Проведеними дослідженнями формування раціонального складу трибологічно активної добавки до базової оливи показана можливість покращення характеристик трибосистем. Виявлено, що дана трибологічно активна добавка дає можливість формувати трибологічні властивості, що забезпечують нормальні умови експлуатації спряжень деталей трибосистеми. На основі оптимізації технічного стану трибологічно активної добавки отримано раціональні значення кожного з її складових. Оптимізацію проведено за умов, що величина зношування повинна прямувати до мінімуму, а критичне навантаження та навантажування зварювання – до максимуму. На основі експериментальної бази даних на чотирикульковій машині тертя отримано рівняння для кожної з функцій відгуку результуючих ознак. Отриманні регресійні рівняння та значення функції бажаності порядку 0,698 дали можливість визначити склад трибологічно активної добавки: метакаолін, дисперсійний порошок глини з Катеринівського родовища, олеат натрію, гідрооксид літію та сірки. Встановлено, що при використанні отриманої трибологічної активної добавки в лабораторних умовах зафіксовано зменшення зношування зразків на 26,8 %, збільшення критичного навантаження на 17,2 %, збільшення навантажування зварювання на 4,89 %. Аналіз експериментальних даних свідчить, що пропоновану трибологічно активну добавку можливо експлуатувати при локальному контактному навантажуванні в контакті до 1078 Н та при піковому перенавантажуванні до 2372 Н.

Результати досліджень дають підстави стверджувати, що пропонована трибологічно активна добавка дає можливість покращити характеристики трибосистем. Вона може бути корисною сервісним та автотранспортним підприємствам при проведені технічного сервісу та для виготовлення композиційної оливи

Ключові слова: трибологічно активна добавка, геомодифікатор, зношування, сірка, літієві мила, натрієві мила, метакаолін, навантаження зварювання

UDC 629.083
DOI: 10.15587/1729-4061.2019.184496

DETERMINING THE RATIONAL COMPOSITION OF TRIBOLOGICALLY ACTIVE ADDITIVE TO OIL TO IMPROVE CHARACTERISTICS OF TRIBOSYSTEMS

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Received date 15.08.2019
Accepted date 18.11.2019
Published date 26.12.2019

1. Introduction

At present, the characteristics of the tribosystems for transport machines are enhanced by selecting wear-resistant structural materials, choosing oil for them and creating the conditions for liquid lubrication of mated parts [1]. More intense studies in this direction are carried out and significant progress has been made in designing tribojunctions of parts. In turn, the processes of friction and wear in them during the operation are largely dependent on the properties of the lubricating environment and films of antifriction materials formed on working surfaces.

In this case, metal-organic materials of surfaces of mated parts, which are operated under conditions of contact with overheated, abrasive substances, are used. However, having high electrical and thermal conductivity, working surfaces of the parts have low wear resistance [2]. In turn, high electrical conductivity of the materials of parts leads to rapid tribo-electrization of surface layers, which create an internal electric field that can affect the particles of additives and particles of wear. The impact is positive during the operation of the additive, but with increasing the number of wear particles and the use of additives in oil, this effect starts playing a negative role. Under such conditions there is rapid drifting of particles of wear, followed by their operation as an abrasive on the surface of a part. This significantly reduces wear resistance of working surfaces of mated parts.

One of the ways to eliminate this shortcoming is to create non-homogeneous tribotechnologies of antifriction surface layers with high operational properties on the working surfaces of mated parts [3]. Tribotechnologies have a significant advantage of the formation of the required complex of operational characteristics and the properties of surface layers of parts. Their use makes it possible to ensure a rational equidistance of working surfaces of moving making parts. The conditions for the creation and implementation of certain processes and states of self-organization of a tribosystem both during friction in the initial period of mated parts, and during their further operation, are created.

The use of the tribotechnological methods for the preparation of lubricating media makes it possible to reduce the magnitude of wear of working surfaces of parts and increase their wear resistance during different operating modes, which is undoubtedly an urgent problem.

2. Literature review and problem statement

In the general approach, all the methods for increasing wear resistance of mated parts of transport machines as the simplest tribosystems are divided into three main groups: structural, technological and operational [1]. That is why it is desirable to focus mainly on the relations between operational and technological group of oils from the tribotechnological point of view, as well as on assessment of the working additives. Structural solutions on increasing wear resistance are laid at the stage of designing the mated parts of power units of machines in general. At that, it is possible to reduce power consumption at friction up to 12 %, but the restriction is the complication of their composition, oil oxidation, etc. [4]. In this case, it will be necessary to perform the task of selecting technological operations for creation of additives to oil. The magnitude of wear of the working surface of the mated parts is directly influenced by the level of mechanical and thermal loading, the type and performance of mated parts [5]. However, the issue of wear control by optimizing the characteristics of a tribosystem was not resolved.

Working oils with tribopreparations significantly affect the friction surface. The composition of tribological preparations most often includes the following additives: surface-active; chemically active; inactive; metal-melting and plastic-deforming additives [6] and others. At that time, the technology of their formation was not developed and constituents according to tribological conditions were not selected.

The operational properties of oils are enhanced during the introduction of additives into them and the formation of special films or coatings on the friction surfaces of parts [7]. However, it is desirable to develop a method for controlling the physical and mechanical characteristics of the parts. During the operation of mated parts, the conditions with adjustable wear processes and regeneration of working friction surfaces without the additional technical service can be created [8]. The issue of self-organization, which allows reducing internal tensions in the areas of contact of the elements of a tribosystem, was not resolved in this paper.

Significant merit for controlling the states of a tribosystem is the introduction of oils with additives. In turn, it is necessary to consider the conditions under which the effective action of additives lasts at their sufficient concentration [9], while their improperly selected composition can even increase the friction force [10]. For such conditions, it is advisable to perform the correct selection of the composition of a tribological additive. Rationally selected composition and concentration of an additive can positively affect the rheological properties of the lubricant during friction. Due to the complexity of physical and chemical processes and transformations, additives are not universal for the materials of the tribosystem parts and the modes of their operation [11]. The paper does not describe the technology of preparation of additives and the mechanism to increase the wear resistance of mated parts based on them.

When exploring the character and the nature of tribophysical processes in mated parts during operation, it is possible to increase their operational reliability during the formation of wear-resistant coatings [12]. To increase the wear resistance of working surfaces, it is possible to use the methods of surface influence of laser rays on the areas of working surfaces [13]. To reduce the intensity of wear processes, it is advisable to control the stressed-strained state of the working surfaces of parts [14]. The authors did not consider the possibility of forming the renovation coatings from the composite oil medium, which also makes it possible to increase the operational reliability.

An important technological operation of obtaining high-quality working oils for different modes of operation of mated parts is determining a rational composition of additives to oils under variable external influences [15]. It is reasonable in this case to apply the appropriate mathematical apparatus for determining the values of the components to the additive to oil within the applied task when using fuzzy responses [16] and forming a complete experimental database [17]. The desirability function was not used in this research, which makes it possible to reduce the volume of mathematical toolkit.

Application of oils with additives at different stages of operation of machines promotes the transition to the normal mechanic-chemical wear of mated parts and the formation of juvenile surfaces capable of receiving operational loading [18]. However, it was necessary to solve the problem of reducing the tribophysical characteristics of moving mated parts during liquid friction. It stabilizes the technical condition, composition and operational properties of friction surfaces of parts through their alignment [19]. However, alignment of working surfaces of parts in different media was not considered. The development of tribotechnologies makes it possible to form new wear resistant structures in the surface layers of materials of mated parts. Accordingly, during the flow of friction processes in the presence of composition oil, it is possible to observe a similar process of formation of local areas with the best physicochemical properties, which are realized during the treatment by the concentrated energy flow [21]. It is advisable to consider the mechanism of tribological activation of the local areas of the mated parts, as well as to improve their quality during the operation with the use of tribologically active additives.

The methods based on tribochemical reactions occurring under conditions of mechanical activation in the system «metal – composite olive medium» or «metal – electrolyte» are the most promising for tribotechnologies of alignment and recovery [22]. The main cause of such reactions is the substance transfer by electrically charged components. If additives or electrolyte are chosen carefully and working surfaces of parts are activated, it is possible to achieve directed delivery of wear-resistant components to the friction zone [23]. The paper does not describe or explore the issue of controlling the tribochemical reactions by means of the composition of the functional elements of tribologically active additives during friction.

Today, a decrease in the level of wear and enhancement of reliability of mated machine parts are achieved mainly by using different methods. The most available methods are the methods for ensuring the accuracy of machining the mated parts [24] and the equilibrium roughness of surfaces [25], but these methods do not ensure operational and technological requirements for the complete life cycle of tribojunction cycle. Important in this direction is the formation of coatings on the working surface of tribojunction, which decrease the friction coefficient [26] and the use of materials with significant cyclic strength, electric conductivity, damping ability, etc. [27]. Instead, it was necessary to develop algorithms for selection of these coatings for characteristic operating conditions of moving mated parts. Development of surface layers creates the conditions of self-organization for parts with normal dissipative and rheological properties [28]. In this case, it was desirable for the authors to establish regularities of tribological characteristics depending on the presence of surface layers in mated parts. The methods may be implemented with the realization of tribotechnologies of alignment and restoration of mated parts in the composite olive medium [29]. However, these problems were solved exclusively using synthetic additives without friction geomodifiers.

For optimal conditions of operation of mated parts, it is necessary to create a thin layer of coating of antifriction materials on their working surfaces that promote plasticizing and smoothing of micro roughness on friction surfaces [30]. At the same time, the peculiarities of the processes of filling the surface layer and optimizing condition of additives was not considered. This is achieved with the use of oils with metal-containing and metal-organic additives [31], that is, composite olive media. The most widespread among them are metal-organic compounds of copper and molybdenum [32]. These additives have low magnitude of displacement, and in this connection it would be desirable to identify the impact of these additives on internal stresses in detail. It is possible to achieve comprehensive consideration of the problem with the analytic approach and decomposition of the general task into simpler research points [33]. In this case, the general synthesis of the data will make it possible to present a full picture of research. The malfunction of a cylinder-piston group of transport machines causes from 3...15 % failures of power units of transport machines. The analysis of their failures shows that the main reasons are as follows: exceeding the load conditions; severe operating conditions; failure to observe periodic maintenance of lubrication system and the use of oils that are unsuitable to the operating conditions [34]. In addition, it is necessary to consider the issue of changes in the technical state of mated parts with geomodifiers and their impact on wear processes. An important criterion of implementation of tribotechnologies of formation of additives is their automation both in the manufacture of systems and aggregates of transport machines and during their exploitation. One of the key areas at present is the hydro-elements operating on the effect of sticking of a stream of the working medium [35]. The use of these elements makes it possible to distribute hydraulic flow, as well as to redirect it to different technological operations, which is extremely important during the formation of composite oils [36]. At the same time, in these elements, it was desirable to solve the problem of dynamic stirring of mixtures and the possibility of temperature regulation in a tribosystem.

The stressed-strained state is also possible to detect at friction by various non-destructive methods, namely, the methods of acoustic emission and a coercive force on working surfaces of parts [37]. The areas of stressed-strained state were found in the places of maximum wear, but to analyze the complete pattern of parts' wear, it is also necessary to study the area of minimum wear. In addition, the magnitude of wear in mated parts is affected by the abrasive particles, which getting into the tribological contact, in turn, form the local compression zones that are the concentrators of stress during operation [38]. It was not found in the work if this process flows with participation of the elements of a tribologically active additive or synthetic additive based on surfactants. The formation of these abrasive particles is possible due to clustering wear particles and paint-and-varnish inclusions in the working oil, which in turn is unacceptable during the operation of transport machines [1]. The formation of wear resistant surface layers with favorable rheological properties is carried out to reduce the internal stressed-strained state of machine parts. This is possible at the rational composition of the hydro mixture or composite lubricant media [39]. The first step is to determine the regularities of the influence of additives from the composition oil on the working surface of parts and wear of the mated elements. The latter under some conditions can provide much lower welding temperature in tribological contact [40]. For further research in this direction, it is necessary to describe additionally the mathematical models of wear in the presence of active additives to oil.

It is possible to increase the effectiveness of tribological junctions by selecting rational materials, such as polymer composites. Instead, it is advisable to detect tribological regularities of polymeric composites under conditions of liquid lubrication with composite oil [41]. The tribosystems with hydrodynamic lubrication have better tribological characteristics, but they should be evaluated [42]. The paper did not reveal any regularities depending on a change in friction coefficient with functional additives in oil. It is possible to ensure oil resistance in heavily loaded tribosystems through the use of oils based on stearic acid and isocyanine [43]. It is necessary to additionally establish the effect of these types of lubricants on the operating additives. The functioning of additives at different stages of operation can be estimated by assessing the quality criterion of a tribosystem [44], but it is necessary to develop additionally an algorithm of recommendations for the selection of functional additives.

The solution of the problem of improving the durability of tribo-elements requires a comprehensive approach and consideration of each element of the tribojunction, which positively influences the accuracy of determining the characteristics of working surfaces of parts. Most power units of machines require the studies of the formation of surface layers with tribologically active additives on working surfaces of parts under operating conditions. There are virtually no studies of the dynamics of formation of coatings that purposefully change the durability of mated parts in the composite olive medium.

3. The aim and objectives of the study

The aim of this study is to form a tribologically active additive to the working oil and to determine its rational composition, which makes it possible to improve the physical and mechanical characteristics of a tribosystem.

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

– to propose the components of a tribologically active additive and coordinate their content for obtaining minimal values of the magnitude of wear, as well as maximum values of critical load and welding load of mated parts of the «ball – ball» type;

– to set boundary values of the operational action of a tribologically active additive under laboratory conditions for a tribosystem «ball – working oil – ball», specifically, critical load and welding load in the contacts of a tribosystem.

4. Materials and methods for the formation and research into characteristics of the tribological additive to oil

In practical terms, it was determined that an increase in the quality of oil is possible due to the introduction of functional active additives in it. Under such conditions, the development and the use of tribologically active additives makes it possible to form working oils for different conditions and operating modes by means of their introduction to oil. It is also advisable to conduct tribological studies on the compatibility of additives and their effectiveness for these modes. Transmission oil Grom Ex QUATTRO API GL-5 80W-90 was used as the basic oil.

The following components were chosen as basic components for the tribologically active additive: metakaolin MK-40; dispersive clay powder from Katerynivka deposit; sodium oleate; lithium hydroxide; sulfur. Each of these components plays a significant role during friction. Metakaolin makes it possible to activate the working surface of metallic samples and perform local charging in the surface layers of the samples. The dispersive clay powder from Katerynivka deposit forms ceramic coating on working friction surfaces and inclusion in gel components that can be formed from sodium oleate and lithium hydroxide. Its particles are partially deposited on the surfaces of the metal samples and serve as solid lubrication in the boundary friction modes. Sulfur reduces the formation of chippings during the metakaolin activation. The amount of each element of the composition of the additive was selected empirically. Metakaolin and dispersive powder of Katerynivka deposit previously underwent cleaning and crushing. Cleaning is performed by soaking untreated powder of 300 g/l in distilled water and stirring for 30 minutes at 300 rpm by the HG-15A homogenizer.

After this, the solution was left for 3 minutes, so that heavy particles of silicon-containing impurities should settle. The top layer of 95 % of the volume of the obtained solution was poured out and was subjected to evaporation at the temperature of 100 °C.

The next stage is crushing the elements of the additive, which passes using an electric crusher. Crushing was performed for 35 minutes for each portion of the powder (50 g).

After the grinding operation had been finished, the powder was sifted on a laboratory sieve to get a fraction of not more than 100 microns, so that the particles of an additive should not have the phenomenon of sedimentation. The laboratory sieve GB/T 6003.1-2012 with a cell size of 74 microns was selected for sifting.

Sifted fractions of the powdered material subsequently go through the stage of the formation of tribological additive. Each element of the additives is weighted on scales TBE-0.21-0.001, the second class of precision of DSTU EN 45501.

Preliminary selection of the weight of the components of the additives was carried out under laboratory conditions: basic oil 50 ml, metakaolin (50...550 mg), dispersive clay powder of Katerynivka deposit (250...750 mg), sodium oleate (650...1,150 mg), lithium hydroxide (50...550 mg), and sulfur (50...550 mg).

The liquid variance of the samples of the tribologically active additive was formed by means of the homogenizer by stirring each sample. Stirring was performed within 20 minutes at room temperature, the number of revolutions was increased every 5 minutes by 1,000 rpm, the initial value of the revolution of homogenizers was 1,000 rpm. Working oil was formed at stirring of 50 ml of the tribological additive from 1 l of fresh oil, stirring was performed by the homogenizer for 10 min homogenizer at 1000 rpm.

Through the point contact of the samples, the four-ball friction machine serves as a reliable tool to determine the lubricating ability of basic oils and their compositions, the effectiveness of additives to lubricants.

The experiments were carried out on the oil composition GL-5 80W-90+, the additive was formed. We used the four-ball friction machine, in which the oil media of mated were tested in a pyramid of four balls, three lower balls of which were fixed motionless in the cup, where the tested oil was poured, and the top ball rotated in a vertical spindle. The four-ball friction machine was used to study the lubricating capacity of composite lubricants [2]. Average values of critical load, welding load, wear indicator, characterized by the diameter of a spot from wear from the applied critical load for the corresponding lubricating media were determined in the course of research. The characteristics of the lubricating media on a four-ball friction machine were tested and determined in accordance with the unified standards GOST 9490-75, in Germany DIN 51350 standards, in the USA – STM D2783.

To identify the optimum values of the composition of a tribological additive, it is necessary to optimize its composition.

In the process of the active experiment, the influence of the composition of the additive on the change of tribological characteristics; their factors and levels are shown in Table 1. Responses or resulting features during the experiment were: $Y1$ – magnitude of wear (mm) $\rightarrow$ min; $Y2$ – critical load (N) $\rightarrow$ max; $Y3$ – welding load (N) $\rightarrow$ max.

Table 1

Formation of factors and their levels for the experiment

Factors	Levels	
	Lower (–1)	Upper (+1)
$X1$ – content of metakaolin, mg/50 ml	100	500
$X2$ – content of dispersive powder of clay of Katerynivka deposit, mg/50 ml	300	700
$X3$ – content of sodium oleate, mg/50 ml	700	1,100
$X4$ – content of lithium hydroxide, mg/50 ml	100	500
$X5$ – content of sulfur, mg/50 ml	100	500

The preliminary choice of factors' levels was made based on the formalization of a priori information of the series of experiments [1, 2, 6, 23]. The values of the lower factor were determined according to their impact on the corresponding response function or the possibility of manifestation of the negative processes in the junction during of the friction process. The metakaolin content of less than 100 mg/50 ml contributes to a significant decrease and unstable obtaining the values of such response function as welding load. The content of the dispersed clay powder of Katerynivka deposit is less than 300 mg/50 ml contributes to a significant decrease and unstable obtaining the values of magnitude of wear and critical load in the tribological contact. The content of sodium oleate in the amount of 700 mg/50 ml is characterized by cleaning the local working surface and the formation of a better access of the particles of metakaolin and of dispersive powder of Katerynivka deposit to it. The content of lithium oxide in the amount of less than 100 mg/50 ml leads to the intensification of the phenomenon of sedimentation of metakaolin particles and dispersive clay powder. The content of sulfur in the amount of 100 mg/50 ml is characterized by sufficient chipping resistance, which is well recommended at the stabilization of wear magnitude in the initial start modes in the presence of particles of metakaolin and dispersive powder. The values of the upper levels of factors were determined according to their influence and restrictions on the operating oils. The metakaolin content is limited due to its considerable surface activity, that is, at the content of more than 500 mg/50 ml of oil, it significantly affects the synthetic surface-active additives of basic oil, thereby reduces its viscosity under minor temperature conditions. The content of dispersive powder in the amount of 500 mg/50 ml of oil is limited due to its high ability to clustering, which in turn contributes to the formation of additional wear particles. The content of sodium oleate in the amount of 1,100 mg/50 ml is limited due to its highly emulsifying and detergent capacity. At greater values of the content of sodium oleate in the oil, the formation of a water-oil emulsion is intensified, which leads to the intensification of corrosion wear. In addition, the content of sodium oleate that is more than this contributes to washing the working oil off the friction surface, which in turn creates the conditions of boundary lubrication. The content of lithium oxide in the amount of 500 mg/50 ml is limited because of its high thickening capacity, which contributes to an increase of density of working oil even at the temperature of about 0° C. The sulfur content of 500 mg/50 ml is limited due to the intensity of the oil oxidation process. The average value of the factor between its initial and critical impact values of the influence on any response function was taken as the basic level of the factor.

To conduct the experiment, it was decided to explore five factors and their two levels. In this case, the number of experiments that should be conducted can be calculated from formula:

$$N_e = 2^{n_f}, \tag{1}$$

where 2 is the number of levels, n_f is the number of factors.

From formula (1), it was determined that 32 experiments were needed to solve the optimization tasks. The plan for the full-factorial experiment indicating the levels and factors, as well as response functions of the experiments, was formed. Experiments must be performed in a random sequence in order to reduce the impact on the results of response function.

Analysis of this plan of the experiment was performed using portable software (Statistica 10.0.1011.0, CD-key 42347678921334567692). The experiment results were processed based on regression analysis, that is, a mathematical model was constructed and unknown coefficients of the regression equation were determined:

$$\begin{aligned} Y &= b_0 + b_1 \cdot X1 + b_2 \cdot X2 + b_3 \cdot X3 + b_4 \cdot X4 + b_5 \cdot X5 + \\ &+ b_6 \cdot X1 \cdot X2 + b_7 \cdot X1 \cdot X3 + b_8 \cdot X1 \cdot X4 + b_9 \cdot X1 \cdot X5 + \\ &+ b_{10} \cdot X2 \cdot X3 + b_{11} \cdot X2 \cdot X4 + b_{12} \cdot X2 \cdot X5 + \\ &+ b_{13} \cdot X3 \cdot X4 + b_{14} \cdot X3 \cdot X5 + b_{15} \cdot X4 \cdot X5. \end{aligned} \tag{2}$$

The type of this model is characterized by the existence of linear and paired effects based on analysis of the obtained experimental data and the possibility of automated mode of the construction of the extended matrix of the experiment matrix.

It was determined that the effects of the interaction of the factors of higher levels are almost absent, and that is why they were not included in the overall form of the model (2). The unknown constant coefficients were determined using the least squares method. The coefficients of the resulting model were calculated from the following formulas:

$$b_0 = \sum_{i=1}^{N} Y_i / N; \;\; b_j = \sum_{i=1}^{N} Y_i / N; \;\; b_{j^2} = \sum_{i=1}^{N} Y_i (X_{ji})^2 / N. \tag{3}$$

The description of the factors and response using mathematical model (2) is characterized by determination factor, which should be not less than 0.95 for a qualitative description of the object of study, this factor is calculated from formula:

$$R^2 = 1 - \frac{\sigma_{zl}^2}{\sigma_Y^2} = 1 - \left(\sum_{i=1}^{N} \left(Y_i - \hat{Y}_i \right)^2 \Big/ \sum_{i=1}^{N} \left(Y_i - \bar{Y}_i \right)^2 \right), \tag{4}$$

where σ_{zl}^2, σ_Y^2 are the variances of residues of regression, response; Y_i, $\bar{Y}$, $\hat{Y}_i$ are the actual, mean, calculated value of response.

The standard error that characterizes the standard deviation of the studied regression coefficients from the mean value is calculated from formula:

$$S_{b_{j^r}} = \sqrt{\frac{\sum_{i=1}^{N} \left(Y_i - \hat{Y}_i \right)^2}{\sum_{i=1}^{N} \left(X_{ij} - \bar{X}_j \right)^2} \cdot \frac{1}{n-2}}, \tag{5}$$

where n is the volume of the sample.

From the statistical point of view, the values of regression coefficients are estimated according to the Student criterion. At the same time, the calculated value was compared with the tabular value at the assigned confidence level of significance of 0.05 and the calculated degrees of freedom:

$$\left| t_{\alpha,f} \right| = \left| \frac{b_{j^r}}{S_{b_{j^r}}} \right| > t_{\alpha/2, f_{tb}}, \tag{6}$$

where b_{j^r} is the estimated regression coefficient, α is the confident probability 0.95, f is the degree of freedom. At significant regression coefficient, the calculated Student criterion is more than the tabular one.

The calculation of the boundary error of deviation was established from the following formula:

$$\Delta_{j^r} = t_{\alpha,f.tb} \cdot S_{b_{j^r}}. \tag{7}$$

Confidence interval for each regression factor was determined according to inequality:

$$b_{j^r} - \Delta_{j^r} \le b_{j^r} \le b_{j^r} + \Delta_{j^r}. \tag{8}$$

Conformity of the mathematical model with the experimental data, that is, its adequacy was determined by the Fischer criterion F. In this case, the calculation criterion should be higher than the tabular one:

$$F = \frac{\sigma_X^2}{\sigma_Y^2} = \frac{R^2}{1-R^2} \cdot \frac{f_2}{f_1} \geq F_{\alpha, f. tbl}, \quad (9)$$

where

$$\sigma_X^2 = \left(\sum_{i=1}^{N} \left(X_i - \bar{X} \right)^2 \right) \Big/ f_1$$

is the variance of the factor; f_1 is the degree of freedom;

$$\sigma_Y^2 = \left(\sum_{i=1}^{N} \left(Y_i - \bar{Y} \right)^2 \right) \Big/ N - f_1 - 1$$

is the variance of response, N is the number of experiments, R^2 is the determination factor.

To solve the optimization problem, we used the method of analysis of desirability function of E. K. Harrington. This method has such useful properties as continuity, monotony and smoothness. In this method, the recalculation of specific parameters into abstract numerical values is quite simple. The logical function in the following form is used as the recalculation basis:

$$d_i = \exp\left(-\exp(-Y)_i\right), \quad (10)$$

where Y is the response function.

Function (10) is characterized by two saturation sections ($d \to 0$ and $d \to 1$) and the linear section ($d \to 0.2$ and $d \to 0.63$). For more qualitative abstract representation of desirability function, it is necessary to divide it into ranges where specific values of the desirability scale correspond to the studied indicators. Multi-factor optimization of desirability function was formed from the following formula:

$$Z = \sqrt[n]{\prod_{i=1}^{n} d_i}. \quad (11)$$

Further research into desirability function should determine the analysis regarding the studied responses and factors of the process. The values of the condition of the response optimum were assigned by the boundaries of the desired level of response. Within these limits, the most appropriate values of the factors were selected. Note that the method of desirability function is visual.

5. Results of selection of the rational composition of the tribologically active additive to oil and obtaining the regularities of wear of corresponding industrial additives

Based on the conducted tribological studies of the proposed active additive on the four-ball friction machine, we formed the following experimental database of Table 2, from which the augmented matrix for detection of paired effects is subsequently plotted in the automated mode.

Processing of the experimental data with the use of applied software makes it possible to automate calculations from the given formulas. Regression analysis of the experimental results is reflected in Tables 3–5; insignificant coefficients were not included in the Table 3–5.

Table 2

Experimental database

No. of experiment (number by order of conducting the experiment)	Factors					Functions (Responses)		
	$X1$	$X2$	$X3$	$X4$	$X5$	$Y1$, mm	$Y2$, N	$Y3$, N
1(27)	–1	–1	–1	–1	–1	0.398	991	2,218
2(8)	+1	–1	–1	–1	–1	0.392	979	2,539
3(1)	–1	+1	–1	–1	–1	0.328	942	2,074
4(11)	+1	+1	–1	–1	–1	0.321	1,031	2,424
5(6)	–1	–1	+1	–1	–1	0.367	883	2,078
6(15)	+1	–1	+1	–1	–1	0.351	861	2,421
7(21)	–1	+1	+1	–1	–1	0.328	652	2,185
8(26)	+1	+1	+1	–1	–1	0.332	751	2,505
9(14)	–1	–1	–1	+1	–1	0.227	947	2,284
10(19)	+1	–1	–1	+1	–1	0.256	947	2,367
11(28)	–1	+1	–1	+1	–1	0.231	1,083	2,151
12(27)	+1	+1	–1	+1	–1	0.261	1,204	2,232
13(22)	–1	–1	+1	+1	–1	0.232	919	2,156
14(32)	+1	–1	+1	+1	–1	0.265	901	2,243
15(24)	–1	+1	+1	+1	–1	0.279	871	2,282
16(3)	+1	+1	+1	+1	–1	0.312	971	2,364
17(25)	–1	–1	–1	–1	+1	0.551	937	2,285
18(30)	+1	–1	–1	–1	+1	0.522	907	2,361
19(12)	–1	+1	–1	–1	+1	0.481	871	2,158
20(29)	+1	+1	–1	–1	+1	0.471	978	2,243
21(10)	–1	–1	+1	–1	+1	0.524	965	2,187
22(13)	+1	–1	+1	–1	+1	0.501	926	2,234
23(17)	–1	+1	+1	–1	+1	0.482	678	2,284
24(31)	+1	+1	+1	–1	+1	0.464	798	2,361
25(29)	–1	–1	–1	+1	+1	0.525	801	2,605
26(16)	+1	–1	–1	+1	+1	0.531	756	2,447
27(5)	–1	+1	–1	+1	+1	0.546	918	2,481
28(5)	+1	+1	–1	+1	+1	0.571	1,005	2,324
29(7)	–1	–1	+1	+1	+1	0.531	869	2,483
30(4)	+1	–1	+1	+1	+1	0.543	839	2,321
31(9)	–1	+1	+1	+1	+1	0.587	812	2,606
32(2)	+1	+1	+1	+1	+1	0.604	885	2,442

Table 3

Regression analysis of experimental results for resultant feature Y_1

R^2=0.9991 is the determination factor of regression model of experimental data						
Regression factors	Regression factors (values)	Standard error	Student's coefficient	Significance level p, (p<0.05)	Confidence interval – 95 %	Confidence interval – 95 %
b_0	0.638	0.032	20.19	$1.0\cdot10^{-6}$	0.56	0.72
b_2	$-4.3\cdot10^{-4}$	$5.3\cdot10^{-5}$	–8.2	$1.72\cdot10^{-4}$	$-5.6\cdot10^{-4}$	$-3.1\cdot10^{-4}$
b_3	$-2.1\cdot10^{-4}$	$3.3\cdot10^{-5}$	6.4	$7.1\cdot10^{-4}$	$-2.9\cdot10^{-4}$	$-1.3\cdot10^{-4}$
b_4	$-8.5\cdot10^{-4}$	$6.6\cdot10^{-5}$	12.9	$1.3\cdot10^{-5}$	$-1.01\cdot10^{-3}$	-6.910^{-4}
b_5	$1.76\cdot10^{-4}$	$6.6\cdot10^{-5}$	2.7	$3.7\cdot10^{-2}$	$1.5\cdot10^{-5}$	$3.4\cdot10^{-4}$
b_{10}	$2.8\cdot10^{-7}$	$5.4\cdot10^{-8}$	5.1	$2.1\cdot10^{-3}$	$1.4\cdot10^{-7}$	$4.1\cdot10^{-7}$
b_{11}	$4.1\cdot10^{-7}$	$9.9\cdot10^{-8}$	4.1	$6.4\cdot10^{-3}$	$1.6\cdot10^{-7}$	$6.5\cdot10^{-7}$
b_{13}	$2.4\cdot10^{-7}$	$6.7\cdot10^{-8}$	3.6	$1.0\cdot10^{-2}$	$8.0\cdot10^{-8}$	$4.0\cdot10^{-7}$
b_{15}	$8.9\cdot10^{-7}$	$1.0\cdot10^{-7}$	8.4	$1.6\cdot10^{-4}$	$6.3\cdot10^{-7}$	$1.1\cdot10^{-6}$

Table 4

Regression analysis of experimental results for resultant feature Y_2

R^2=0.9961 is the determination factor of the regression model of experimental data						
Regression factors	Regression factors (values)	Standard error	Student's coeffi-cient	Significance level p, (p<0.05)	Confidence interval – 95 %	Confidence interval – 95 %
b_0	1145.59	71.06	16.12	$4.0\cdot10^{-6}$	971.71	1319.48
b_1	–0.245	$4.8\cdot10^{-2}$	1.65	$4.9\cdot10^{-2}$	$-6.0\cdot10^{-1}$	$1.2\cdot10^{-1}$
b_2	$4.64\cdot10^{-1}$	0.118	3.9	$7.8\cdot10^{-3}$	$1.7\cdot10^{-1}$	$7.5\cdot10^{-1}$
b_4	$-7.56\cdot10^{-1}$	$1.48\cdot10^{-1}$	5.09	$2.2\cdot10^{-3}$	–1.12	$3.93\cdot10^{-1}$
b_5	$-6.21\cdot10^{-1}$	$1.48\cdot10^{-1}$	4.19	$5.8\cdot10^{-3}$	$-9.8\cdot10^{-1}$	$-2.58\cdot10^{-1}$
b_6	$1.0\cdot10^{-3}$	$2.2\cdot10^{-4}$	3.17	$1.91.0\cdot10^{-2}$	$2.0\cdot10^{-4}$	$1.0\cdot10^{-3}$
b_{10}	$1.1\cdot10^{-3}$	$1.2\cdot10^{-4}$	9.8	$1.0\cdot10^{-4}$	$1.4\cdot10^{-3}$	$1.0\cdot10^{-3}$
b_{11}	$1.08\cdot10^{-3}$	$2.2\cdot10^{-4}$	5.21	$1.9\cdot10^{-3}$	$6.0\cdot10^{-4}$	$2.0\cdot10^{-3}$
b_{13}	$4.61\cdot10^{-4}$	$1.5\cdot10^{-4}$	3.05	$2.2\cdot10^{-3}$	$9.1\cdot10^{-5}$	$8.3\cdot10^{-4}$
b_{14}	$8.5\cdot10^{-4}$	$1.52\cdot10^{-4}$	5.64	$1.3\cdot10^{-3}$	$4.8\cdot10^{-4}$	$1.2\cdot10^{-3}$

Table 5

Regression analysis of experimental results for resultant feature Y_3

R^2=0.9979 is the determination factor of the regression model of experimental data						
Regression factors	Regression factors (values)	Standard error	Student's coefficient	Significance level p, (p<0.05)	Confidence interval – 95 %	Confidence interval – 95 %
b_0	2,739.05	60.8	44.9	$8.0\cdot10^{-7}$	2,590.08	2,888.02
b_1	1.099	$1.2\cdot10^{-1}$	8.6	$1.3\cdot10^{-4}$	0.788	1.41
b_2	–1.378	$1.01\cdot10^{-1}$	13.6	$1.0\cdot10^{-5}$	–1.626	–1.13
b_8	$-1.6\cdot10^{-3}$	$2.1\cdot10^{-4}$	7.89	$2.19\cdot10^{-4}$	$2.1\cdot10^{-3}$	$1.1\cdot10^{-4}$
b_9	$-1.5\cdot10^{-3}$	$2.1\cdot10^{-4}$	7.69	$2.5\cdot10^{-4}$	$2.0\cdot10^{-3}$	$1.3\cdot10^{-3}$
b_{10}	$1.4\cdot10^{-3}$	$1.1\cdot10^{-4}$	13.79	$9.02\cdot10^{-6}$	$1.1\cdot10^{-3}$	$1.7\cdot10^{-3}$
b_{15}	$1.85\cdot10^{-3}$	$2.1\cdot10^{-4}$	9.01	$1.04\cdot10^{-4}$	$1.3\cdot10^{-3}$	$2.3\cdot10^{-3}$

For more accurate display of the results of the experiment, the mathematical regression model was complicated to the second order of dependence of factors without interaction, on conditions that determination factor should not be lower than 0.95. Analyzing the data from Tables 3–5, it is possible to conclude that all included factors are statistically significant, as evidenced by their significance level, the model describing the process of friction with the tribologically active additive. Substitute the data of Tables 3–5 in the general form of regression equations and obtain:

$$Y_1 = 0.638 - 4.3\cdot10^{-4}\cdot(X2) - 2.1\cdot10^{-4}\cdot(X3) - 8.5\cdot10^{-4}\cdot(X4) + 1.76\cdot10^{-4}\cdot(X5) + 2.8\cdot10^{-7}\cdot(X2\cdot X3) + 4.1\cdot10^{-7}\cdot(X2\cdot X4) + 2.4\cdot10^{-7}\cdot(X3\cdot X4) + 8.9\cdot10^{-7}\cdot(X4\cdot X5); \quad (12)$$

$$Y_2 = 1145.59 - 2.45\cdot10^{-1}\cdot(X1) + $$
$$+ 4.64\cdot10^{-1}\cdot(X2) - 7.56\cdot10^{-1}\cdot(X4) - $$
$$- 6.21\cdot10^{-1}\cdot(X5) + 1.0\cdot10^{-3}\cdot(X1\cdot X2) + $$
$$+ 1.1\cdot10^{-3}\cdot(X2\cdot X3) + 1.08\cdot10^{-3}\cdot(X2\cdot X4) + $$
$$+ 4.61\cdot10^{-4}\cdot(X3\cdot X4) + 8.5\cdot10^{-4}\cdot(X3\cdot X5); \qquad (13)$$

$$Y_3 = 2739.05 - 1.09\cdot(X1) - 1.378\cdot(X2) - $$
$$- 1.6\cdot10^{-3}\cdot(\%1X4) - 1.5\cdot10^{-3}\cdot(\%1X5) + $$
$$+ 1.4\cdot10^{-3}\cdot(X2\cdot X3) + 1.85\cdot10^{-3}\cdot(X4\cdot X5). \qquad (14)$$

To assess the adequacy of the models, the variance analysis of experimental data was carried out and Fisher criterion was determined. The implementation of the variance analysis is shown in Tables 6–8.

Table 6

Variance analysis of experimental results of resultant feature Y_1

Coefficients	Variance	Fisher criterion	Significance level *p*, (*p*<0,05)
b_2	$4.35\cdot10^{-4}$	13.15	$2.27\cdot10^{-3}$
b_3	$2.5\cdot10^{-4}$	7.65	$1.3\cdot10^{-2}$
b_4	$3.0\cdot10^{-3}$	91.92	$2.1\cdot10^{-6}$
b_5	$3.94\cdot10^{-1}$	119.27	$3.4\cdot10^{-7}$
b_{10}	$2.21\cdot10^{-3}$	66.81	$7.4\cdot10^{-7}$
b_{11}	$1.45\cdot10^{-2}$	436.6	$1.2\cdot10^{-7}$
b_{13}	$3.2\cdot10^{-3}$	96.7	$3.6\cdot10^{-6}$
b_{15}	4.48	136.5	$5.3\cdot10^{-7}$

Table 7

Variance analysis of experimental results of resultant feature Y_2

Coefficients	Variance	Fisher criterion	Significance level *p*, (*p*<0,05)
b_1	1,250.0	109.8	$1.42\cdot10^{-8}$
b_2	756.7	1.48	$2.0\cdot10^{-15}$
b_4	10,440.1	101.94	$2.41\cdot10^{-8}$
b_5	30,504.0	297,85	$9.2\cdot10^{-12}$
b_6	30,752.0	300.27	$8.62\cdot10^{-12}$
b_{10}	71,442.0	697.58	$1.3\cdot10^{-14}$
b_{11}	72,010.0	703.12	$1.2\cdot10^{-14}$
b_{13}	8,712.0	85.06	$8.4\cdot10^{-8}$
b_{14}	26,106.13	254.91	$3.0\cdot10^{-11}$

Table 8

Variance analysis of experimental results of resultant feature Y_3

Coefficients	Variance	Fisher criterion	Significance level *p*, (*p*<0.05)
b_1	53,710.0	578.49	$5.47\cdot10^{-14}$
b_2	399	4.19	$4.6\cdot10^{-2}$
b_8	116,041.5	1,249.9	$1.29\cdot10^{-16}$
b_9	127,891.5	1,377.49	$5.96\cdot10^{-17}$
b_{10}	115,800.0	1,247.27	$1.31\cdot10^{-16}$
b_{15}	120,172.5	1,294.35	$9.76\cdot10^{-17}$

Tables 6–8 show that the factors included in the mathematical model (12) to (14) adequately describe the examined friction process with the tribologically active additive, since significance level p for each factor is below the allowable level.

It is necessary to estimate additionally the emissions of residues between the experimental and forecasted values for obtained models (12) to (14). Significant emissions of ≥10 % of studied values (blue markers) from the forecasted values (red lines) should not be present in the analyzed data of the experiment. In the case where residues are ≥10 %, the model cannot be accepted for further research. The results of emissions of residues of the corresponding models are shown in Fig. 1.

Analyzing Fig. 1, it is possible to observe that there are no significant data emissions, so the mathematical model can be accepted as reliable.

The surfaces of the response of development of the studied response functions of the studied friction process with a tribologically active additive with the display of the values of factors and desirability scale are identical in character and presented in Fig. 2. The levels of response function by the desirability scale are shown in Fig. 3.

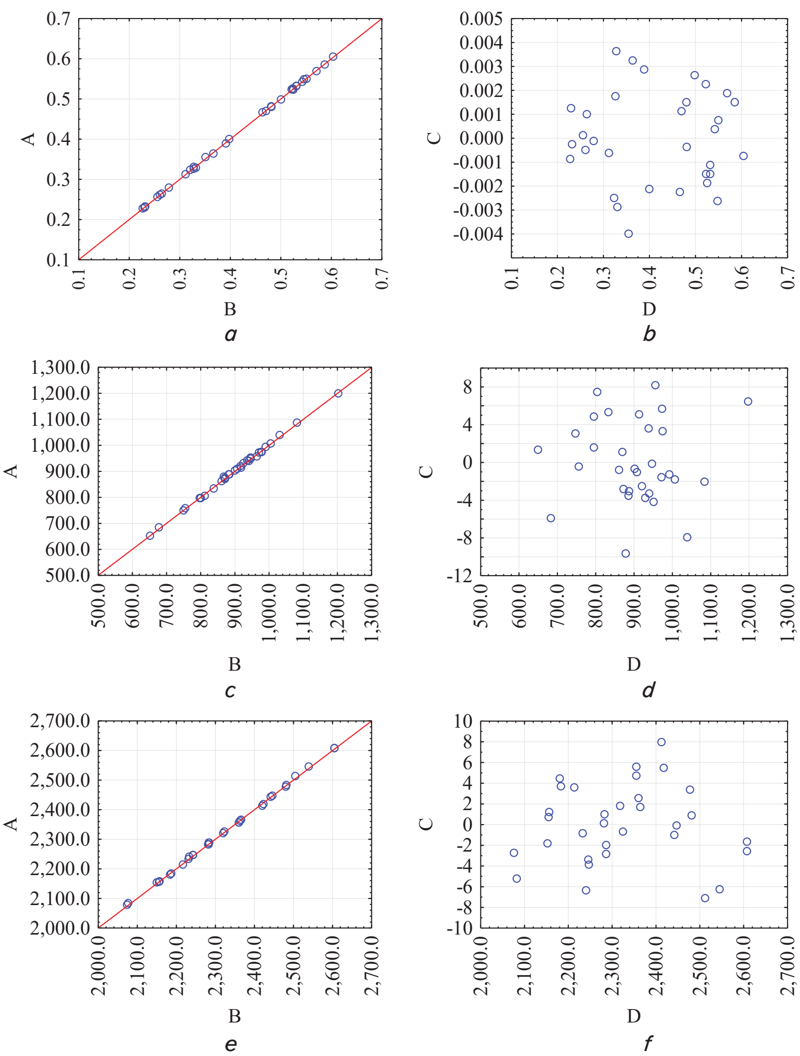

Fig. 1. The diagram of forecasted and observed residues, as well as estimation of their span: A — Forecasted values; B — Studied values; C — Output residues; D — Studied residues; *a, b* — for function of magnitude of wear; *c, d* — critical loading; *e, f* — welding loading

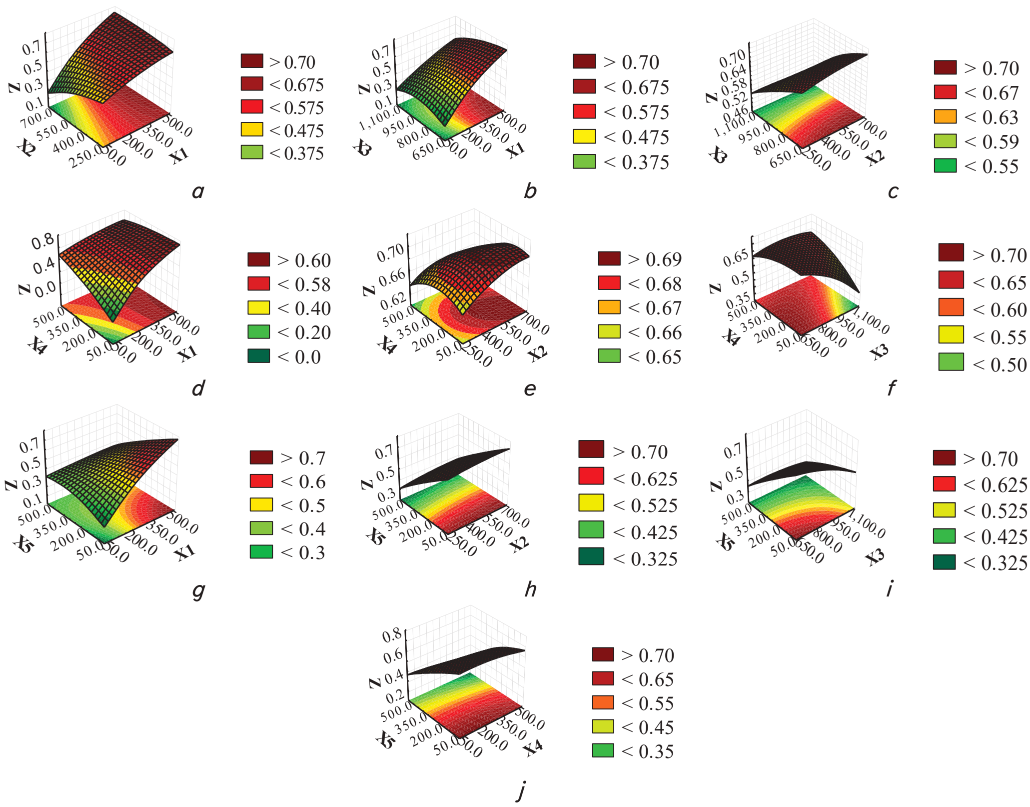

Fig. 2. Graphic display of response surfaces by the desirability scale of the studied tribologically active additive: *a* – dependence of *Z* on *X*2 and *X*1; *b* – dependence of *Z* on *X*3 and *X*1; *c* – dependence of *Z* on *X*3 and *X*2; *d* – dependence of *Z* on *X*4 and *X*1; *e* – dependence of *Z* on *X*4 and *X*2; *f* – dependence of *Z* on *X*2 and *X*1; *g* – dependence of *Z* on *X*5 and *X*1; *h* – dependence of *Z* on *X*5 and *X*2; *i* – dependence of *Z* on *X*5 and *X*3, *j* – dependence of *Z* on *X*5 and *X*4

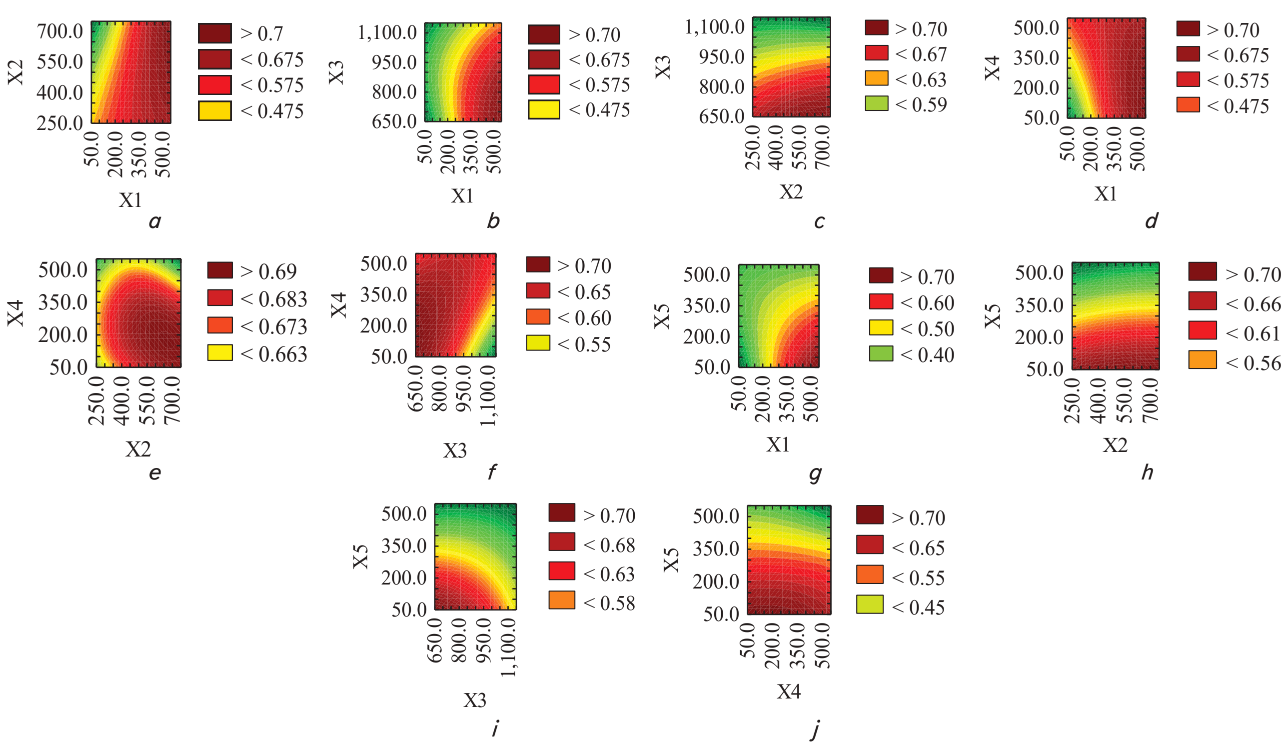

Fig. 3. Graphic display of response levels by the desirability scale of the studied tribologically active additive: *a* – dependence of *Z* on *X*2 and *X*1; *b* – dependence of *Z* on *X*3 and *X*1; *c* – dependence of *Z* on *X*3 and *X*2; *d* – dependence of *Z* on *X*4 and *X*1; *e* – dependence of *Z* on *X*4 and *X*2; *f* – dependence of *Z* on *X*2 and *X*1; *g* – dependence of *Z* on *X*5 and *X*1; *h* – dependence of *Z* on *X*5 and *X*2; *i* – dependence of *Z* on *X*5 and *X*3, *j* – dependence of *Z* on *X*5 and *X*4

By analyzing these diagrams visually, it is possible to note unambiguously that the optimum composition of the tribological additive exists in the studied ranges of the values of factors $X1$, $X2$, $X3$ $X4$, $X5$. The implementation of the composition of the tribologically active additive to oil by the developed model is possible with the help of desirability function.

To determine the optimal composition of tribologically active additive, we will set the boundaries of the examined response, which would include the entire experimental database, which is available for analysis. In these circumstances, it is possible to find the necessary maximum values of response by desirability function. Consider the above for each factor and each response function. Implementation of the procedure of determining the optimization of the tribologically active additive is shown in Fig. 4.

Fig. 4 shows that the rational option of the variant of the components is at the intersection of the maximum value of desirability function in the specified interval of each factor. At this composition of the tribologically active additive, it is possible to assert the rationality for the solution of the applied task. The results, which enable assessing the effectiveness of the additives, are summarized in Table 9.

The formed tribologically active additive allows increasing the quality of the lubricating composition as evidenced by the data of Table 9. There is a decrease in the indicator of wear in the tribological contact, an increase in critical load and in welding load.

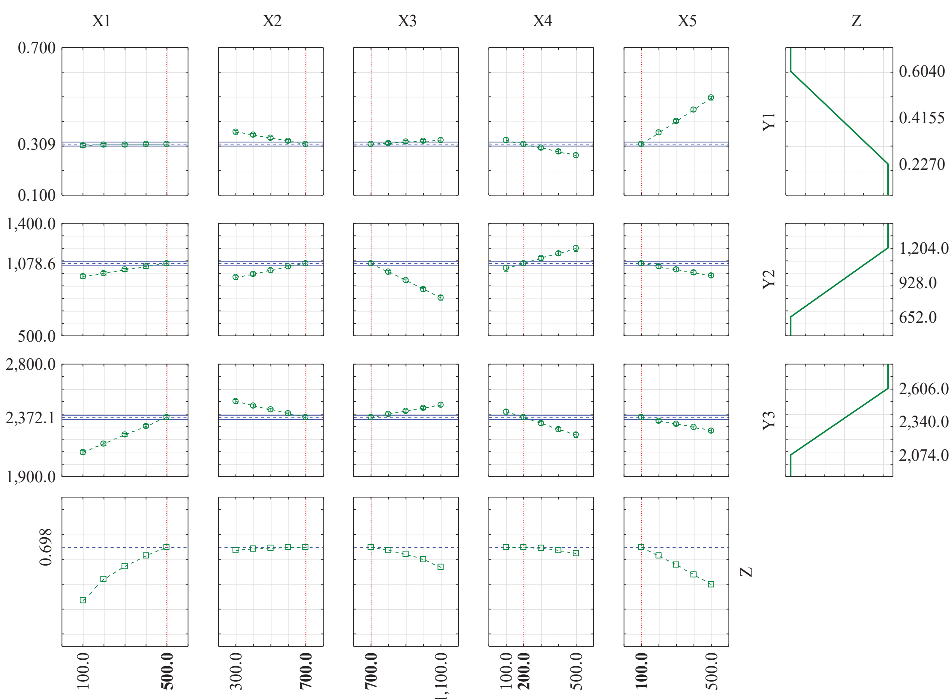

Fig. 4. Graphic display of the procedure of finding the rational composition of the tribologically active additive by the profile of desirability function

Table 9

Averaged results of testing on a four-ball friction machine of lubricating media

Averaged indicators	Basic oil GL-5 80W-90	Basic oil GL-5 80W-90+formed additive
Wear indicator, mm	0.392±0.05	0.309±0.04
Critical loading, N	893.9±3.0	1,078.6±3.0
Welding loading, N	2,252±17.0	2,372.1±25.0

6. Discussion of results of tribological examination of the obtained additives to oils

To prepare the tribologically active additive to oil and to achieve maximum values of response in the friction process of the tribosystem system, it is necessary to ensure the rational composition of the studied substances, at which response function has the maximum of desirability function. The maximum achievable desirability according to the experimental database and optimization conditions is 0.698. For this magnitude of desirability, the rational composition of the tribologically active additive will contain metakaolin in the range of [100.0...500.0 mg/50 ml], rational value is 500.0 mg/50 ml; dispersive powder of clay of Katerynivka deposit in the range of [300.0...700.0 mg/ 50 ml], the rational value is 700.0 mg/50 ml; sodium oleate in the range of [700.0...1,100.0 mg/50 ml], rational value is 700.0 mg/50 ml; lithium hydroxide in the range of [100.0...500.0 mg/50 ml], rational value is 200.0 mg/50 ml; sulfur in the range of [100.0...500.0 mg/50 ml], rational value is 100.0 mg/50 ml, according to the results in Fig. 4. Note that for another desirability level, the rational composition of the tribologically active additive will be different from the obtained one.

The formed composition of the tribologically active additive makes it possible to improve the physical and mechanical characteristics of tribosystems. According to the obtained tribological results, it is possible to assert that the tribologically active additive in the composition with oil is effective in comparison with the basic oil by its tribological characteristics. Under laboratory conditions, it was found that the wear indicator decreased by 26.8 %, critical load increased by 17.2 %, and welding load increased by 4.89 %. Working surfaces have a lower friction coefficient. For a more detailed study of functional coatings with the use of the tribologically active additive, in subsequent studies, it is necessary to pay attention to spectrometric analysis of functional coatings of samples and parts. The oil composition can be used for operation in power gearboxes with contact loading on the tooth surfaces up to 1,078 N and with peak overloading up to 2,372 N, according to the results in Table 9. Gear switch boxes and reducers of main transmission of transport machines can be the most rational for these data, as these units and their parts constantly operate under conditions of varying cyclic loading.

Limitations in the formation of components of the tribologically active additive by the input factors are described in more detail during the formation of Table 1.

The harmful effect on the reproduction of results primarily includes a failure to comply with the technological operations on preparation of dispersed clay powder, as well as metakaolin of necessary fraction dimensions. The operating modes of the use of this tribological active additive, first of all, must by characterized by its loading modes, which are specified above. The limitation during the operation in the temperature mode reaches about 150 °C, because this boundary is characterized by the level of oxidation of the basic oil GL-5 80W-90.

Extracts and components from clay must be effectively used for further research in the direction of the formation of tribologically active additives.

7. Conclusions

1. The rational composition of the tribologically active additive was found: oil – 50 ml, metakaolin – 500 mg/50 ml; variance clay powder of Katerynivka deposit – 700 mg/50 ml; sodium oleate – 700 mg/50 ml; lithium hydroxide – 200 mg/50 ml; sulfur – 100 mg/50 ml.

2. The introduction to oil of the tribologically active additive in the amount of 5.3 % under laboratory conditions reduces wear by 26.8 %, increases critical load by 17.2 % and increases welding load by 4.89 %. It was established that during the technical operation of the tribosystem with the composition oil with the developed tribologically active additive, the boundary loading should not exceed 1,078 N, and peak contact overloading should be not more than 2,372 N.

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