens the thermo-oxidative stability of the base CaX grease. The use of synthesized bioSAA made it possible to increase the resistance of the thixotropic system to oxidative transformations. A long-term test under the conditions of thermo-oxidizing factors (high temperature, copper catalyst and oxygen atmosphere) led to the inhibition of acid number growth. That is, FAAA interrupted the chain reaction at an early stage of peroxide radical generation.

The use of dioctyl sebacate as a dispersion medium of lubricating compositions made it possible to expand the temperature limits of their operation. Samples of greases do not solidify at 243 K, which is confirmed by the low effective viscosity at this temperature.

An environmentally safe lubricating composition was obtained due to the selected components, the indicator of its biodegradability is above 90 %. A product with a degradation value of more than 60 %, determined by CEC L-33 or OECD 301 methods, is considered biodegradable.²²

4. Conclusions

SAA and components of dispersed phases of lubricating compositions were synthesized based on waste food oils. Tests of these SAA prove that they are capable

of operating at temperatures up to 463 K and are effective multifunctional additives. The obtained complex calcium greases are characterized by the high colloidal stability. In addition, these thixotropic systems do not cause corrosion of non-ferrous metals and are able to operate in contact with water. Due to the use of bioSAA in the lubricating composition, resistance to oxidation increases. Derivatives of phosphoric acid in the composition of waste food oils form a strong structural framework of lubricants, which is characterized by an increased melting point – above 523 K and tribological properties with a welding loading above 2450 N, which in turn determines the capability of operating in highload friction points. Due to the use of a synthetic dispersion medium in the composition of lubricating compositions, the temperature limits of application are extended from below 243 K to 523 K, and the bio splitting ability is more than 90 %.

In addition, synthesized SAA combine with almost all mineral and synthetic oils and based on them compositions of lubricating materials²³ were developed.

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ЕКОБЕЗПЕЧНІ МАСТИЛА НА ОСНОВІ ВІДПРАЦЬОВАНИХ ХАРЧОВИХ ОЛІЙ

Анотація. Продемонстровано можливість одержання на основі відходів – відпрацьованих харчових олій екологічно безпечних поверхнево-активних речовин і високотемпературної дисперсної фази тиксотропних пластичних систем. ІЧ- та ¹H ЯМР-спектрометрією підтверджено будову синтезованих алканоламідів жирних кислот. Методом термогравіметричного аналізу визначено, що верхня температурна межа застосування синтезованих алканоламідів жирних кислот і комплексного кальцієвого мастила перевищує 463 К. Встановлено, що введення до складу високотемпературного мастила алканоламідів жирних кислот поліпшує його змащувальні, антиокиснювальні й екологічні характеристики.

Ключові слова: відпрацьована харчова олія, алканоламіди жирних кислот, високотемпературне мастило.