Determination of the Rational Composition of the Additive to Oil with the Use of the Katerynivka Friction Geo Modifier

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ABSTRACT

Studies of the formation of the optimal composition of Tribological additives to the base oil have shown the possibility of improving the characteristics of the tribosystem. It was revealed that this Tribological additive makes it possible to form functional surface layers that provide normal operating conditions for tribological conjugation of parts. Based on the optimization of the technical condition of the Tribological additives, the optimal values of each of its components are obtained. Optimization of the condition provided that the amount of wear should tend to a minimum, and the maximum load and loading of the weld should tend to a maximum. Based on the experimental database on the 4-ball friction machine, an equation is obtained for each of the indicated response functions of the resulting features, based on which, with a desirability value of the order of 1.0, the composition of Tribological additives is obtained: from Katerynivka Friction geo modifiers; Sodium oleate, copper sulfate and phosphor TAT 33. It was found that the adding of TM-3-18k into the oil Tribological additives in the amount of 5.75% under laboratory conditions can reduce the wear rate by 35.1–35.5%, the maximum load also increases by 17.1–17.2%, and the welding load increases by 5.3–8.4%. The analysis of experimental data shows that this Tribological additive can be operated at locally loaded parts of 1035 N, and the peak contact overload is up to 2385 N.

The research results suggest that the proposed Tribological supplement makes it possible to improve the characteristics of the tribosystem. It can be useful to service and trucking enterprises during technical service and for the manufacturing of composite oil.

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1. INTRODUCTION, LITERATURE DATA ANALYSIS AND PROBLEM STATEMENT

Improving the characteristics of the triboshift of systems parts and assemblies of transport vehicles is carried out by selecting wear-resistant structural materials, selecting oils for them and creating favorable friction modes [1]. More intensive research is being carried out on the design of triboshift parts. The occurrence of friction and wear processes during operation to a large extent depends on the properties of the lubricating medium and the anti-friction films formed on the working surfaces of the films.

In practice, organometallic materials are widely used for the surfaces of mating parts that are operated in contact with high-temperature, abrasive substances. Having high electrical conductivity and thermal conductivity, the working surfaces of the parts have low wear resistance [2]. The high electrical conductivity of the materials of the parts in this case leads to rapid triboelectrization of the surface layers, which create an internal electric field that can affect additives and wear parts. Influence can be both positive and negative. During the operational use of additives, a positive effect is observed. With an increase in the number of wear parts and the activation of additives in the oil, an abrasive begins to play a negative role in friction in tribological conjugation. In the latter case, a rapid drift of wear parts occurs with the work on the surface of the part. In this connection, the wear resistance of the working surfaces of the interface parts decreases.

The elimination of this drawback is the formation of anti-friction layers with high performance properties on the working surfaces of heterogeneous tribotechnological [3].

Among the methods for increasing the wear resistance of the joints of parts of transport vehicles, they can be divided into three main groups: structural, technological and operational [1]. Therefore, the main attention should be paid to the relationship between the operational and technological groups of oils from the tribotechnological point of view, as well as the assessment of working additives. Structural solutions to increase the wear resistance are laid at the design stage of the mates of parts of power units of machines in general. In this case, it is possible to reduce the cost of friction power by up to 12 %, but the limitation is the complication of their layout, oil oxidation, etc. [4]. In this case, it will be necessary to perform the tasks of selecting technological operations for additives to the oil.

The amount of wear on the working surface of the mates of parts directly affecting the level of mechanical and thermal loads, the type and performance of structural tribojoints [5]. But the issue of managing wear by optimizing the characteristics of the tribosystem has not been resolved in the work.

Working oils from tribological preparations significantly affect the friction surface including the following additives: surface-active; chemically active; inactive; metal-crying and plastic deforming additives [6] and others.

But the technology of their formation and selection in accordance with tribological conditions was not developed.

The operational properties of oils are significantly improved when additives are added to them and the formation of special films or coatings on the friction surfaces of parts [7]. At that time, a method for managing the operational properties and characteristics of the working surfaces of parts was not developed. Under difficult operating conditions of mating parts, controlled wear processes with regeneration of friction working surfaces without additional technical service can be observed [8]. The issue of self-organization has not been resolved in the work; it makes it possible to reduce internal stresses in the contact areas of the tribological link of parts.

The state control of the tribological parts is carried out by introducing oils with additives at a sufficient concentration [9]. But in some conditions, they can even increase friction resistance [10]. In such conditions, it is desirable to perform the selection of the composition of the tribological additives to the oil. Optimum additive concentrations have a positive effect on the rheology of the lubricant; due to the complexity of physicochemical processes and material transformations, there is no universality of additives to materials and operating modes of tribological parts [11].
The nature of tribophysical processes during the operation of mating parts indicates that the formation of wear-resistant coatings increases operational reliability [12]. Under such conditions, it is advisable to use the methods of surface action of laser beams on working surfaces [13] and controlling the stress-strain state of the working surfaces of parts [14], which helps to reduce the intensity of wear processes. But the authors did not consider the possibility of forming renovation coatings from an oil medium, directly increasing operational reliability.

An important finishing technological characteristic is the identification of the rational composition of oil additives under various external influences [15]. It is advisable to identify and establish the optimum with the indicated implementation functions [16] and obtain a sufficient experimental database [17]. But the authors did not use optimization using the desirability function, which will reduce the volume of mathematical tools.

The use of oils with additives at various stages of operation promotes the transition to normal mechanochemical wear of mating parts and the formation of juvenile surfaces capable of absorbing operational loads [18]. But it was necessary to solve the problem, reducing the tribophysical characteristics of moving mates during various friction modes. The latter stabilizes the technical condition, composition, and operational properties of the friction surfaces of parts by their running-in [19]. But the author did not consider the working surfaces of parts in various environments and friction modes. The development of tribotechnology makes it possible to form new wear-resistant structures in the surface layers of tribological parts [20]. The course of the friction process in tribological conjugates with composite oil determines the formation of local regions with better physicochemical properties in the same way as when treated with a concentrated energy flow [21]. In this work, the mechanism of tribological activation of local areas of the mating surfaces of parts, as well as the improvement of their quality during operation with the help of oil additives, is not considered.

The reason for such reactions is primarily the transfer of matter electrically charged components. If you select additives or electrolyte and activate the working surface of the part, you can achieve targeted delivery of wear-resistant components to it [23]. But it is neither described nor considered reactions governance through changing the composition of the functional elements tribological additives during friction.

Reducing the level of wear and improving the reliability of the mating of machine parts is achieved mainly by applying the following methods: precision machining of parts [24] ensuring equilibrium surface roughness [25]; the formation of coatings on the working surfaces of the triboscrew reducing the coefficient of friction [26]; the use of materials with significant cyclic strength, electrical conductivity and damping ability, etc. [27]; development of surface layers creating conditions for parts with normal dissipative and rheological properties [28]. It is possible to realize the above with the introduction of tribotechnologies of running-in and restoration of mating parts in a composite oil medium [29]. But their use was carried out on the basis of synthetic additives without friction geo-identifiers.

For rational and optimal conditions for the functioning of the mates of parts, it is necessary to create a thin coating layer of antifriction materials that promote plasticization and smoothing of micro roughnesses on friction surfaces [30]. The paper did not consider the features of filling the surface layer with a substance and did not optimize the composition of additives. This is achieved using oils with metal-containing and organometallic additives [31], that is, composite oil media. The most widespread among them were organometallic compounds of copper and molybdenum [32]. These additives have a low displacement value, and therefore it would be desirable to identify the effect of these additives on the internal stresses in the parts. A comprehensive solution to the problem can be achieved with an analytical approach and decomposition of the general problem into simpler research points [33]. At the same time, the general synthesis of data will illuminate the full research picture. A malfunction of the cylinder-piston group of transport vehicles causes 3 ... 15 % of failures in the power units of transport vehicles. An analysis of these failures
indicates that the main reasons are: excess load conditions; severe operating conditions; non-compliance with periodic maintenance of the lubrication system and the use of unsuitable oils to the operating conditions [34].

But the work did not address the issue of changing the technical condition of the mates of parts from geo modifiers and their effect on wear processes. An important criterion for the implementation of tribological technologies for the formation of additives is their automation both in the manufacture of systems and units of transport vehicles, and during their operation. One of the key areas at this time is hydraulic elements operating on the sticking effect of the working fluid stream [35]. But in these elements it is desirable to solve the issue of dynamic mixing of mixtures and the possibility of temperature regulation in the tribosystem.

The stress-strain state can also manifest during friction due to various methods. Methods of acoustic emission and coercive force on the working surfaces of parts deserve attention [36]. Identification of this condition was carried out in places of maximum wear, which did not make it possible to assess the overall picture of the state of wear of triboelement. Additional values of wear in the mating parts are carried out by abrasive particles, which form local compression zones in the contact zone, which are further stress concentrators [37]. But the work did not reveal the causes of the occurrence of the elements of the particles of abrasives or they arise from tribological additives, or from synthetic additives of surface substances. The formation of these abrasive particles is possible due to the clustering of wear particles and paint and varnish inclusions in the working oil, which in turn is unacceptable during the operation of transport vehicles [1]. The formation of wear-resistant surface layers with favorable rheological properties is carried out to reduce the internal stress-strain state of machine parts. This is possible with a rational composition of slurry or composite lubricating media [12]. At the same time, it is necessary to identify the patterns of the effect of additives from composite oil on the working surface of parts and on the nature of their wear. In some conditions, it can provide a significantly lower temperature of welding in the Tribological contact [38], but mathematical models of wear in the presence of additives to the oil should be described.

The solution to the problem of increasing the durability of the tribo-link of parts of systems and units of machines requires an integrated approach and consideration of each element of the tribo-link. This positively affects the accuracy of determining the characteristics of the working surfaces of parts. The formation of surface layers with tribological additives on the working surfaces of parts under operating conditions will require most of the power units of the machines. There are practically no studies on the dynamics of coating formation; they change the wear resistance of triboelements in a composite oil environment.

Tribotechnologies have a significant advantage in the formation of the necessary set of operational characteristics and properties of the formed surface layers of parts. It is possible to ensure the equidistance of the working surfaces of the mating parts and to create conditions for the implementation of certain processes and states of self-organization of the tribal link both in the initial period of operation of the mates and during further operation. This ensures minimum wear of the parts of the mates, an increase in their wear resistance, which is certainly an urgent problem.

2. THE PURPOSE AND OBJECTIVES OF THE STUDY

The aim of the work is the formation and optimization of a composite additive to working oil and determining the rational values of its components, which allows improving the physical and mechanical characteristics of tribological conjugations.

Research Objectives:
- substantiate the composition of the proposed composite additives to the working oil and determine the rational and optimal values of its components;
- to establish the boundaries of the operational effects of the composite additive in the laboratory for the triboshift samples.
3. MATERIALS AND METHODS

From a practical point of view, it has been established that an increase in the quality of oil is possible due to the formation of functional additives in it. Also, these findings provide an opportunity for the development of composite additives; qualitative composition is being formed. For each operating conditions of the oil its own modes are formed. For these modes, it is also advisable to conduct tribological studies on the compatibility of additives and their effectiveness. As the base oil used transmission oil TM-3-18k: transmission oil, corresponds to the viscosity class SAE 80W-90 and API GL-3; flash temperature in open crucible +220 °С; thickening temperature -30 °С; density at 20 °С – 0,91 g/cm³.

The main components for the composite additive were selected: Geo modifiers friction KG MF-1, is a dispersion powder of clay from the Katerynivka field; Sodium Oleate Copper sulfate Phosphor TAT 33. Each of these components plays a significant role during friction. KG MF makes it possible to activate the working surface of metal samples and forms a ceramic coating on the working surfaces of friction, as well as a gel component that reinforces with its particles, which can be created from sodium oleate in the surface layer. Also, its parts are partially deposited on the surfaces of metal samples and serve as solid lubricants in extreme friction conditions. The phosphor TAT 33 reduces the formation of scoring during activation of KG MF-1. The amount of each element of the composition of the additive was selected empirically. KG MF-1 preliminarily passes the stage of cleaning and grinding. Cleaning takes place by soaking a crude powder of 300 g / l in distilled water and stirring for 30 minutes at 300 rpm by the homogenizer HG-15A Fig. 1.

After this, the solution is left for 3 minutes, so that heavy parts of abrasive impurities settle. The upper layer 95 % of the volume of the resulting solution was poured and subjected to evaporation at a temperature of 100 °С.

The next step is the grinding of the elements of the additive, which takes place using electric grinder rice. 2. Grinding is performed for 35 minutes for each serving of powder (50 g).

After completing the grinding operation, the powder was sieved on a laboratory sieve to obtain a fraction no larger than 100 microns in size, and the additive parts practically did not experience the phenomenon of sedimentation. For sieving, a GB / T6003.1-2012 laboratory sieve with a mesh size of 74 μ was selected (Fig. 3).

Sifting fractions of the powder material further go through the stage of formation of the composite additive. Weighing of each element of the additive is performed on a TBE-0.21-0.001 balance; the second accuracy class is DSTU EN 45501 Fig. 4.

Preliminary selection of the weight of the constituent units of the composite additive was
performed in laboratory conditions: base oil 50 ml, KGMF-1 (1500 – 3000 mg), sodium oleate (500 – 2000 mg), copper sulfate (500 – 2000 mg) phosphor TAT 33 (500 – 2000 mg).

A liquid dispersion was formed using a mixing homogenizer for each sample for 20 min at room temperature, the number of revolutions was increased every 1000 min by 1000 rpm, the initial value of the homogenizer revolutions was 1000 rpm. The formation of the working oil was carried out with stirring 50 ml of a composite additive with 1 l. Oil mixing was performed with a homogenizer for 10 minutes at 1000 rpm.

Through the point contact of the 4-ball friction machine, the friction machine (Fig. 5) serves as a reliable tool for determining the lubricating ability of base oils and their compositions, the effectiveness of additives and additives to lubricants.

As comparative studies, tests were carried out on oil compositions TM-3-18k (API GL-3) + Liquid Moly Getriebeoil-Additive and TM-3-18k (API GL-3) + an additive was formed.

The 4-ball friction machine is a friction machine in which olives were tested on a tribojoint in a pyramid of four balls, of which the three lower balls are fixed motionless in a cup where the test oil is poured, and the upper ball rotates in a vertical spindle. The 4-ball friction machine was used to study the lubricity of composite lubricants [2].

![Fig. 5. The 4-ball friction machine.](image)

During the study, we determined the averaging of the critical load value, the welding load, the wear indicator, characterized by the diameter of the wear spot from the applied critical load for the corresponding lubricating media. Tests and characterization of lubricating media on the 4-ball friction machine were carried out in accordance with the same standards of GOST 9490-75, in Germany DIN 51350, in the USA ASTM D2783.

To identify the optimal composition of the additive for oil, it is necessary to optimize its content.

In the course of an active experiment, the effect of the composition of the composite additive on the change of tribological characteristics was investigated. The feedbacks or resulting features during the experiment are: \( Y_1 \) - the amount of wear (mm) → min; \( Y_2 \) - critical loading (H) → max, and the combination of factors and their levels are presented in Table 1.

For the experiment, it was decided to investigate four factors and their two levels. In this case, the number of experiments that must be carried out can be calculated by the formula:

\[
N_e = 2^n, \quad (1)
\]

where \( 2 \) - the number of levels, \( n_f \) - the number of factors.

By the Eq. (1), it was determined that 16 experiments are needed to solve optimization problems. We will formulate a plan for a full-factor experiment with an indication of levels and factors, as well as the response functions of the experiment. In order to reduce the effect on the response results, experiments must be performed randomly.

| Factors               | Levels          |
|-----------------------|-----------------|
|                       | Lower (-1)     | Upper (+1)    |
| X1 – KGMF-1, mg/50ml  | 1500            | 3000          |
| X2 – sodium oleate, mg/50ml | 500            | 2000          |
| X3 – sulfate copper, mg/50ml | 500            | 2000          |
| X4 – phosphor TAT 33, mg/50ml | 500            | 2000          |

The analysis of this experimental design was performed using portable software (Statistica v.10). Processing the experimental results began with a regression analysis, that is,
they built a model and determined the unknown coefficients of the regression equation:

\[
Y = b_0 + b_1 X_1^2 + b_2 (X_2)^2 + b_3 (X_3)^2 + b_4 (X_4)^2 + b_5 X_1 X_2 + b_6 X_1 X_3 + b_7 X_2 X_4 + b_8 X_1 + b_9 X_2 + X_3 + b_{10} X_4.
\] (2)

It was determined that the effects of the interaction of factors are practically absent, and therefore they were not included in the general form of the model (2). The determination of unknown constant coefficients was carried out by the method of least squares. The coefficients of the resulting model can be obtained by the following formulas:

\[
b_0 = \sum_{i=1}^{N} \frac{Y_i}{N}; \quad b_j = \sum_{i=1}^{N} \frac{Y_i (X_{ij})^2}{N}.
\] (3)

The description of factors and response using the mathematical model (2) is characterized by a determination coefficient, which should be at least 0.95, for a qualitative description of the object of study, this coefficient is calculated by the Eq.4:

\[
R^2 = 1 - \frac{\sigma_y^2}{\sigma_{\hat{y}}^2} = 1 - \left(\frac{\sum_{i=1}^{N} (Y_i - \hat{Y}_i)^2}{\sum_{i=1}^{N} (Y_i - \bar{Y})^2}\right).
\] (4)

Where \(\sigma_y^2\), \(\sigma_{\hat{y}}^2\) is the variance of the regression residues, response; \(Y, \bar{Y}, \hat{Y}\) - is the actual, average calculated value of the response.

The standard error, which characterizes the standard deviation of the studied regression coefficients from the average value, is calculated by the Eq.5:

\[
S_{b_j} = \sqrt{\frac{\sum_{i=1}^{N} (Y_i - \hat{Y}_i)^2}{\sum_{i=1}^{N} (X_{ij} - \bar{X}_j)^2} \cdot \frac{1}{n-2}}.
\] (5)

where \(n\) - vibration volume.

From a statistical point of view, the importance of regressing health is for Student’s criterion. When you select a calculating value with a table value at a predetermined priority, a value of 0.05 and a rooted level of freedom:

\[
t_{a,f} = \frac{b_j}{S_{b_j}} \geq t_{a/2,f},
\] (6)

where \(b_j\) are the estimated regression coefficients, \(\alpha\) is the confidence probability of 0.95, and \(f\) is the degree of freedom. With a significant regression coefficient, the calculated Student’s criterion is more tabular.

The calculation of the marginal error of deviation was established from the following calculations:

\[
\Delta_f = t_{a,f,\Delta} S_{b_j},
\] (7)

The determination of the confidence interval for each regression coefficient was performed according to inequality:

\[
b_j - \Delta_f \leq b_j \leq b_j + \Delta_f.
\] (8)

Mathematical model fits to the experimental data, that is, its adequacy was determined by the Fisher F test. The calculated criterion should be greater than the tabular one:

\[
F = \frac{\sigma_y^2}{\sigma_{\hat{y}}^2} = \frac{R^2}{1 - R^2} \cdot \frac{f_{2}}{f_{1}} \geq F_{a,f,\Delta},
\] (9)

where \(\sigma_y^2 = \left(\sum_{i=1}^{N} (X_i - \bar{X})^2\right) / f_i\) is the variance of the factor; \(f_i\) is the degree of freedom; \(\sigma_{\hat{y}}^2 = \left(\sum_{i=1}^{N} (Y_i - \bar{Y})^2\right) / N - f_i - 1\) - response variance, \(N\) - number of experiments, \(R^2\) - determination coefficient.

To solve the optimization problem, the method of analyzing the desirability function of E. Harrington was used. This method has useful properties of continuity, monotony, and smoothness. The method recalculates specific parameters into abstract numerical values. The basis of the conversion is using a logical function of the Eq. 10:

\[
d_i = \exp(-\exp(-Y_i)),
\] (10)

where \(Y\) is the response function.

Eq.10 is characterized by two sections of saturation (\(d \rightarrow 0\) and \(d \rightarrow 1\)) and a linear section (\(d \rightarrow 0.2\) and \(d \rightarrow 0.63\)). For a better abstract representation of the desirability function, it is necessary to divide it into ranges where the specific values of the desirability scale correspond to the studied indicators. To form multivariate optimization, desirability functions are formed using the following Eq.11:

\[
Z = \sqrt[n]{\prod_{i=1}^{n} d_i}.
\] (11)
Further study of the desirability function should determine the analysis of the studied responses and process factors. The value of the optimum response condition is set by the limits of the desired response level and within these limits the selection of the most suitable values of the factors takes place. Note that the desirability function method is visual.

4. THE RESULTS

On the basis of tribological studies of the composite additive on the four-ball friction machine, the following experimental database was formed (Table 2).

Table 2. Experimental database.

| No experiment (ordinal No experiment conduct) | Factors | Functions (Responses) |
|---------------------------------------------|---------|----------------------|
| X1 | X2 | X3 | X4 | Y1, mm | Y2, N |
| 1 | 2 | 3 | 4 | 5 | 7 | 8 |
| 1(6) | -1 | -1 | -1 | -1 | 0.366 | 844 |
| 2(10) | +1 | -1 | -1 | -1 | 0.332 | 881 |
| 3(15) | -1 | +1 | -1 | -1 | 0.281 | 956 |
| 4(2) | +1 | +1 | -1 | -1 | 0.316 | 896 |
| 5(11) | -1 | -1 | +1 | +1 | 0.297 | 963 |
| 6(13) | +1 | +1 | -1 | +1 | 0.351 | 938 |
| 7(8) | -1 | +1 | +1 | -1 | 0.256 | 1014 |
| 8(3) | +1 | +1 | +1 | -1 | 0.377 | 911 |
| 9(14) | -1 | -1 | -1 | +1 | 0.308 | 892 |
| 10(5) | +1 | -1 | -1 | +1 | 0.328 | 915 |
| 11(16) | -1 | +1 | -1 | +1 | 0.231 | 1033 |
| 12(4) | +1 | +1 | +1 | +1 | 0.322 | 952 |
| 13(9) | -1 | -1 | +1 | +1 | 0.252 | 973 |
| 14(12) | +1 | -1 | +1 | +1 | 0.367 | 931 |
| 15(7) | -1 | +1 | +1 | +1 | 0.227 | 1041 |
| 16(1) | +1 | +1 | +1 | +1 | 0.402 | 925 |

The processing of experimental data using application software makes it possible to automate the calculations according to the formulas given. The regression analysis of the experimental results is shown in Tables 3 and 4. We will not write insignificant coefficients in them.

Table 3. Regression analysis of experimental results for the optimizing trait Y1.

| R²=0.9991 - the coefficient of determination of the regression model of the experimental data | Regression coefficients (values) | Standard error | Student’s coefficient | Significance level p. (p=0.05) | Confidence interval -95% | Confidence interval +95% |
|---------------------------------------------|-------------------------------|---------------|---------------------|-----------------|-----------------|-----------------|
| b0 | 0.53 | 7.32·10⁻¹ | 73.02 | 9.12·10⁻⁸ | 0.52 | 0.55 |
| b1 | -0.57·10⁻¹ | 2.76·10⁻³ | 20.53 | 5.80·10⁻⁶ | -6.38·10⁻³ | -4.96·10⁻³ |
| b2 | -9.45·10⁻¹ | 3.67·10⁻¹ | 26.49 | 1.43·10⁻¹ | -1.04·10⁻¹ | -8.53·10⁻¹ |
| b3 | -8.51·10⁻⁵ | 3.73·10⁻³ | 23.86 | 2.41·10⁻⁸ | -9.43·10⁻⁵ | -7.59·10⁻⁵ |
| b4 | -9.09·10⁻⁵ | 3.57·10⁻⁵ | 25.49 | 1.73·10⁻¹ | -1.10·10⁻⁸ | -8.18·10⁻⁵ |

Table 4. Regression analysis of experimental results for the optimizing trait Y2.

| R²=0.9962 - the coefficient of determination of the regression model of the experimental data | Regression coefficients (values) | Standard error | Student’s coefficient | Significance level p. (p=0.05) | Confidence interval -95% | Confidence interval +95% |
|---------------------------------------------|-------------------------------|---------------|---------------------|-----------------|-----------------|-----------------|
| b0 | 675.60 | 15.75 | 62.90 | 1.3·10⁻¹ | 635.12 | 716.88 |
| b1 | 0.04 | 9.43·10⁻² | 7.67 | 8.78·10⁻⁴ | 0.03 | 0.06 |
| b2 | 0.13 | 7.57·10⁻² | 16.80 | 1.37·10⁻⁴ | 0.11 | 0.15 |
| b3 | 0.09 | 8.14·10⁻² | 11.70 | 8.01·10⁻⁴ | 0.07 | 0.11 |
| b4 | 0.08 | 7.87·10⁻² | 8.23 | 4.37·10⁻⁴ | 0.04 | 0.08 |
| b5 | -1.9·10⁻² | 2.57·10⁻² | -7.32 | 7.43·10⁻⁵ | -2.57·10⁻² | -4.21·10⁻² |
| b6 | 8.99·10⁻² | 2.54·10⁻² | 3.81 | 3.22·10⁻⁵ | -4.66·10⁻² | 1.66·10⁻² |
| b7 | -7.22·10⁻² | 2.69·10⁻² | -2.78 | 0.04 | -4.39·10⁻² | -5.54·10⁻² |
| b8 | -2.43·10⁻² | 2.12·10⁻² | -9.38 | 0.04 | 2.32·10⁻² | -3.1·10⁻² |
| b9 | -2.28·10⁻² | 3.10·10⁻² | -7.87 | 0.04 | 3.18·10⁻² | -1.63·10⁻² |
| b10 | -3.92·10⁻² | 8.19·10⁻² | -15.12 | 2.72·10⁻² | -4.59·10⁻² | -3.26·10⁻² |

To display the experimental results more accurately, the mathematical regression model was complicated to a second order of dependence of factors with their interaction, provided that the determination coefficient should not be lower than 0.95. Analyzing the data of tables 3-4, we can conclude that all the factors included are statistically significant. What is evidenced by the level of their significance in the model describing the process of friction with a composite additive. Substituting the data of the tables in the general form of the regression equations, we obtain:

\[
x_1 = 0.53 - 5.67 \cdot 10^{-4} (x_1)^2 - 9.45 \cdot 10^{-6} (x_2)^2 - 8.51 \cdot 10^{-5} (x_3)^2 - 9.09 \cdot 10^{-5} (x_4)^2 + 9.22 \cdot 10^{-1} x_1 x_2 + 3.67 \cdot 10^{-1} x_1 x_3 + 2.39 \cdot 10^{-1} x_1 x_4 + 2.10 \cdot 10^{-1} x_2 x_3 + 4.43 \cdot 10^{-3} x_2 x_4 + 8.26 \cdot 10^{-1} x_3 x_4
\]

\[
x_2 = 675.60 + 0.04 (x_1)^2 + 0.13 (x_2)^2 + 0.09 (x_3)^2 + 0.06 (x_4)^2 - 1.9 \cdot 10^{-5} x_1 x_2 + 9.89 \cdot 10^{-5} x_1 x_3 + 7.43 \cdot 10^{-5} x_1 x_4 - 2.43 \cdot 10^{-3} x_2 x_3 - 2.28 \cdot 10^{-3} x_2 x_4 - 3.92 \cdot 10^{-3} x_3 x_4
\]

In order to evaluate the adequacy of the models, we performed a varied analysis of experimental data and then we determined the Fisher criterion. Realization of dispersion analysis is shown in Tables 5 and 6.

Table 5. Dispersion analysis of experimental results for optimizing signs Y1.

| Coefficients | Dispersion | Fisher’s Criterion | Significance level p. (p=0.05) |
|--------------|------------|-------------------|-------------------------------|
| b1 | 1.04 · 10⁻⁴ | 141.27 | 7.4 · 10⁻⁵ |
| b2 | 7.7 · 10⁻⁵ | 10.39 | 2.3 · 10⁻⁴ |
| b3 | 2.0 · 10⁻³ | 271.99 | 1.5 · 10⁻⁵ |
| b4 | 2.15 · 10⁻² | 292.53 | 1.02 · 10⁻⁷ |
Table 6. Dispersion analysis of experimental results for optimizing signs $Y_3$.

| Coefficients | Dispersion | Fisher’s Criterion | Significance level $p$, $(p<0.05)$ |
|--------------|------------|--------------------|----------------------------------|
| $b_1$        | 4192.56    | 123.08             | $1.04 \cdot 10^{-4}$             |
| $b_2$        | 6683.06    | 196.20             | $3.34 \cdot 10^{-5}$             |
| $b_3$        | 9555.06    | 280.52             | $1.39 \cdot 10^{-5}$             |
| $b_4$        | 8418.06    | 247.14             | $1.89 \cdot 10^{-5}$             |
| $b_5$        | 1827.56    | 53.65              | $7.43 \cdot 10^{-4}$             |
| $b_6$        | 495.06     | 14.53              | 0.01                              |
| $b_7$        | 264.06     | 7.75               | 0.04                              |
| $b_8$        | 2997.56    | 88.00              | $2.32 \cdot 10^{-4}$             |
| $b_9$        | 2626.56    | 77.11              | $3.18 \cdot 10^{-4}$             |
| $b_{10}$     | 7788.06    | 228.64             | $2.29 \cdot 10^{-5}$             |

From Tables 5 and 6 it can be seen that factors are included in mathematical models (12, 13) adequately describe the process of friction under study with a composite additive, since the significance level $p$, for each factor is below the permissible level.

Visually, we additionally evaluate the residual emissions between the obtained and predicted values according to the obtained models (12, 13). Significant emissions of 10 % of the maximum predicted numbers, from the graph of the mathematical model of the corresponding response function, should not be present in the analyzed experimental data, since this mathematical model will not satisfactorily describe the physical meaning of the studied process. The indicated procedure is graphically presented in Fig. 6.

Fig. 6. Schedule of predicted and observed residuals, as well as estimation of their magnitude: $a$ and $b$ - for the function of the amount of wear; $c$ and $d$ - for the critical load function.

From this graph it is visually evident that there are no significant data emissions, and therefore the mathematical model can be considered reliable.

The response surfaces of the development of the response functions of the test friction process with a composite additive displaying the values of the factors and on the scale of desirability are identical in character and are presented in Fig. 7. The levels of function response on the scale of desirability $Z$ are shown in Fig. 8. Visually, analyzing these graphs can be clearly stated, that the optimal composition of tribological additive is present in the studied ranges of values of
factors $X_1$, $X_2$, $X_3$, $X_4$. Optimization of the composition of the composite additive to the oil, according to the developed model is possible using the function of desirability. The desirability function must be considered within [0 - 1.0]. All results outside do not satisfy the physical meaning of the research task. The breakdown of the desirability scale [0 - 1.0] in relation to various groups of factors is carried out in the range from 5 to 20 % of the change in each level in an automated mode. The lower and upper values of the desirability function are limited by the given levels of factors of Table 1.

Fig. 7. Graphical representation of response surfaces on the scale of desirability of the investigated composite additive: a) dependence of $Z$ on $X_2$ and $X_1$; b) dependence of $Z$ on $X_3$ and $X_1$; c) the dependence of $Z$ on $X_3$ and $X_2$; d) dependence of $Z$ on $X_4$ and $X_1$; e) dependence of $Z$ on $X_4$ and $X_2$; f) the dependence of $Z$ on $X_4$ and $X_3$. 
Fig. 8. Graphic display of response levels on the scale of desirability of the studied composite additives: a) the dependence of $Z$ on $X_2$ and $X_1$; b) the dependence of $Z$ on $X_3$ and $X_1$; c) the dependence of $Z$ on $X_3$ and $X_2$; d) the dependence of $Z$ on $X_4$ and $X_1$; e) the dependence of $Z$ on $X_4$ and $X_2$; f) the dependence of $Z$ on $X_4$ and $X_3$.

To determine the optimal composition of the composite additive, we will establish the boundaries of the studied responses, they will include the entire experimental database, which is available for analysis. Under such conditions, it is possible to find the necessary maximum response values by the desirability function. Let’s consider the indicated for each factor and each response function. The implementation of the procedure for determining the optimization of composite additives is presented in Fig. 9.

From Fig. 9 it can be seen that the optimal variant of the components is at the intersection of the maximum value of the desirability function in the indicated interval of each factor. With this composition of the composite additive, it can be argued that the solution to the applied problem is optimal. The results to evaluate the effectiveness of the additives are summarized in Table 7.
From Fig. 9 it can be seen that the optimal variant of the components is at the intersection of the maximum value of the desirability function in the indicated interval of each factor. With this composition of the composite additive, it can be argued that the solution to the applied problem is optimal. The results to evaluate the effectiveness of the additives are summarized in Table 7.

Table 7. The averaged test results on the 4-ball friction machine for working lubricating media.

| Averages       | Base oil TM-3-18k | Base oil TM-3-18k + Liqui Moly Getriebooil-Additiv | Base oil TM-3-18k + formed additive |
|----------------|-------------------|-----------------------------------------------------|-------------------------------------|
| Wear index, mm | 0.351±0.02        | 0.257±0.01                                          | 0.227±0.02                          |
| Critical load, N | 889±4.0           | 1075±3.0                                            | 1041±5.0                            |
| Welding load, N | 2252±12.0         | 2342±21.0                                           | 2406±21.0                           |

The existing composite additive allows you to increase the quality of the lubricating composition, as shown by the data in Table 7. In laboratory conditions, there is a decrease in the rate of wear in the tribological contact, an increase in the critical load and welding load.

5. DISCUSSION

To prepare a composite additive in oil and to achieve maximum response values during the friction process in the tribosystem, it is necessary to ensure a rational composition of the studied substances (factors) for which the response function has a maximum region beyond the desirability function. The maximum achievable desirability by the experimental database of optimization conditions is 1.0. For this value of desirability, the optimal composition of the composite additive will contain: KGMF-1 in the interval [1500.0 – 3000.0 mg / 50 ml], optimum - 3000.0 mg / 50 ml; sodium oleate in the interval [500.0 – 2000.0 mg / 50 ml], optimum - 2000.0 mg / 50 ml; copper sulfate in the interval [500.0 – 2000.0 mg / 50 ml], optimum - 2000.0 mg / 50 ml; the phosphor TAT 33 in the interval [500.0 – 2000.0 mg / 50 ml], the optimum is 500.0 mg / 50 ml.
The formed composition of the composite oil additive allows increasing the physicomechanical characteristics of the tribosystem. According to the obtained Tribological studies, it is possible to precisely state that the compositional additive in the composition with oil increases its tribological characteristics in comparison with the base oil. In laboratory conditions, a decrease in the wear indicator by 35.1 – 35.5 % was detected, the maximum load increased by 17.1 – 17.2 %, and the welding load increased by 5.3 – 8.4 %. Comparing the characteristics of the existing composite additive with the industrial analogue of Liquid Moly Getriebeoil-Additive gear oil additives, it was found that in laboratory conditions the following relative values were obtained: reduction of wear by 11.2 – 12.1 % and critical load by 3.0 – 3.3 %, and the welding load increased by 2.7 – 2.8 %. Under such conditions, it can be argued that a composite additive is formed that makes it possible to form functional surface layers. These surface layers have local strength and a lower coefficient of friction. For a detailed study of functional coatings with the participation of the prevailing composite additives, it is necessary to conduct spectrometric analysis of the working surfaces of triboelement. The proposed composite oil additive can be used for operation in power reducers with a contact load on the tooth surfaces of up to 1035 N and with a peak overload of up to 2385 N. The most rational for these data can be the main gearboxes of transport vehicles, since they constantly work in conditions of variable cyclic load.

The use of composite additives to gear oils is effective in cases where it is necessary to increase the tribological efficiency of mating parts with minor technological operations and economic costs. This can be observed according to the results of [3-5]. A change in the rheological properties of the existing composite additive was not recorded as, for example, in [10], which is also promising when used with other types of oils. However, it should be remembered that this composite oil is formed for the specified brand of oil. Therefore, when using it with other brands of oils, it is necessary to conduct additional experimental studies of optimizing its composition [2].

6. CONCLUSIONS

1. The rational ranges and the optimal composition of the additive were revealed: oil 50 ml, KGMF-1 - 3000 mg / 50 ml; sodium oleate - 2000 mg / 50 ml; copper sulfate - 2000 mg / 50 ml; the phosphor TAT 33 is 500 mg / 50 ml, which ensures a minimum of wear rate and a maximum of critical load.

2. Introduction to oil TM-3-18k Tribological additives in the amount of 5.75 % in laboratory conditions a decrease in the wear rate by 35.1 – 35.5 %, an increase in the critical load by 17.1 – 17.2 %, and the welding load by 5.3 – 8.4 %. A comparative analysis of the characteristics of the existing composite additive with an industrial analogue of Liquid Moly Getriebeoil-Additives gear oil additives indicates that in laboratory conditions a relative decrease in wear by 11.2 – 12.1 % and critical load by 3.0 – 3.3 % was observed, and the welding load increased by 2.7 – 2.8 %.

3. It was found that during the technical operation of the tribosystem with composite oil with developed composite additive, the maximum load should exceed 1035 N, and the peak contact overload - up to 2385 N.

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