



МАТЕРИАЛОВЕДЕНИЕ В МАШИНОСТРОЕНИИ

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DEVELOPMENT OF THE COMPONENT COMPOSITION OF THE BIODEGRADABLE LIQUID LUBRICANT FOR SAW CHAINS

The procedure of working out of the component composition of the biodegradable liquid lubricant for the saw chains is shown. The experimental statistical mathematical model is developed for the optimization of the formulation of the main base taking into account the necessary viscosity and temperature parameters. At the same time the kinematic viscosity of the base body at the temperature of 40 °C and its pure point are chosen as the optimization criteria and the following three factors are used as the optimization parameters: the content of vegetable (rapeseed) oil in the composition, the viscosity of mineral oil at the temperature of 40 °C and the content of an adhesive additive in the base composition. The component composition of the lubricant is adjusted to achieve the required level of tribological properties of the saw chain oil and to ensure the stability of all its characteristics during storage (at least 12 months) and operation at a given level of biodegradability (at least 90 %). In particular, the biodegradable calcium sulfonate grease OIMOL KSC BIO was selected as an additive to improve tribological parameters. The special adhesive additive for vegetable oils of the Petrolad 484BD brand is used to increase the sedimentation stability, and the highly refined oil of group III according to the API standard is recommended to use as a mineral component. The developed lubricant has the following characteristics: density at 15 °C — 926 kg/m³, kinematic viscosity at 40 °C — 47.3 mm²/s, kinematic viscosity at 100 °C — 9.9 mm²/s, viscosity index — 202, pour point — -28 °C, flash point — 272 °C, critical load — 872 N, welding load — 1,600 N, wear index at 200 N — 0.39 mm, biodegradability — 93 %.

Keywords: liquid lubricant, saw chain, vegetable oil, experimental and statistical model, rheological and tribological properties, property stability, biodegradability

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Introduction. Global trends in the development of the green economy include strengthening the role of renewable energy, clean transport, organic agriculture, green construction, improving the efficiency of raw materials and energy use, increasing the degree of waste recycling, protecting and restoring natural ecosystems [1]. In this regard, along with solar and hydrogen energy, information technologies and electric transport, the use of biodegradable materials in many areas (for example, lubricants, disposable packaging) are important components of the green economy, the development of which has recently received increasing attention [2, 3].

Most lubricants are currently produced on the basis of mineral oils, which at the stages of manufacture, transportation, storage, use and disposal create biotoxicological threats to service personnel and living organisms, as well as environmental threats (to soil, water, air). The struggle to reduce these threats is conducted primarily through the selection and use of more acceptable raw materials, primarily of plant origin. A number of countries have developed regulatory documents and recommendations of a promotional and compulsory nature aimed at increasing the use of vegetable oils in the formulations of lubricants [4–6].

Biodegradability refers to the ability of organic matter to be degraded by microorganisms. Ideally, this chemical decomposition occurs before complete mineralization. The term “biodegradable lubricants” refers to lubricants that are not primarily harmful to the environment. These lubricants must be biologically easily degradable, have a low toxicity to various organisms and do not accumulate in the environment. At the same time, these products must remain operational for a certain period of time, as well as be environmentally friendly.

It should be noted that all lubricants undergo biodegradation, the difference is only in the process intensity. Mineral (petroleum) oils are also biodegradable to a certain extent due to the microorganisms contained in the environment, but the decomposition of such materials takes months or even years. At the same time, the natural decomposition system is overloaded due to the significant amounts of accumulation of these products as a result of unforeseen or technologically determined leaks.

The development and implementation of biodegradable lubricants is one of the most promising areas for the development of the global lubricant industry. The leading role in solving this problem belongs to bioresources — vegetable oils, in particular rapeseed oil and its transesterification products, which are an acceptable alternative to petroleum feedstock for the lubricant production. The global production of rapeseed oil is steadily expanding and reached 60 million t in 2019, of which about 10 % is used for the production of biofuels and only about 1 % for the manufacture of biodegradable lubricants.

The main objective criterion by which the material is classified as biologically safe lubricants is the degree of biodegradability of at least 60 % [7]. The technical documentation for biodegradable lubricants most often indicates the degree of biodegradability at the level of 80–90 %.

The use of biodegradable lubricants in transport and other machinery has been expanding in recent years. From an ecological point of view, the use of these products is especially justified where their rapid decomposition is necessary, for example, in agriculture, forestry and water management, mobile hydraulic systems of machines, in the construction industry [8, 9]. Currently, the use of biodegradable lubricants is especially relevant when there is an inevitability or a high probability of their entering the soil, water bodies, the atmosphere, including in forestry (small-sized equipment (chainsaws, brush cutters) and equipment for logging (harvesters, forwarders, etc.), in agriculture (tillage and harvesting equipment), in transport (two-stroke internal combustion engines), etc.

The objective of this work is to study the properties and develop the component composition of biodegradable oil for saw chains, made on the basis of domestic vegetable raw materials (rapeseed oil), according to the technical characteristics corresponding to the best world analogs, with a degree of biodegradation of at least 90 %.

Research method. The following components were used to produce experimental samples and pilot batches of saw chain oil: group I mineral oil according to the API standard of the И-40А (I-40A) brand [10] and highly refined oil of group III according to the API standard of the HC7 (NS7) brand (TU BY 300042199.062-2017) produced by OJSC Naftan (Belarus), rapeseed oil [11] produced by JSC “Raps” (Belarus), adhesive additives КП-20 (KP-20) (TU 38/101209-90) produced by “Industrial commercial firm “RUSMA” Limited company (Russia) and biodegradable adhesive additive Petrolad 484BD produced by BRB company (Poland), additive in the form of biodegradable calcium sulfonate lubricant OIMOL KSC BIO (TU190410065.023-2021) produced by ODO “Spetsmazki” (Belarus).

The rheological characteristics of biodegradable liquid lubricants were determined according to the following parameters: density at 15 °C according to [12]; kinematic viscosity at 40 and 100 °C according to [13]; pure point according to [14]; flash point according to [15]. Tribological characteristics (critical load, welding load, wear index) were determined on a four ball machine according to [16]; the friction coefficient and the wear rate of the friction pairs were determined according to the scheme of reciprocating movement of the contacting bodies on the automated tribometer АТВІІ-9М (ATVP-9M) according to the method described in [17]; the mass fraction of mechanical impurities according to [18]; the water content according to [19]. The degree of oil

biodegradability was estimated based on the content of fatty acid methyl esters determined by gas-liquid chromatography (GLC analysis) using an Agilent 7820A GC gas chromatograph (Agilent Technologies, USA).

Selection of the initial components of the saw chain oil. Parameters of liquid lubricants for saw chains are set by the saw manufacturers. Quite often, they produce branded lubricants under their own name, without disclosing their parameters, but only recommending them for the maintenance of their equipment. However, an analysis of the literature and products available on the market makes it possible to develop the basic requirements for the composition and parameters of such lubricants. As a rule, to ensure the operability of saw chains, the oil for them must have a certain viscosity, be characterized by the necessary level of tribological and adhesive properties, and have a set pour point based on the climatic conditions of its use. The formulation of the lubricant composition for saw chains consists of a single base oil (or a mixture of them of a certain viscosity) and a set of functional additives. With the help of additives, if necessary, the oil stickiness (adhesive additives), tribotechnical properties (anti-wear and extreme pressure additives) and the pour point (depressant additives) are adjusted.

Viscosity characteristics. The oil for saw chains must provide lubrication of the chain rail along the entire length, sprockets and penetrate into the bushing connections of the chain links. There is also a partial lubrication of the cutting edge of the chain, which reduces the cutting force when sawing wood. The delivery of the oil to all the specified locations is ensured due to the compliance with the viscosity parameters of the base oil. On average, the kinematic viscosity of such oils is normalized in the range of 35–55 cSt at 40 °C. The most approachable option is provided by the use of the base mineral oil И-40А (I-40A). To obtain a biodegradable oil, it is necessary to use vegetable oils of the appropriate viscosity. As a rule, in such cases, a mixture of vegetable and mineral oils is used, which provides a given level of biodegradation. It should be noted that the adhesive additives also affect the lubrication and viscosity of the oil. Thus, in the case of a biodegradable lubricating oil, the key viscosity parameter is influenced by three components: vegetable oil, mineral oil, and adhesive additive.

Adhesive properties. The oil stickiness is necessary to prevent the lubricant from quickly dropping off the chain, which is especially important when working at high speeds. High-molecular-weight hydrocarbons, which dramatically increase the viscosity, such as polybutenes with a large molecular weight, are used as adhesive additives [20, 21]. In the CIS, the most common adhesive additive for lubricants is the additive КИ-20 (КР-20) (ТУ 38/101209-90), which is a concentrated solution of polyisobutylene with an average molecular weight of 15,000–25,000

in industrial oil И-12А (I-12A). For biodegradable lubricants, biodegradable additives are offered, for example, the biodegradable adhesive additive Petro-lad 484BD. The level of adhesive properties of oils for saw chains is not standardized and is set by each manufacturer individually. However, a common practice for the use of adhesive additives is the introduction in an amount of 1.0–2.0 wt. %.

Tribological characteristics. The main tribological characteristics for liquid and plastic lubricants are determined according to [16]: load-bearing capacity (according to the critical load of the P_c), maximum load capacity (according to the welding load of the P_w), anti-wear properties (according to the wear scar diameter D_w) and extreme pressure properties (according to the load wear index LWI).

Low-temperature properties. In the climatic conditions of Belarus, all-season oils for saw chains should ensure the normal operation of the saw when the air temperature drops to –15 °C, and therefore the pure point of saw oils should not be higher than –25 °C. The pure point of oils depends on their composition, and for mineral oils it decreases with an increase in the content of naphthenes and a decrease in the content of aromatic hydrocarbons and paraffins, and it is also directly related to the oil viscosity [22, 23]. So, for mineral oil И-40А (I-40A), the pour point is –12...–15 °C. To reduce the pure point of mineral oils, depressant additives are introduced into them. The pure point of rapeseed oils, as a rule, is –25...–35 °C and mainly depends on the content of erucic acid, the amount of which in technical rapeseed oils is quite large [24]. The pour point of rapeseed oil produced by JSC “Raps” is –30 °C. At the same time, for example, a mixture of rapeseed oil produced by JSC “Raps” and mineral oil И-40А (I-40A) produced by OJSC Naftan in a ratio of 80:20 has a pure point of –28 °C.

Storage life. For biodegradable lubricants, as a rule, the shelf life in sealed containers is three years. After opening, such materials, due to the requirements for rapid biodegradation, must remain operational for one year (season).

Development of an experimental and statistical model of the process of obtaining a biodegradable liquid lubricant. As mentioned above, the main requirement for saw chain oil is to provide viscosity characteristics over a wide temperature range. This indicator is mainly affected by the oil base body. Therefore, to optimize its composition, it is advisable to use mathematical modeling tools.

When developing an experimental and statistical model of the process of obtaining the base body of biodegradable oil for saw chains, the kinematic viscosity of the finished oil at a temperature of 40 °C ν_{40bo} (mm²/s (cSt)) — Y_1 and the pour point of the oil T_{pp} (°C) — Y_2 were selected as optimization criteria, and the following three factors were used as optimization parameters: the content of rapeseed

oil C_{ro} (wt.%) — x_1 ; the viscosity of mineral oil at a temperature of 40 °C v_{40mo} (mm²/s (cSt)) — x_2 ; the content of the adhesive additive in the finished oil C_{aa} , (wt.%) — x_3 . When developing an experimental statistical model of all-season biodegradable oil for saw chains, experimental data were obtained using industrial oil of И-40А (I-40A) grade [10], synthetic group III oil of the HC7 (NS7) brand (TU BY 300042199.062-2017), rapeseed oil [11], and an adhesive additive КП-20 (KP-20).

To make a reasonable choice of the component composition of the base body and reduce the duration and volume of tests, the method of mathematical planning of the experiment was used [25].

Based on a priori information, the levels and intervals of variation of factors (optimization parameters) were selected (Table 1).

In accordance with the experiment conditions, samples of biodegradable oil for saw chains were prepared and their viscosity values were determined at 40 °C v_{40bo} (mm²/s) and the pour point T_{pp} (°C). The values of viscosity $v_{40bo}(Y_1)$ and pour point $T_{pp}(Y_2)$ are obtained as the average of three measurements. The matrix of the composition second-order plan for the three factors is presented in Table 2.

As a result of the analysis of the experimental data given in Table 2, similar to the one described for the case of biodegradable grease [26], the regression equation is obtained:

$$Y_1 = 46.67 - 6.5x_1 + 4.5x_2 + 2.25x_1x_3 - 2.46x_{12} + 3.04x_{22} + 5.04x_{32}. \quad (1)$$

The adequacy of the obtained model was checked by Fischer's F -test. To calculate the variance of s_{ad}^2 adequacy, we find the sum of the s_R squared deviations of the calculated values \hat{Y}_1 from the experimental values Y_1 at all points of the plan (see Tables 1, 2). The calculated values \hat{Y}_1 were determined by the expression (1), $s_E = 8.67$. The calculation data is listed in Table 3.

The variance was found:

$$s_{ad}^2 = \frac{s_R - s_E}{N - k' - (n_0 - 1)} = \frac{88.17 - 8.67}{15 - 7 - (3 - 1)} = 13.25,$$

where N is the total number of experiments; k' is the number of coefficients of the approximating polynomial (the number of significant factors); n_0 is the number of experiments in the center of the plan.

The variance $s^2\{Y_1\} = 4.33$, therefore, the calculated value of the F -test:

$$F_p = \frac{s_{ad}^2}{s^2\{Y_1\}} = 3.06.$$

The tabular value of the F -test at a 5 % significance level and the number of degrees of freedom for the larger variance $m_1 = N - k' - n_0 + 1 = 5$, the smaller variance $m_2 = n_0 - 1 = 2$, corresponds to $F_T = 19.37$. Since $F_p < F_T$, the resulting model (1) is adequate at a 5 % significance level.

Table 1 — Levels and intervals of factors

Factors (parameters)	Code designation	Variation intervals	Factor levels		
			main 0	upper +1	lower -1
Rapeseed oil content C_{ro} , wt.%	x_1	5.0	80.0	85.0	75.0
Mineral oil viscosity v_{40mo} , mm ² /s	x_2	28.0	68.0	96.0	40.0
Adhesive additive content C_{aa} , wt.%	x_3	0.5	1.0	1.5	0.5

Table 2 — Planning matrix and experiment results

Experiment number	x_0	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	x_{12}	x_{22}	x_{32}	Y_1 , viscosity v_{40bo} , mm ² /s	Y_2 , pure point T_{pp} , minus °C
1	+	+	+	0	+	0	0	+	+	0	42	27
2	+	+	-	0	-	0	0	+	+	0	34	24
3	+	-	+	0	-	0	0	+	+	0	63	25
4	+	-	-	0	+	0	0	+	+	0	50	28
5	+	0	0	0	0	0	0	0	0	0	45	24
6	+	+	0	+	0	+	0	+	0	+	47	24
7	+	+	0	-	0	-	0	+	0	+	44	27
8	+	-	0	+	0	-	0	+	0	+	50	28
9	+	-	0	-	0	+	0	+	0	+	56	25
10	+	0	0	0	0	0	0	0	0	0	46	25
11	+	0	+	+	0	0	+	0	+	+	60	26
12	+	0	+	-	0	0	-	0	+	+	57	25
13	+	0	-	+	0	0	-	0	+	+	53	25
14	+	0	-	-	0	0	+	0	+	+	49	27
15	+	0	0	0	0	0	0	0	0	0	49	24

Table 3 — Auxiliary table for calculating s_R

Experiment number	Y_1	\hat{Y}_1	$Y_1 - \hat{Y}_1$	$(Y_1 - \hat{Y}_1)^2$
1	42	44.00	-2.00	4.00
2	34	37.50	-3.50	12.25
3	63	59.50	3.50	12.25
4	50	48.00	2.00	4.00
5	45	46.67	-1.67	2.78
6	47	45.00	2.00	4.00
7	44	40.50	3.50	12.25
8	50	53.50	-3.50	12.25
9	56	58.00	-2.00	4.00
10	46	46.67	-0.67	0.44
11	60	59.00	1.00	1.00
12	57	59.50	-2.50	6.25
13	53	50.50	2.50	6.25
14	49	50.00	-1.00	1.00
15	49	46.67	2.33	5.44
$s_R = \sum (Y_1 - \hat{Y}_1)^2 = 88.17$				

The analysis of equation (1) shows that within the established intervals of variation of factors, an increase in factor x_3 has a greater effect on the increase in the viscosity index than in factors x_1 and x_2 , however, due to the presence of quadratic terms in equation (2), this dependence is nonlinear, which is most strongly manifested through factor x_3 .

For the convenience of interpreting the results obtained and using the equation (1) for practical calculations, it is necessary to switch from the coded values (x_1, x_2, x_3) of the factors to the natural values (C_{ro}, v_{40mo}, C_{aa}). For this purpose, the formulas were used:

$$x_1 = \frac{C_{ro} - C_{ro0}}{\Delta C_{ro}}; \quad x_2 = \frac{v_{40mo} - v_{40mo0}}{\Delta v_{40mo}}; \quad x_3 = \frac{C_{aa} - C_{aa0}}{\Delta C_{aa}},$$

where $C_{ro0}, v_{40mo0}, C_{aa0}$ are the natural values of the factors at the main levels; $\Delta C_{ro}, \Delta v_{40mo}, \Delta C_{aa}$ are the values of the variation intervals.

Thus, according to Table 2,

$$x_1 = \frac{C_{ro} - 80}{5}; \quad x_2 = \frac{v_{40mo} - 68}{28}; \quad x_3 = \frac{C_{aa} - 1}{0.5}.$$

Taking into account the transition to the natural values of the factors, the regression equation (1) will take the form $v_{40bo}(Y_1)$:

$$v_{40bo}(Y_1) = -153.868 + 5.8 \cdot C_{ro} - 0.1373 \cdot v_{40mo} + 0.4 \cdot C_{ro} \cdot C_{aa} - 40 \cdot C_{aa} - 0.04 \cdot C_{ro}^2 + 0.00127 \cdot v_{40mo}^2 + 4 \cdot C_{aa}^2 \quad (2)$$

Similarly, the regression equation for the pure point of $T_{pp}(Y_2)$ is obtained:

$$T_{pp}(Y_2) = 24.33 - 0.50 \cdot x_1 + 1.5 \cdot x_1 \cdot x_2 - 1.5 \cdot x_1 \cdot x_3 + 0.75 \cdot x_2 \cdot x_3 + 0.96 \cdot x_1^2 + 0.71 \cdot x_2^2 + 0.71 \cdot x_3^2 \quad (3)$$

The resulting model is adequate at a 5 % significance level, since

$$F_p = \frac{S_{ad}^2}{s^2 \{Y_2\}} = 0.23 < F_T = 19.37.$$

After switching from the coded values (x_1, x_2, x_3) of the factors to the natural values (C_{ro}, v_{40mo}, C_{aa}), the equation (3) will take the form:

$$T_{pp}(Y_2) = 317.927 - 6.685 \cdot C_{ro} + 0.00714 \cdot C_{ro} \cdot v_{40mo} - 0.8144 \cdot v_{40mo} - 0.4 \cdot C_{ro} \cdot C_{aa} + 19.143 \cdot C_{aa} + 0.0714 \cdot v_{40mo} \cdot C_{aa} + 0.04 \cdot C_{ro}^2 + 0.00127 \cdot v_{40mo}^2 + 4 \cdot C_{aa}^2 \quad (4)$$

The analysis of the equation (4) shows that within the established intervals of variation of factors, an increase in factor x_1 also has a greater effect on the increase in the value of the pure point than factors x_2 and x_3 , however, due to the presence of quadratic terms in equation (4), this dependence is nonlinear, which is most strongly manifested through factor x_1 .

The regression equations (2) and (4) can be used to select the component composition of the base body that provides optimal values of the kinematic viscosity and pour point of the biodegradable oil for saw chains, depending on the factors under study (C_{ro}, v_{40mo}, C_{aa}). Figures 1–6 show the graphical dependences of the kinematic viscosity and pure point of the base body of the biodegradable oil obtained using the equations (2) and (4) on the studied factors. When constructing the response surface (Sigma Plot 12 program), two factors varied each time with a fixed third factor.

From the analysis of the obtained data presented in Figures 1–6, it can be seen that the main factor affecting the value of the kinematic viscosity of the base body of the biodegradable oil v_{40bo} is the content of rapeseed oil C_{ro} , then the content of the adhesive additive C_{aa} and the viscosity of the mineral oil v_{40mo} . The highest values of the kinematic viscosity of the base body of the biodegradable oil (at the level of $v_{40bo} = 48-50 \text{ mm}^2/\text{s}$) are observed when it contains

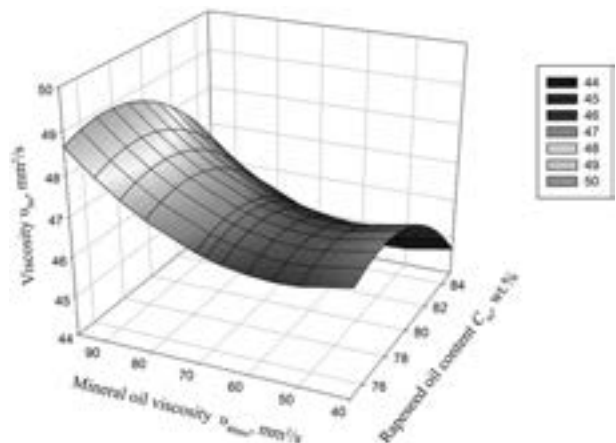


Figure 1 — Dependence of the kinematic viscosity of the base body of the biodegradable oil v_{40bo} on the content of rapeseed oil C_{ro} and the kinematic viscosity of mineral oil v_{40mo} with the content of the adhesive additive $C_{aa} = 1 \text{ wt.}\%$

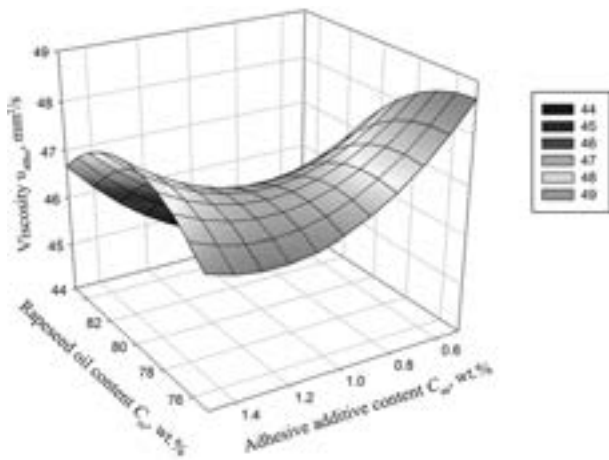


Figure 2 — Dependence of the kinematic viscosity of the base body of the biodegradable oil v_{40bo} on the content of rapeseed oil C_{ro} and the adhesive additive C_{aa} with the kinematic viscosity of mineral oil $v_{40mo} = 68 \text{ mm}^2/\text{s}$

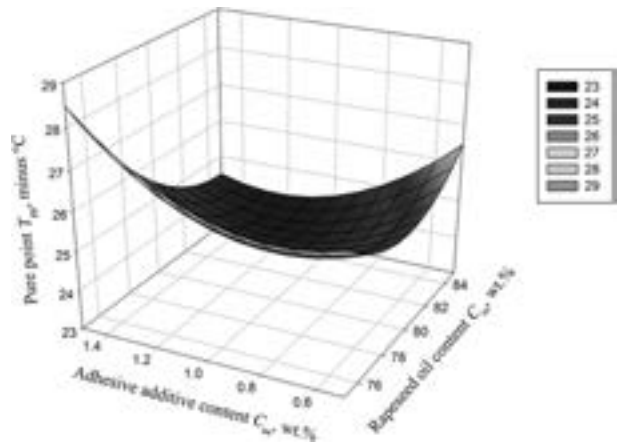


Figure 5 — Dependence of the pure point of the base body of the biodegradable oil T_{pp} on the content of rapeseed oil C_{ro} and the adhesive additive C_{aa} with the kinematic viscosity of the mineral oil $v_{40mo} = 68 \text{ mm}^2/\text{s}$

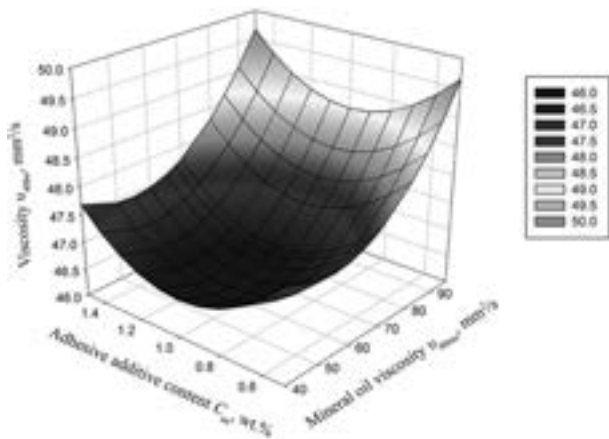


Figure 3 — Dependence of the kinematic viscosity of the base body of the biodegradable oil v_{40bo} on the content of the adhesive additive C_{aa} in it and the kinematic viscosity of the mineral oil v_{40mo} with the content of rapeseed oil $C_{ro} = 80 \text{ wt.}\%$

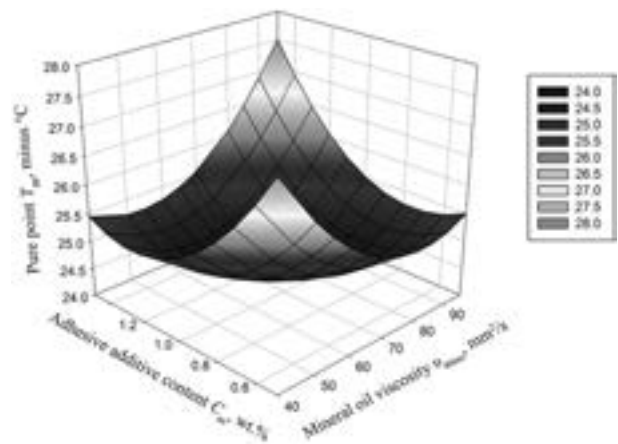


Figure 6 — Dependence of the pure point of the base body of the biodegradable oil T_{pp} on the content of the adhesive additive C_{aa} and the kinematic viscosity of the mineral oil v_{40mo} with the content of rapeseed oil $C_{ro} = 80 \text{ wt.}\%$

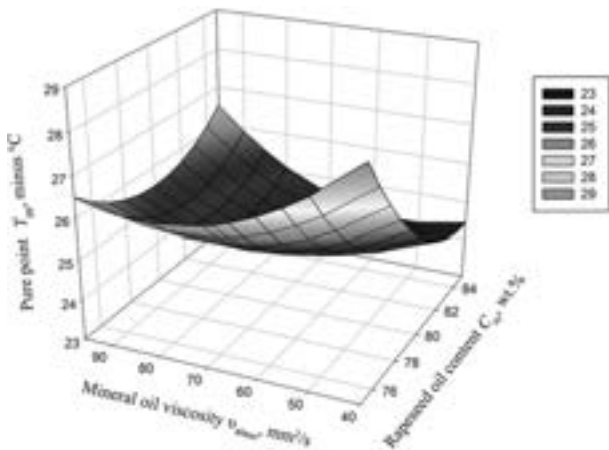


Figure 4 — Dependence of the pure point of the base body of the biodegradable oil T_{pp} on the content of rapeseed oil C_{ro} and the kinematic viscosity of mineral oil v_{40mo} with the content of the adhesive additive $C_{aa} = 1 \text{ wt.}\%$

vegetable oil in the amount of $C_{ro} = 75\text{--}78 \text{ wt.}\%$ with the kinematic viscosity of mineral oil $v_{40mo} = 68\text{--}96 \text{ mm}^2/\text{s}$ and the content of the adhesive additive in the amount of $C_{aa} = 0.5\text{--}1.0 \text{ wt.}\%$, and the lowest

pure points of the base body of the biodegradable oil T_{pp} were found in the samples, the component composition of which corresponds to the content of vegetable oil in them at the level of $C_{ro} = 75\text{--}80 \text{ wt.}\%$ with the kinematic viscosity of mineral oil $v_{40mo} = 40\text{--}68 \text{ mm}^2/\text{s}$ and the content of the adhesive additive in the amount of $C_{aa} = 0.5\text{--}0.6 \text{ wt.}\%$.

If it is necessary to obtain a base body of biodegradable oil with kinematic viscosity at $40 \text{ }^\circ\text{C}$ and pure point that meet the requirements of TU BY 190410065.0181-2019 “Biodegradable all-season oil for saw chains “ECO CS BIO” (kinematic viscosity at $40 \text{ }^\circ\text{C}$ — $35\text{--}55 \text{ mm}^2/\text{s}$, pure point — not higher than $-25 \text{ }^\circ\text{C}$), its formulation, according to the developed experimental and statistical model, should correspond to the following values: the content of vegetable oil $C_{ro} = 75\text{--}80 \text{ wt.}\%$, the kinematic viscosity of mineral oil $v_{40mo} = 45\text{--}55 \text{ mm}^2/\text{s}$, the content of the adhesive additive $C_{aa} = 1.0\text{--}1.5 \text{ wt.}\%$.

Table 4 shows the values of the parameters of the technically optimal version of the formulation of the base body of biodegradable oil for saw chains, which corresponded to the conditions for

Table 4 — Parameters of the technically optimal version of the formulation of the base body of biodegradable oil for saw chains

Parameter	Values of process parameters	
	calculated	actual
Content of vegetable oil C_{ro} , wt. %	77.5±2.5	80
Kinematic viscosity of mineral oil ν_{40mo} , mm ² /s	50±5	48
Content of adhesive additive C_{aa} , wt. %	1.25±0.25	1.5

Table 5 — Controlled parameters of the base body of biodegradable oil for saw chains

Quality indicator	Values of indicators		
	required level	calculated values	actual values
Kinematic viscosity at 40 °C, ν_{40bo} , mm ² /s	35–55	42–53	48–52
Pure point T_{pp} , °C	not higher than –25	–25...–28	–25...–26

obtaining the lubricant when checking the adequacy of the developed experimental and statistical mathematical model.

The controlled parameters of the base body of biodegradable oil for saw chains, obtained by checking the adequacy of the developed experimental and statistical mathematical model, are shown in Table 5.

The analysis of the data given in the Table 5 indicates the adequacy of the developed experimental and statistical mathematical model of the process of obtaining the base body of biodegradable oil for saw chains.

Thus, based on the results of computational and experimental modeling, it can be concluded that to obtain a base body of biodegradable oil for saw chains with kinematic viscosity values at 40 °C and pour point that meet the requirements of TU BY 190410065.018-2019 “Biodegradable all-season oil for saw chains “ECO CS BIO”, its component composition must meet the following conditions:

- content of vegetable oil (C_{ro}) — 77.5±2.5 wt. %;
- kinematic viscosity of mineral oil at 40 °C (ν_{40mo}) — 50±5 mm²/s;
- content of adhesive additive (C_{aa}) — 1.25±0.25 wt. %.

This composition provides the required viscosity, adhesion and low-temperature parameters of the base body.

Development of the component composition of biodegradable oil for saw chains. In order to meet the full range of requirements for the properties of biodegradable saw chain oil, the base body determined

by the experimental statistical mathematical model should be supplemented by the introduction of special additives and possible adjustments to the component composition within the requirements of the experimental statistical mathematical model.

Low-temperature characteristics. Due to the good low-temperature indicators of rapeseed oil and the selected component composition of the base body, it is not necessary to improve the low-temperature properties by introducing depressant additives.

Tribological characteristics. Next, it is necessary to adjust the tribological parameters of the saw chain oil by introducing an anti-wear additive into the base body. At the same time, it should be taken into account that this additive must be quickly biodegradable and not have a toxic effect on microorganisms.

The main tribological characteristics were evaluated according to [16], which applies to liquid and plastic lubricants: load-bearing capacity (according to the critical load P_c), maximum load capacity (according to the welding load P_w), anti-wear properties (according to the wear scar diameter D_w).

To evaluate the tribotechnical characteristics, individual components and a mixture of oils were tested in comparison with the analog oil of the Husqvarna Bio Advanced brand. The test results are presented in Table 6.

From the analysis of the presented data, it can be concluded that the oils for saw chains have sufficiently high tribological characteristics in compa-

Table 6 — Results of testing of lubricants on four ball machine

Load ranges	kgf	63	80	89	100	112	126	150	160	200	224	250	282
Wear scar diameter, mm	Oil И-40А (I-40А)	0.41	2.16	2.30	2.54	2.86	3.00	—	—	—	—	—	—
	Rapeseed oil	0.5	1.1	1.22	1.67	2.11	2.17	2.29	2.38	2.54	2.68	3.00	—
	Husqvarna Bio Advanced Oil	0.4	0.6	1.02	1.48	1.97	2.05	2.08	2.12	2.33	2.53	2.67	3.00
	Developed base body	0.56	1.64	1.85	2.07	2.11	2.12	2.24	2.38	2.83	3.00	—	—

risson with mineral oils. Thus, in the case of Husqvarna Bio Advanced oil, the welding load $P_w = 282$ kgf, and the critical load $P_c = 89$ kgf, while in the case of mineral oil И-40А (I-40A), these indicators had the following values: $P_w = 126$ kgf and $P_c = 63$ kgf. It should be noted that the tribological characteristics of pure rapeseed oil are also quite high ($P_w = 250$ kgf, $P_c = 89$ kgf), but they are slightly lower than the tribological properties of the analog oil. The developed base body due to the mineral oil content has slightly worse tribological characteristics ($P_w = 224$ kgf, $P_c = 63$ kgf). Therefore, it will be necessary to introduce special biodegradable additives to improve the tribological characteristics.

Since most traditional anti-wear additives are complex chemical compounds based on zinc, lead, phosphorus and sulfur, such compounds are usually carcinogenic. In this case, the biodegradable lubricant OIMOL KSC BIO was used as a biodegradable tribological additive [27, 28].

This lubricant is obtained by thickening vegetable rapeseed oil with a complex calcium sulfonate dispersion, which is a separate micromycell with an individual nanoscale core consisting of several salts and a stabilizing shell of amphiphilic liquid crystal polymers that form a macromycellar three-dimensional structure. Thanks to the amphiphilic polymer shell, an affinity with vegetable oil is provided, which is significantly more polar than mineral oil. Due to this, such lubricants provide a good thickening effect in vegetable oils and high stability of properties. When the grease is thoroughly dispersed in the medium of the base body, the three-dimensional structure of the thickener is destroyed and it breaks down into individual associates of micromycells in the activated state. These micelles have good sedimentation stability in vegetable oil, and are also characterized by excellent anti-wear properties characteristic of ultra-alkaline calcium sulfonates.

In order to determine the optimal content of this additive, which ensures the tribological properties of a mixture of rapeseed and mineral oils at the level of the best analog oil (Husqvarna Bio Advanced), studies were conducted to assess the anti-wear properties of oils by determining the wear index D_w at a load of 20 kgf and the test duration of 1 h, the results of which are shown in Table 7.

Table 7 — Anti-wear characteristics of oils and their mixtures

Material	Additive content, wt.%	Wear indicator D_w , mm
Oil И-40А (I-40A)	—	0.65
Rapeseed oil	—	0.49
Husqvarna Bio Advanced oil	—	0.41
Developed base body with different additive OIMOL KSC BIO content	1.0	0.47
	2.0	0.39
	3.0	0.42

Analysis of the data in Table 7 indicates that the introduction of 2.0 wt.% OIMOL KSC BIO lubricant to the developed base body made it possible to achieve the required tribological characteristics of the lubricant composition. The content of this additive in an amount of less than 2 wt.% does not provide high anti-wear performance of the oil mixture, obviously, due to the lack of continuity of the oil film in the tribocontact zone due to its insufficient thickness, and when the additive content is more than 2 wt.%, its effect on the viscosity properties of the oil mixture probably begins to affect due to the thickening qualities of the associates of micromycells of calcium sulfonate lubricant. According to the welding load, this composition also corresponds to the level of values of this indicator for the analog oil.

Stability during storage. For lubricants, especially biodegradable ones, one of the most important indicators that characterize their quality is the preservation of properties during a given storage life and during operation. Therefore, at the next stage of the development of oil for saw chains, the conformance of the resulting product with the requirements for this parameter was checked.

Tests of experimental samples of saw chain oil were carried out in two stages:

- testing of the component composition to meet the requirements for the level of rheological and tribological properties in the fresh state;
- stability evaluation of the properties during storage for 6 and 12 months.

Oil samples of two compositions were made for testing:

- *sample no. 1:* initial base body + anti-wear additive OIMOL KSC BIO – 2 wt.%;
- *sample no. 2:* base body, in which the adhesive additive КП-20 (KP-20) is replaced with the adhesive additive identical in terms of viscosity and temperature characteristics Petrolad 484BD – 1.0 wt.% + anti-wear additive OIMOL KSC BIO – 2 wt.%.

The test results at the first stage are presented in Table 8.

The test results confirmed the compliance of the indicators of fresh samples of oil for saw chains with the requirements of the technological process and technical specifications of TU BY 190410065.018-2019 “Biodegradable all-season oil for saw chains ECO CS BIO”.

Table 8 — Test results for liquid biodegradable lubricants

Sample no.	Storage life, mos.	Density at 15 °C, kg/m ³	Kinematic viscosity at 40 °C, mm ² /s	Kinematic viscosity at 100 °C, mm ² /s	Viscosity index, not less than	Pour point, °C	Flash point, °C	Critical load, N	Welding load, N	Wear index at load of 200 N, mm	Mass fraction of mechanical impurities, wt. %	Water content, wt. %
1	0	920	48	9.8	196	-28	270	872	1,600	0.49	0.017	absent
2		926	49	10.1	201	-28	272	872	1,600	0.39	0.016	absent
1	6	921	51	10.5	201	-28	272	872	1,600	0.48	0.017	absent
2		928	51.5	10.7	204	-28	275	872	1,600	0.37	0.017	absent
1	12	924	53.2	10.9	202	-26	273	872	1,600	0.48	0.021	absent
2		930	54.1	11.4	210	-27	277	872	1,600	0.36	0.019	absent

At the same time, it was found that the change in the amount and brand of the adhesive additive had a slight effect on the oil density, flash point and pour point. The anti-wear characteristics measured on the four ball machine also had almost the same level. The introduction of the adhesive additive had the greatest impact on the viscosity parameters of the oils. The adhesive additive Petrolad 484BD has a greater thickening capacity than the additive КП-20 (КР-20). In particular, with the content of these additives in the liquid lubricant for saw chains in the amount of 1 wt.% the thickening capacity of the Petrolad 484BD additive was 3–4 % higher than that of the КП-20 (КР-20) additive.

After 6 months of storage of both samples of saw chain oil in an open container under the influence of sunlight, the changes in all indicators were within the limits that meet the requirements of the technical specifications, while during the tests after 12 months of storage, changes in the values of some parameters were detected. At the same time, the viscosity characteristics changed most significantly (the kinematic viscosity at 100 °C increased by 2–2.5 mm²/s, the kinematic viscosity at 40 °C — by 5.0–5.5 mm²/s, and the viscosity index — by 5–6 units), and a whitish cloudy precipitate was formed during the storage of

oil samples with the КП-20 (КР-20) adhesive additive. The absence of changes in the water content in the oil samples excludes the whitish precipitation as the cause, that is, allegedly, an insufficient level of their hydrolytic stability. At the same time, the results of IR spectroscopy of the resulting sediment indicate that it is mainly formed by a substance having C—C and C—H bonds, characteristic of the КП-20 (КР-20) adhesive additive, which is a dissolved polyisobutylene. Thus, it can be concluded that the sedimentation instability of these compositions is due to the insufficient affinity of the КП-20 (КР-20) adhesive additive with vegetable (rapeseed) oil and it is more appropriate to use special adhesive additives for vegetable oils in such compositions, in particular, the Petrolad 484BD additive, as evidenced by the test results of the sample no. 2, in which no precipitation was detected.

The absence of changes in the content of mechanical impurities in both samples indicates that during storage there were no chemical transformations in the composition of the complex calcium sulfonate additive and no agglomeration of solid particles.

The change in the viscosity characteristics over time is most likely due to the oxidation of the mineral part of the dispersion medium and

Table 9 — Test results of oil for saw chains with corrected component composition and increased mixing time of components

Oil storage life, mos.	Kinematic viscosity, mm ² /s, at 100 °C	Kinematic viscosity, mm ² /s, at 40 °C	Viscosity index, not less than	Density, kg/m ³	Open cup flash point, °C	Pour point, °C	Mass fraction of mechanical impurities, wt. %	Water content, %
0	9.9	47.3	202	925	282	-29	0.011	absent
6	10.2	48.5	204	925	282	-29	0.011	absent
12	10.5	49.3	209	926	279	-27	0.012	absent

Table 10 — Degree of degradation of predominant fatty acids and biodegradability of the test samples

Test sample	Degree of degradation of fatty acids, %				Degree of degradation under experimental conditions, %	Sample biodegradability, %
	Palmitic	Palmitoleic	Oleic	Linoleic		
Rapeseed oil	71	92	83	93	85	100
Oil for saw chains	84	92	67	62	79	93

the insufficient mixing time of the composition consisting of vegetable and mineral oils. To increase the stability of the viscosity characteristics and sedimentation stability of the saw chain oil, as well as to reduce the overall carcinogenicity of the oil, it is necessary to exclude from the formulation the mineral oil of group I according to the API standard of the И-40А (I-40А) brand, containing a number of chemically active compounds, replacing it with a highly purified oil of group III according to the API standard of the HC7 (NS7) brand, which has identical viscosity and temperature characteristics. It is also necessary to increase the duration of preparation of the oil mixture at the cooling stage from 30 to 60 min, using the Petrolad 484BD adhesive additive. The results of testing the oil for saw chains with a corrected composition and an extended mixing time of the components are shown in Table 9.

The degree of biodegradability of rapeseed oil and saw chain oil was estimated by the average degree of degradation of acids with a carbon chain length of C_{16} and C_{18} , which predominate in rapeseed oil, according to the method described in [29, 30]. The results of the studies are presented in Table 10.

Starting from day 21, all samples showed a decrease in pH from 7.0 to 6.0–6.5 due to the accumulation of oxidation products and a decrease in the dose of activated sludge from 4.1 to 1.9–2.1 g/dm³ due to the lack of substrate. Under the test conditions, the degree of degradation of rapeseed oil was 85 %, that of the oil for saw chains was 79 %. With a rapeseed oil degradation rate of 100 %, the biodegradability of the saw chain oil was 93 %. These values meet the requirements for rapidly biodegradable lubricants.

Conclusion. The article presents the results of the development of a rapidly biodegradable liquid lubricant for saw chains. To optimize the base composition according to its viscosity and temperature characteristics, experimental and statistical mathematical modeling was used. At the same time, the kinematic viscosity of the base body at a temperature of 40 °C and its pure point were chosen as optimization criteria, and the following three factors were used as optimization parameters: the content of vegetable (rapeseed) oil in the composition, the viscosity of mineral oil at a temperature of 40 °C and the content of an adhesive additive in the base composition. To achieve the required level of tribological properties of the saw chain oil and ensure the stability of the entire required set of its characteristics during storage (at least 12 months)

and operation at a given level of biodegradability (at least 90 %), the component composition of the lubricant composition was adjusted and the duration of mixing of the components was changed. In particular, the biodegradable calcium sulfonate grease OIMOL KSC BIO (TU BY 190410065.023-2021) was selected as an anti-wear additive to improve tribological parameters. To increase the sedimentation stability of the lubricant composition, a special adhesive additive for vegetable oils of the Petrolad 484BD brand was used instead of the КП-20 (KP-20) adhesive additive, and to ensure the stability of the viscosity characteristics of the saw chain oil during its storage and operation, as a mineral component it is recommended to use a highly purified oil of group III according to the API standard of the HC7 (NS7) brand (TU BY 300042199.062-2017) instead of mineral oil of group I according to the API standard of the И-40А (I-40А) brand [10]. The developed lubricant has the following component composition: rapeseed oil — 78.0 wt.%; oil HC7 (NS7) — 19.0 wt.%; Petrolad 484BD adhesive additive — 1.0 wt.%; OIMOL KSC BIO lubricant — 2.0 wt.%. The resulting saw chain oil has the following characteristics: density at 15 °C — 926 kg/m³; kinematic viscosity at 40 °C — 47.3 mm²/s; kinematic viscosity at 100 °C — 9.9 mm²/s; viscosity index — 202; pour point — -28 °C; flash point — 272 °C; critical load — 872 N; welding load — 1600 N; wear index at 200 N — 0.39 mm; mass fraction of mechanical impurities — 0.016 wt.%; water content — absence; biodegradability — 93 %.

The developed biodegradable oil for saw chains has passed several stages of optimizing the component composition and technological modes of its production (working out the base body, ensuring the required level of tribological properties and specified duration of stability of all technical characteristics, evaluating the required level of biodegradability). A cycle of laboratory studies of its rheological and tribological characteristics was carried out. Taking into account these results, TU BY 190410065.018-2019 “Biodegradable all-season oil for saw chains ECO CS BIO” and the technological process of its manufacture were developed. In accordance with these technical specifications and the developed technological process, pilot batches of products in the amount of about 30 t were manufactured and sold to the consumer. The products have passed successful operational tests in the conditions of the State Nature Protection Institution “NATIONAL PARK “BELOVEZHSKAYA PUSHCHA”.

Taking into account the data analysis of the completed complex of scientific and technological studies and the test results, the developed oil for saw chains is recommended for introduction into industrial production.

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ОТРАБОТКА КОМПОНЕНТНОГО СОСТАВА БИОРАЗЛАГАЕМОГО ЖИДКОГО СМАЗОЧНОГО МАТЕРИАЛА ДЛЯ ПИЛЬНЫХ ЦЕПЕЙ

Показана последовательность отработки компонентного состава биоразлагаемого жидкого смазочного материала для пильных цепей. Для оптимизации рецептуры базовой основы с учетом необходимых вязкостно-температурных параметров разработана экспериментально-статистическая математическая модель. При этом в качестве критериев оптимизации выбраны кинематическая вязкость базовой основы при температуре 40 °С и температура ее застывания, а параметрами оптимизации выступали следующие три фактора: содержание растительного (рапсового) масла в композиции, вязкость минерального масла при температуре 40 °С и содержание адгезионной присадки в базовом составе. Для достижения требуемого уровня трибологических свойств масла для пильных цепей и обеспечения стабильности всех его характеристик в период хранения (не менее 12 мес.) и эксплуатации при заданном уровне биоразлагаемости (не менее 90 %) проведена корректировка компонентного состава смазочной композиции. В частности, для повышения трибологических показателей в качестве добавки выбрана биоразлагаемая сульфонат кальциевая пластичная смазка OIMOL KSC BIO. Для повышения седиментационной устойчивости применена специальная адгезионная присадка для растительных масел марки Petrolad 484BD и рекомендовано использовать в качестве минерального компонента высокоочищенное масло III группы по стандарту API. Разработанный смазочный материал имеет следующие характеристики: плотность при 15 °С — 926 кг/м³; вязкость кинематическая при 40 °С — 47,3 мм²/с; вязкость кинематическая при 100 °С — 9,9 мм²/с; индекс вязкости — 202; температура застывания — -28 °С; температура вспышки — 272 °С; критическая нагрузка — 872 Н; нагрузка сваривания — 1600 Н; показатель износа при нагрузке 200 Н — 0,39 мм; биоразлагаемость — 93 %.

Ключевые слова: жидкий смазочный материал, пильная цепь, растительное масло, экспериментально-статистическая модель, реологические и трибологические свойства, стабильность свойств, биоразлагаемость

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