

*The possibility of using the wastes of the oil and fat industry – phosphatide concentrates as components of lubricating materials was demonstrated in this paper. At the first stage, amidation of phosphatide concentrate from the purification of rapeseed oil by ethylene diamine was carried out using two procedures: without a catalyst and with the use of reagent-catalyst CaO.*

*The samples of urea greases were subsequently synthesized by interaction of amidated phosphatide concentrates with polyisocyanate in the oil media. Aminoamides of fatty acids with polyisocyanate form urea dispersion phase of thixotropic systems, and glycerolphosphatides and calcium glycerolphosphatides perform the function of tribological additives. The method of infrared spectroscopy proved that the full interaction between polyisocyanate and amidated phosphatide concentrate takes place at the molar ratio of 1:3.*

*Physicochemical properties of the developed urea greases were studied and comparative analysis of their quality indicators with the Maspol brand lubricant was performed. The synthesized urea greases are characterized by high mechanical (a change in penetrations after moving of 100,000 double cycles of  $42\text{--}45\text{ mm}\cdot 10^{-1}$ ), colloidal stability (5.2–5.6 % of extracted oil) and high-temperature properties (dropping point above  $230\text{ }^{\circ}\text{C}$ ). In addition, these thixotropic systems are resistant to oxidation, do not cause corrosion of non-ferrous metals, and are able to operate in contact with water. Phosphorous residues improve the lubricating properties of synthesized compositions without any additional introduction of tribological modifiers (critical load is 980–1,039 N, welding load – 1,568–1,744 N). Due to the use of raw materials of plant origin in the composition of lubricating compositions, their biodegradation is enhanced by 6–7 %*

*Keywords: phosphatide concentrate, amidation, urea grease, high-temperature properties, tribological characteristics, biodegradation*

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# SYNTHESIS AND PROPERTIES OF UREA GREASES BASED ON AMINOAMIDES OF PLANT OIL PHOSPHATIDES

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## 1. Introduction

The oil and fat industry, being a structural sub-complex of the agro-industrial sector, concentrates on the cultivation of oilseeds by agricultural households, their subsequent treatment, production, and supply of fat-and-oil products with ensuring the transport logistics of these processes. Oilseeds include the plants, in which the oil content of seeds or fruits is at least 15 %. Among the oilseeds, there are the crops that are grown exclusively for oil production (sunflower, castor, rapeseed, etc.) and the plants of comprehensive use, from which oil is obtained as a by-product during processing. These include spinning oilseeds (cotton, flax, hemp, etc.) and protein oilseeds (soybeans, peanuts, and so on). Since the beginning of 2019/20 marketing year (MY) (July 2019–June 2020) as of May 2020, the world cultivation of oilseeds amounted to 575.23 million tons against 599.92 million tons in 2018/19 MY [1]. Among them, soybeans gave the highest harvest – 335.35 million tons, rapeseed – 68.20 million tons, sunflower seeds – 54.98 million tons, cotton seeds –

44.96 million tons and soybeans – 46.05 million tons [1]. The volume of oil production in the world market in 2019/20 MY amounted to 204.09 million tons [1], the annual increase of this indicator amounted to 0.3 % in the last season and 2.7 % in 2017/18 MY. The largest production volume of oils made up: palm oil – 72.27 million tons, soybean oil – 56.60 million tons, rapeseed oil – 27.31 million tons, sunflower oil – 21.25 million tons [1].

There is always a variety of impurities in freshly squeezed oils. Some of which, such as phospholipids and tocopherols, increase their value. The other, such as waxes, coloration substances, and decomposition products of these substances (free fatty acids, mono- and diglycerides, and other substances), on the contrary, decrease the quality of oils. The peculiarity of phospholipids, which distinguishes them from triglycerides, is the existence, instead of one acyclic group, of phosphoric acid or its derivatives, they are well soluble in oils and their content is 0.2–3.2 % [2]. However, polar phospholipids, as well as protein molecules and other similar substances, even with a small amount of water getting into oil,

form hydrogen bonds with water, intensively absorb water, swell, increase in size, forming flakes, lose solubility in oil and precipitate – fus. At a decrease in temperature, sedimentation separation of phases accelerates. This sediment spoils the marketable look of oil and complicates conducting some technological operations, such as refining. In addition, under the influence of oxygen in the air, phospholipids are easily oxidized with the formation of dark compounds that worsen the oil quality. That is why phospholipids are removed from raw oils, intended for further refining, by hydration in the form of phosphatide concentrates, which have not found a wide practical application until now. Abroad, phosphatide concentrate is manufactured under the commercial brand “lecithin”.

Summing up the outlined, the research into the expansion of possible spheres of using by-products from oil refining – phosphatide concentrates – is quite relevant.

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## 2. Literature review and problem statement

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Phosphatide concentrate (PC) consists of phospholipids (40–60 %), ester non-soluble substances (1–5 %) and oil (triacylglycerides) (39–59 %) with residual moisture content (0.5–3 %) [3].

Due to its structure, phospholipids are amphiphilic, since they consist of a hydrophilic polar head that is formed by a charged phosphate group and two hydrophobic non-polar tails formed by the remains of fatty acids. Due to their surface activity, phosphatide concentrates were used in technological systems for the oil and gas industry as ecologically safe surfactants [3–5]. However, emulsions and suspensions formed from the PC are not stable, so it is used together with other cosurfactant [5, 6]. In paper [6], in order to obtain effective PC-based surfactants and to increase its hydrophilic properties, sunflower PC was subjected to oxidation. The resulting oxidation products showed a greater emulsifying activity compared to non-oxidized. The authors of [7] improved the surface activity of sunflower PC by oil removing, fractionation by alcohols and enzymatic hydrolysis.

The studies carried out in [8] prove the possibility of using in remote rural areas of local untreated plant oil with a biological additive – soybean PC – as an alternative fuel for small diesel generators without modifying their engines. This bio additive lowers surface tension, the average diameter of the drops injected through the oil nozzle (the Sauter diameter), and decreases the deposition on the engine.

In addition, the PC is a mixture of polar molecules that contain phosphorus- and nitrogen-containing compounds, as a result of which they found their application as ecologically safe multifunctional additives to lubricants. The addition of the PC to lubricating materials improves anticorrosive characteristics due to the strong molecular polarity of phospholipids, they are easily adsorbed on metal surfaces, forming a protective barrier that prevents the access of water medium. At the same time, the existence of two active elements (*P*, *N*) simultaneously increases the tribological properties of lubricating compositions [9, 10]. However, these lubricant compositions are characterized by low thermo-oxidative stability [9, 11], due to the existence of triglyceride groups and double bonds in the acyl residues of unsaturated higher fatty acids. That is why it is possible to use the PC as an additive to lubricants only for friction nodes that operate at moderate temperatures.

The use of the PC in high-temperature lubricants requires an additional introduction of oxidation inhibitors or its modification. In paper [11], the antioxidation stability of lubricating systems with the PC, oxidation inhibitors and its modifications were explored. The addition of soybean PC to mineral oil reduced its resistance to oxidation, while performed modifications by various processes of fractionation, oil removal and hydroxylation did not change the value of this indicator. The use of the PC borates did not result in the improvement of antioxidation stability, although by thermogravimetric analysis, the beginning of its weight loss increased by 50 °C [12] compared with the original product, which made up 190 °C [10]. Among all the options, examined in paper [11], it was possible to improve the antioxidation resistance of the PC by the interaction of glycerophospholipids with polyphenol (quercetin) with the formation of a chain-like structure by hydrogen bond and the formation of phenolipids. In [9], the additive, named by the authors “Phospholidin” that was introduced to the composition of bentonite grease, was obtained by the PC transamidation with oxyethylene-diamine. Due to the existence of chelate forming fragments –  $\text{NCH}_2\text{CH}_2\text{OH}$ , amides and phosphatide residues form the complexes that provide improved protective and tribochemical properties of the lubricating composition simultaneously improving their antioxidant properties [9].

In addition, in research [9], the synthesis of surfactants was carried out by the interaction of plant oil phosphatides with monoethanolamines with the use of different catalysts. Research [13] demonstrated the possibility to use aminoamides of stearic acid as components of the disperse phase of urea greases. This all makes it possible to state about the prospects of using the products of PC transamidation by diamines as constituents of urea thickeners of oils. An increase in the production of urea and calcium sulfonate greases is observed now [14]. This is due to the rise in the prices of lithium hydroxide, which is caused by increased demand for lithium by battery manufacturers for mobile electronics and electrified vehicles. This, in turn, resulted in a reduction in the production of simple lithium greases and questions further growth in producing complex lithium greases [14].

In this way, taking into consideration the areas of using the PC and its chemical modifications, explored by the authors, it was proposed to expand the scope of their application as a component of a thickener of lubricating materials, which is promising. On the one hand, the use of the PC makes it possible to expand the raw material base for obtaining urea dispersed phases of greases. On the other hand, the use of the PC solves the problem of involvement and utilization of by-products and wastes of the oil and fat industry.

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## 3. The aim and objectives of the study

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The aim of this study is to involve PC-based nitrogen-containing products for the possibility of obtaining a structural frame of high-temperature urea greases.

To accomplish the aim, the following tasks have been set:

- to obtain the structural frame of urea thixotropic plastic compositions based on the interaction of the product of amidation of ester groups of acylphosphatides by ethylene diamine with isocyanate;
- to explore the physical-chemical, rheological, and operational properties of the resulting urea greases.

#### 4. Materials and methods for the syntheses of aminoamides, urea lubricants; studying their structure and properties

The research used the PC from refined rapeseed oil produced by the Zaporizhzhia butter and fat factory by DSTU 4526. This is the brown flowing mass from the mixture of phosphatides – 55.4 %, plant oil – 42.6 %, ester non-soluble substances – 1.4 %, and residual moisture content – 0.6 %. Fat and acid composition of the PC, determined by GOST 30418 “Vegetable oils. Method for determining the fat and acid composition”, chromatically with the combination with chemical functional analysis of acid and ester numbers made up, % by weight palmitic  $C_{16:0}$  – 3,7, stearic  $C_{18:0}$  – 2,4, oleic  $C_{18:1}$  – 22,5, linoleic  $C_{18:2}$  – 6,1, linolenic  $C_{18:3}$  – 4,8, eicosanic  $C_{20:1}$  – 4,5, erucic  $C_{22:1}$  – 56,0.

The synthesis was carried out by two procedures of PC amidation by diamines with the obtaining of aminoamides (AA). These reactions can be represented as follows (Fig. 1):

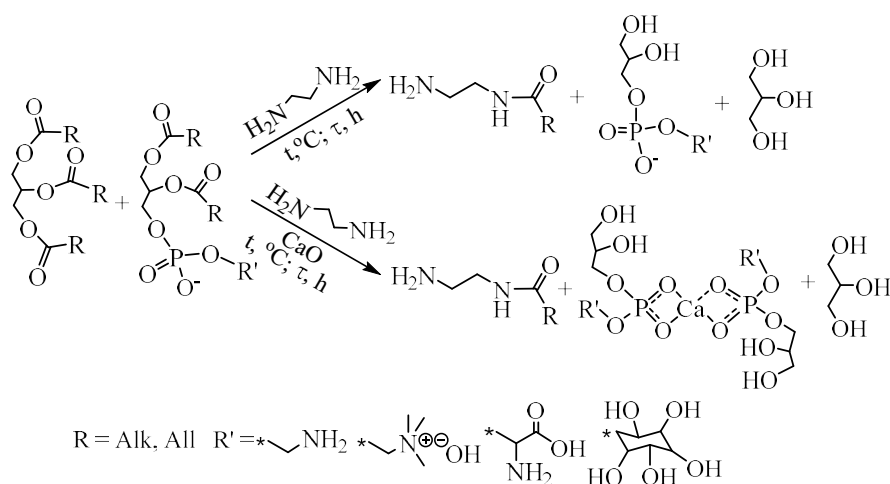


Fig. 1. Scheme of the reaction of PC amidation

According to the first procedure, synthesis was carried out by ethylene diamine amidation (EDA) of the PC without a catalyst at the mol ratio of 1:3.1. The PC was put into a three-neck reactor, equipped with a stirrer, a reverse refrigerator, a thermometer, and a neck for loading ingredients, and heated to 60 °C. The EDA was added to molten PC at constant stirring and the temperature was increased up to 120 °C. At this temperature, the reaction mass was kept for 4 hours. Upon completion of the process, the excess of amine was taken from the reactive mixture. The product from the mixture of AA fatty acids, glycerolphosphatides and glycerin was obtained.

According to the second procedure [15], transamidation was carried out by the interaction of PC, EDA, and reagent-catalyst CaO in nitrogen medium at the mol ratio of 1:3:0.5. The PC was loaded into the reactor and heated up to 60 °C. Calcium oxide and then EDA were added to liquid PC at constant stirring, the temperature was increased up to 120–125 °C and it was kept at constant stirring for 2 hours. The product that consisted of the AA of fatty acids, calcium glycerolphosphatides and glycerin was obtained.

The flow of amidation reactions was determined by decreasing the amine concentration in selected samples by

titration with hydrochloric acid in 2-propanol in presence of bromphenyl blue indicator.

So-called “raw” diphenylmethane diisocyanate (crude MDI) or polyisocyanate (PIC) were used to form the urea dispersed phase of the thixotropic system. This is a viscous liquid of brown color with the characteristic smell, which is the mixture of 55 % MDI (mainly 2,4'- and 4,4'- isomers), 25 % triisocyanates and 20 % polyisocyanates. Basic oil with a viscosity of 21.4 mm<sup>2</sup>/s at 100 °C, which contained 72.0 % by weight of paraffin-naphthene hydrocarbons, 21.6 % of light and 4.7 % of medium aromatic hydrocarbons, as well as 1.7 % of resins, was used as a dispersed medium.

The samples of urea greases (UG) were obtained by technology [16]. Separate solutions of PIC and amidated PC were prepared at the mol ratio of 1:3 in petroleum oil, the mass fraction of the thickener was 23 %. They were heated up to 70 °C and mixed in the reactor, equipped with heating and a stirring device. At this temperature, the urea thickener was formed as a result of the interaction of the PIC

with AA (Fig. 2). This reaction is exothermic and passes at a high rate without the formation of by-products. Glycerolphosphatides (UG 1) and calcium glycerolphosphatides (UG 2) in lubricating compositions acted as additives that enhance tribological characteristics. Subsequently, the mixture was heated, it underwent heat treatment at 150 °C for 30 minutes. After that, the lubricant was cooled to room temperature and homogenized on a laboratory three-roll grinding machine.

The IR spectra of output substances, synthesized products, and finished lubricants 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.

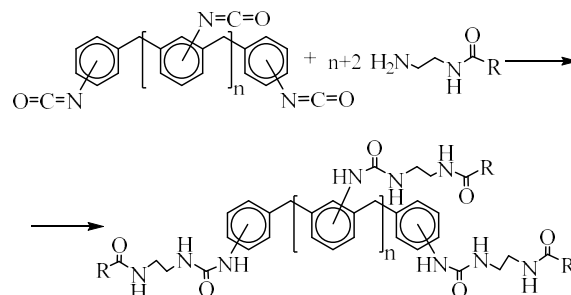


Fig. 2. Scheme of the reaction of polyisocyanate with aminoamides

Thermal properties of greases were studied in static air atmosphere under the following conditions: 23 mg, maximum heating temperature of 1,000 °C, a heating rate of 10 °C/min, on the combined thermal analyzer STA 1400 of Linseis company (USA) with software that makes it possible to record and process data on a computer.

The volumetric-mechanical characteristics of the synthesized greases were assessed by parameters of penetration, mechanical stability, and dropping point. Penetration number was determined by ISO 2137, which is expressed by the depth of immersion in a stirred grease of a cone weighing 150 g for 5 s at the temperature of 25 °C. Mechanical stability was assessed by the difference between the penetration magnitudes after 60 and 100,000 stirring cycles. The dropping point was established by ISO 2176. It is the maximum temperature, at which a grease drop falls from the percussion cup of Ubbelode thermometer.

The stability of greases samples is characterized by the indicators of colloidal stability, resistance to oxidation and water resistance. Colloidal stability was determined in accordance with GOST 7142. The essence of this method is to determine the amount of oil, which was pressed off the lubricant on the KSA device at the load of  $1,000 \pm 10$  g. Resistance to oxidation of greases was analyzed by the change of acid number after the treatment at 120 °C for 10 hours on a copper plate in accordance with GOST 5734. Water resistance was determined by ASTM D 1264 – the ability to a grease to remain in a bearing under the influence of a water jet heated to 79 °C for 1 hour, expressed by the quantity of washed off grease as a percentage.

Anticorrosion properties of greases were evaluated by ASTM D 4048. The method involves keeping a copper strip in the samples of greases for 24 hours at the temperature of 100 °C and a visual comparison of a change of the strip color with corrosion standards.

Tribological characteristics of lubricants were determined on a four-ball friction machine at a constant temperature and step-by-step increase in load according to the indicators of critical load ( $P_c$ ) and welding load ( $P_w$ ) according to GOST 9490.

Environmental characteristics were assessed by the indicator of biodegradation according to DSTU 4247 (CECL 33-A-93). The essence of the method is the comparison of infrared spectra of lubricating materials, kept in mineralized aqueous solution with the inoculant for 21 days in aerobic conditions, and the comparison of the degree of biodegradation in relation to control material RL 130.

## 5. Results of studying the structure and properties of urea lubricating compositions

### 5.1. Obtaining urea thixotropic plastic compositions

Infrared spectra of output PC, amidated product, the PIC, and finished urea grease are shown in Fig. 3.

In the PC, a very broad intensive band in the region of  $3600\text{--}3000\text{ cm}^{-1}$ , which is caused by valence oscillations of hydroxylic groups (OH), appears. At  $3,600\text{--}3,500\text{ cm}^{-1}$ , OH oscillations are related to internal molecular hydrogen bonds. At  $3,500\text{--}3,300\text{ cm}^{-1}$ , OH oscillations are involved in intermolecular hydrogen bonds. At  $3,400\text{--}3,200\text{ cm}^{-1}$ , there are characteristic OH oscillations, bound by hydrogen bond of OH O=C. At  $2,917$  and  $2,849\text{ cm}^{-1}$  – valence oscillations of CH bond in  $\text{CH}^3$ ,  $\text{CH}^2$

and CH groups, at  $1,745$  and  $1,160\text{ cm}^{-1}$  – valence oscillations of the carbonyl group of C=O esters, in addition, at  $1,050\text{ cm}^{-1}$  – valence oscillations P-O of POOH group. In amidated PC, there appears the band at  $3300\text{ cm}^{-1}$  of valence oscillations of NH group, besides, bands of  $1,745$  and  $1,160\text{ cm}^{-1}$ , C=O groups of esters of PC disappear during amidation with the occurrence of corresponding  $1,640\text{ cm}^{-1}$  – valence and  $1,555\text{ cm}^{-1}$  and  $1,055\text{ cm}^{-1}$  – deformation oscillations C=O of the groups of formed amides [17].

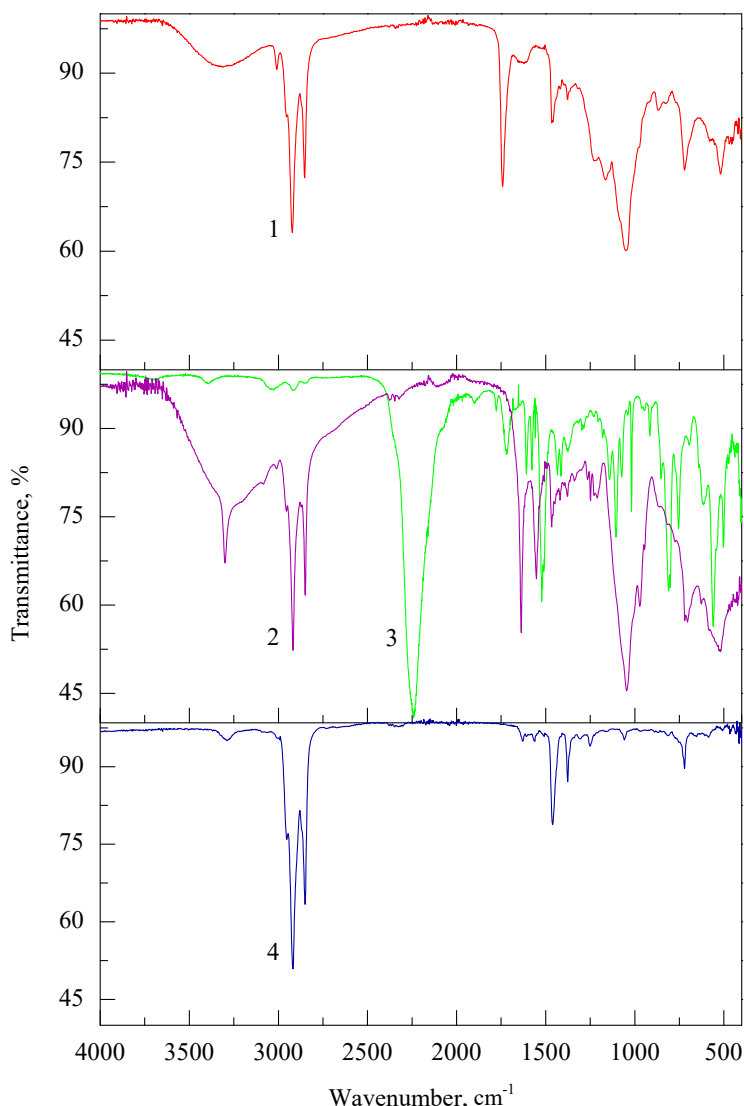


Fig. 3. Infrared-spectra: 1 – output phosphatide concentrate, 2 – amidated phosphatide concentrate, 3 – polyisocyanate, 4 – finished urea grease

Amidated PC are lubricant-like substances of dark brown color with a melting point of  $50\text{--}62\text{ °C}$ , which are well soluble in aliphatic and aromatic hydrocarbons.

On the PIC spectrum, there is an intense band of asymmetric valence oscillations in the region of  $2,280\text{--}2,230\text{ cm}^{-1}$ , characteristic of groups  $\text{-N=C=O}$ . There is no such band in finished UG and it disappears at  $3,300\text{ cm}^{-1}$  that corresponds to the NH group, which testifies to complete interaction of the PIC with AA, which are obtained using different procedures with the formation of urea dispersed phase. The resulting urea greases are homogeneous ointment-like products of dark yellow color.



**5. 2. Studying the physical-chemical, rheological, and operational properties of obtained urea lubricants**

To determine the upper temperature boundary of obtaining synthesized greases, we used, like in research [13], the method of derivatographic analysis. This method is based on the simultaneous measurement of the weight and enthalpy of the studied material during its uniform heating in comparison with the temperature of the standard – a substance, the temperature of which virtually coincides with the temperature of the peak space. In this case, the temperature in the furnace will not differ from the temperature of the sample, only if there are no physical or chemical transformations in the sample. The deviation of the sample temperature from the heating temperature indicates physical and chemical transformations in the dispersed medium and the dispersed phase of lubricant compositions. Fig. 4 shows derivatographic analysis of the sample of lubricant UG 1, the curves of weight loss of the sample – thermogravimetric (TG) and differential thermal analysis of DTA.

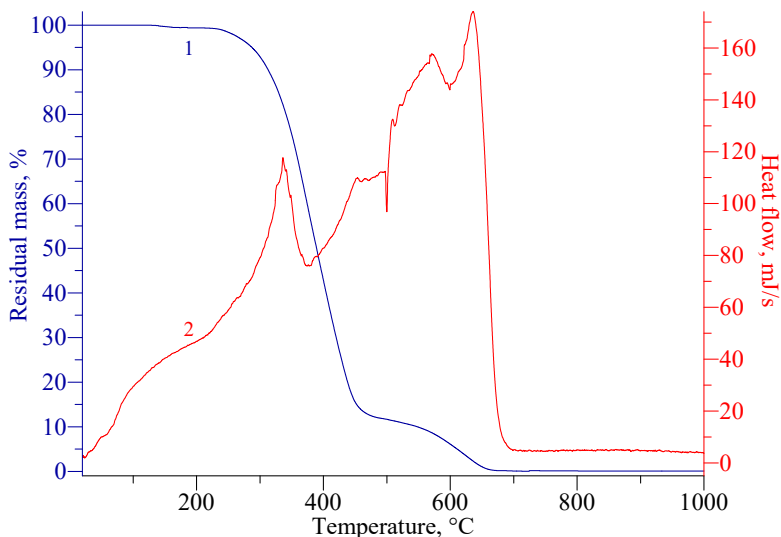


Fig. 4. Derivatogram of the sample of grease UG 1: 1 – thermogravimetric curve; 2 – the curve of differential thermal analysis

Table 1 gives the results of the study of the influence of various PC amidation procedures on the properties of lubricating compositions, made on their basis. In addition, the properties of the synthesized urea thixotropic systems were compared with the brand grease, produced by VAT “AZMOL”. This grease is intended for friction units of technological equipment and is serviceable within temperatures from –20 to 150 °C. It is petroleum oil, thickened by urea and containing multifunctional additives.

Table 1  
Physical-chemical properties of developed urea greases based on amidated PC and brand grease Maspol

Indicator	Testing method	Values of indicators		
		UG 1	UG 2	Maspol
Penetration at 25 °C with stirring, mm·10 <sup>-1</sup> : – 60 double cycles ( <i>P</i> <sub>1</sub> ) – 100,000 doubles cycles ( <i>P</i> <sub>2</sub> ) – mechanical stability, change Δ <i>P</i>	ISO 2137	285	278	270
		330	320	342
		45	42	72
Dropping point, °C	ISO 2176	246	248	228
Colloidal stability, % of extracted oil	GOST 7142 method A	5.6	5.2	10.0
Tribological characteristics on FBM (Four Ball Machine) at the temperature of (20±5) °C: – critical load ( <i>P</i> <sub>c</sub> ), N – welding loading ( <i>P</i> <sub>w</sub> ), N	GOST 9490	980	1039	921
		1,568	1,744	1,568
Water washout at temperature 79 °C, %	ASTM D 1264	1.1	0.8	0.9
Resistance to oxidation: increase in acid number (120 °C, 10 hours), mg KOH/g	GOST 5734	0.16	0.14	0.22
Copper strip corrosion	ASTM D 4048	1a	1a	1a
Biodegradation, %	DSTU 4247 (CECL 33-A-93)	37.3	36.4	29.8

Industrial lubricant belongs by consistency to the NLGI (National Lubricating Grease Institute) grade 2, so for objective comparison of properties, the samples of synthesized lubricating compositions of the corresponding class were produced. The basis of the NLGI lubricant 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 most common among industrially produced lubricants and the value of their worked penetration is in the range from 275 to 295 mm·10<sup>-1</sup>.

**6. Discussion of the results of studying the properties of urea lubricants based on amidated phosphatide concentrates**

In the IR spectrum of the finished urea grease (Fig. 3), there is no band of asymmetric valence oscillations of N=C=O groups. This indicates complete interaction of the PIC with AA, obtained from the PC with the use of molar ratios of the components and the formation of a urea thickener according to the above scheme. Thus, firstly, there is no excessive consumption of reagents. And secondly, the PIC and AA, like amines of fatty acids, according to the Globally Harmonized System (GHS), belong to the substances that are hazardous to human health, so it is possible to obtain a toxic grease if the ratio until their full consumption is not observed. Although the product is complete interaction of these components – urea – is safe, accordingly, the greases based on this organic thickener, are also safe. Due to the use of raw MDI, we obtain an organic thickener, the molecules of which contain a greater number of urea compounds compared with the classical diurea. The greater number of urea compounds, according to some researchers [18], has a positive effect on the lubricating properties of the UG and the performance of the composition as a whole.

In order to find out whether the synthesized greases belong to high-temperature ones, it is necessary to decide on what temperature is considered high. However, there is no consensus about this. Thus, the author of [19] gives the temperature range of 100–120 °C, where the lubricating material can be operated at normal or extended service life without replacement, which can be called high temperature. A researcher into lubricating grease of Petro-Canada Lubricants determines if the greases are able to maintain its consistency, structure, and performance at the temperature above 125 °C, they are high-temperature lubricants. It is considered in paper [20] that high-temperature lubricants are those that are able to function in the temperature range from 170 to 250 °C.

It is also known that greases are sensitive to high temperatures. Melting of a thickener leads to dilution of a lubricant with a loss of strength boundary or, on the contrary, thermostrengthening. At the same time, it provokes the thermal-oxidative destruction of molecules of the dispersed medium. The main indicators of the thermal stability of lubricants are dropping point and maximum operating temperature. As follows from the research results (Table 1), the dropping point of the UG, at which it transfers from a plastic solid into a liquid state, is 246–248 °C. Paper [19] presents the calculation of the maximum working temperature of lubricants by dropping point. At dropping point of the grease of up to 150 °C, the maximum working temperature is by 25 °C lower, at dropping point from 150 to 205 °C, working temperature is by 40 °C lower, and at dropping point above 205 °C, the working temperature will be 70 °C lower. The maximum working temperature of synthesized greases is 176–178 °C, which indicates belonging to this thixotropic system to the high-temperature system. In addition, it is necessary to keep in mind, according to the Arrhenius equation, which concerns the use of lubricating materials at different temperatures, it is noted that at each increase in working temperature by 10 °C, the service life of greases is reduced by half [19].

In addition, to prove the upper temperature of the application of synthesized greases, we used the method of derivatographic analysis. As it can be seen from the thermogravimetry curve (Fig. 4, curve 1) of synthesized grease UG 1, it remains thermally stable up to 180–200 °C, there is no weight loss. Only at reaching 250–300 °C, the intense weight loss begins, apparently with chemical decomposition in the range of 350–450 °C of both a urea thickener itself, and dispersed medium of a grease with its complete decomposition at 650 °C. The sample of grease UG 1 with glycerolphosphatides does not form ash. At the same time, synthesized grease UG 2 with calcium glycerolphosphatides, as well as greases based on soap thickeners, forms sediment that remains constant in the temperature range of 650–1,000 °C.

On the curve of differential thermal analysis (Fig. 4, curve 2), starting from the temperature of 350 °C, there is a range of exothermic effects that characterize the thermo-oxidative destruction of a dispersed medium, dispersed phase, and grease additive. After reaching the temperature of 650 °C, any energy effects disappear due to the complete thermal decomposition of the sample.

Thus, the upper temperature boundary of using the developed greases, according to derivatographic research and by estimation of the dropping point, is at the level of 180–200 °C.

During operation, greases are affected not only by elevated temperatures, but also by rates and loads, which lead to mechanical destruction of their structural frame. The grease influenced by small loads under conditions of normal temperature behaves like a solid, when the critical load value is achieved, it begins to become plastically deformed and flow like a liquid, and after the forced removal, acquires the properties of a solid body [21]. The ability of antifric-tion greases to resist the destruction of the dispersed phase is characterized by their mechanical stability. Mechanical stability is the ability of dispersed systems to maintain their consistency and mechanical properties under a long-term impact of shear deformation, which characterizes the thixotropic properties of greases. That is their ability to restore instantly its structural framework after stopping loading in friction nodes. The grease that is resistant to mechanical destruction does not significantly change its structural frame, is not diluted, and does not flow from a friction node, which extends its shelf life. If a grease hardens strongly after the loading action stops, it ceases to arrive at a friction node. Greases with low mechanical stability get destroyed quickly, get diluted, and flow from friction nodes. Mechanical stability of synthesized greases was assessed by the difference  $P_2 - P_1 = \Delta P$  in the following way: at  $\Delta P < 30$  – excellent; at  $\Delta P = 30 - 60$  – good; at  $\Delta P = 61 - 100$  – satisfactory; at  $\Delta P > 100$  – unsatisfactory [22]. According to the data from Table 1, it is possible to argue that the mechanical stability of both greases is rather high.

Greases based on urea thickeners are known to be serviceable in a wide range of temperatures, at large loads, in the presence of water, to be resistant to oxidation and mechanical destruction. As data of Table 1 show, the synthesized greases are characterized by high water-, anti-oxidation stability and do not cause corrosion of non-ferrous metals. The existence of glycerolphosphatides in the UG 1 sample allowed improving the tribological properties up to the level of industrial grease with additives and the UG 2 product with calcium glycerolphosphatides is superior to it. In addition, the developed lubricants based on the amidated PC are superior by environmental characteristics to the brand analog; the indicator of biodegradation was reduced almost by 7 %.

Summing up the data, it is possible to state that greases based on amidated wastes of the oil and fat industry demonstrated high operational characteristics. Their tribological properties are provided by existing phosphorus-containing residues. And formed AA urea thickener of the lubricant grease is characterized by high volumetric and mechanical properties, resistance to oxidation, water, and workability at elevated temperatures.

However, these studies are limited to the use of the PC from refining rapeseed oil. It is interesting to study the influence of the PC from refining different plant oils on rheological, physical-chemical, and operating properties of urea greases based on them.

In particular, in order to obtain these urea lubricant compositions, petroleum oil was used as a dispersed medium. That is why it is planned to aim the next stage of research to studying the impact of different dispersed media on the quality indicators of these greases. The application of the relevant synthetic oils will expand the temperature boundaries of their use, and biodegradable dispersed media will enable the development of eco-safe lubricating materials.

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## 7. Conclusions

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1. Amidation of phosphatide concentrates by ethylene diamine with obtaining the products consisting of aminoamides of higher fatty acids and phosphorus-containing residues was carried out. With their further use, the possibility of synthesis of urea thickeners of lubricating compositions was proved. The method of the infrared spectroscopy proved that at the mol ratio of 1:3, there occurs complete interaction of the PIC and amidated PC with the formation of urea grease of NLGI grade 2 at the mass fraction of a thickener of 23 %.

2. The developed urea greases are characterized by the regulated thixotropic structure, high mechanical stability, the upper temperature limit of the application boundary is 180–200 °C. Existence of phosphorus-containing residues of glycerolphosphatides and calcium glycerolphosphatides in the samples of synthesized greases improve lubricating properties without the introduction of other tribological additives. In particular, the critical load is 980–1039 N, welding load is 1568–1744 N. Application of eco-friendly components as a part of the lubricating composition enhances their biodegradation to 36.4–37.3 %.

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