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DEVELOPMENT OF RATIONAL TECHNOLOGY FOR SODIUM GLYCEROXIDE OBTAINING

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The process of sodium glyceroxide obtaining by the reaction of glycerol and sodium hydroxide in the form of an aqueous solution was investigated.

Glycerol salts (metal glyceroxides) are important components in the synthesis of many compounds. Glyceroxides are used in the chemical industry, construction, medical practice, etc. Glyceroxides of alkali metals are used in the production of modified fats and biodiesel fuel.

P.a.-grade glycerol (CAS Number 56-81-5) was used with a mass fraction of the main substance of 99.5 %. The parameters of sodium hydroxide (CAS Number 1310-73-2) were studied: the mass fraction of the main substance is 98.0 %, the mass fraction of sodium carbonate is 0.5 %.

Rational conditions for sodium glyceroxide obtaining were determined: temperature (145 °C) and concentration of sodium hydroxide solution (65 %). Under these conditions, the mass fraction of the main substance in the product was 80 %. The melting point (72 °C) and mass fraction of moisture (0.3 %) in sodium glyceroxide were determined. The catalytic activity of the product in the process of transesterification of palm olein was tested. The increase in the melting point of palm olein was 15 °C. Under similar conditions of using potassium glyceroxide with a mass fraction of the main substance of 75.77 %, the increase in the melting point is 12.1 °C. This indicates an increase in the efficiency of the transesterification process using sodium glyceroxide obtained by the developed technology.

The research results make it possible to produce sodium glyceroxide under rational conditions with a high mass fraction of the main substance at enterprises that use metal glyceroxides as a production component or commercial product. The determined rational conditions will make it possible to effectively use the company's resources and predict the quality of the final product

Keywords: sodium glyceroxide, sodium hydroxide, alkali metal glyceroxides, fat transesterification catalyst

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1. Introduction

Metal glyceroxides are used in many directions and are a valuable component for the synthesis of a wide range of compounds. Glyceroxides are salts of glycerol, which is triatomic alcohol (propanetriol-1, 2, 3). Glycerol is a weak acid and forms halogen ethers, salts with alkalis, heavy metal oxides and other compounds. Glyceroxides of alkali metals are formed by the reaction of glycerol with metals, metal oxides and hydroxides [1].

The hygroscopic properties of metal glyceroxides determine the use of these substances in the production of absolute alcohol, for the adsorption of moisture and carbon dioxide [2]. Glyceroxides are used to obtain glycerol esters in the reaction with halogen derivatives, alkyd resins, biologically active medical compounds, etc. [3].

Glyceroxides of alkali metals are catalysts of the transesterification process used in the production of fats with specified technological properties [4]. Glyceroxides are also used in the production of mono- and diglycerides, fatty acid esters [5]. A particularly relevant direction is the production of alternative biodiesel fuel based on fatty acid esters of low molecular weight alcohols [6].

Alkoxides (methoxides and ethoxides of alkali metals) are common catalysts for transesterification. An acute issue of the use of methoxides and ethoxides is the explosive and fire hazard of these substances. Alkoxides are very reactive and interact with carbon dioxide, sulfur dioxide, hydrogen sulfide, moisture and air oxygen, thereby losing their catalytic activity irreversibly. The production of such catalysts is also explosive and fire-hazardous and requires special conditions and equipment. Therefore, productions using these substances purchase alkoxides and must have special loading devices due to the danger of these catalysts. Alkoxides are toxic substances and harmful to the environment [7]. The use of these substances leads to wastewater and soil pollution [8]. The self-ignition temperatures of sodium methoxide and sodium ethoxide are 80 °C and 50 °C, respectively [9]. The presence of 0.1 % moisture, 0.05 % fatty acids and a peroxide value of 1.0 ½ O mmol/kg in transesterifiable fat leads to the deactivation of 0.454 kg of sodium methoxide per 1 ton of fat. For metallic sodium, this figure is 0.193 kg/t, for sodium hydroxide – 0.11 kg/t [10]. Therefore, there is a need to develop technologies with alternative catalysts.

Thus, glyceroxides of metals have a wide range of applications. A promising issue is the replacement of modern dangerous and toxic transesterification catalysts with safer, affordable catalysts based on alkali metal glyceroxides. Transesterification is an important industrial process resulting in valuable products, particularly biodiesel fuel. It is advisable to produce metal glyceroxides at enterprises where technologies have been implemented using them as raw materials, catalysts or commercial products. Therefore, taking into account the needs of the industry for glyceroxides, the efficiency and safety of using these substances, it is urgent to develop a rational technology for obtaining glyceroxides with high concentrations of the main substance. So, research aimed at determining rational conditions for sodium glyceroxide obtaining, in particular, process temperature and concentration of sodium hydroxide solution is important.

2. Literature review and problem statement

Sodium glyceroxide is obtained by adding glycerol to metallic sodium in a medium of absolute alcohol and subsequent heating to complete evaporation of the alcohol under conditions of reduced pressure (at a temperature of about 100 °C). Another method of obtaining sodium glyceroxide is the interaction of anhydrous glycerol with powdered sodium hydroxide at a temperature of (120–145) °C [2].

The authors of the paper [11] considered the preparation of an anhydrous hydroxide-free alkaline catalyst for the methanolysis of rapeseed oil – sodium glyceroxide. Sodium glyceroxide was prepared by adding 16.0 g of sodium hydroxide as a 50 % aqueous solution (0.2 mol) to 18.4 g of glycerol (0.2 mol). Water added to the reaction and formed by the reaction was removed by rotary evaporation. But there are no data on rational conditions for obtaining the catalyst, no relationship between the amount and quality of the obtained substance and the parameters of the reaction process: temperature, duration, the ratio of components, etc. is shown.

The paper [12] describes the preparation of a methanolysis catalyst – a mixture of lithium glyceroxide and lithium hydroxide with a molar ratio of 2:1 and 3:1. The catalyst was prepared by heating a 10 % aqueous solution of lithium hydroxide and glycerol in a vacuum in three molar ratios of lithium to glycerol (1:1, 2:1, and 3:1). A constant amount of lithium hydroxide (0.25 g or 0.0104 mol) was mixed with glycerol to obtain molar ratios of 1:1, 2:1, and 3:1. The mixtures were heated at 120 and 140 °C at 25, 50, 100 and 120 Pa for 3 hours for water evaporation. As a response parameter, water loss was determined by weighing. But such a catalyst leads to partial saponification of fat or oil that is subject to methanolysis, due to the significant content of alkali in the catalyst. The content of lithium glyceroxide in the obtained catalysts is not shown in the work.

Rational conditions for potassium glyceroxide obtaining using glycerol and an aqueous solution of potassium hydroxide were determined in the work [3]: the molar concentration of glycerol in the mixture is 60 %, the reaction duration is 4 hours. A product with a glyceroxide concentration of 75.77 % was obtained. But there are no data on the influence of alkali concentration on the response function. This parameter is important because the formation of glyceroxides occurs under the conditions of effective evaporation of moisture in the reaction mass. Glyceroxides are hygroscopic and decompose into the original compounds – glycerol and alkali. Therefore, the concentration of the applied alkali solution and, accordingly, the amount of moisture in the mass, is essential.

The synthesis and use of calcium glyceroxide are of scientific and practical interest. In [10], the production of calcium glyceroxide from glycerol and calcium oxide was investigated to use it as a catalyst for methanolysis in the amount of 2 wt%. But the influence of technological conditions on the efficiency of the process of calcium glyceroxide formation, quality, concentration of the main substance and physicochemical properties is not shown.

The study [13] describes an environmentally friendly process of obtaining biodiesel fuel from waste oil using calcium glyceroxide as a catalyst. The advantage of this method is the absence of harmful and toxic alkoxides commonly used in this process. Biodiesel fuel with the content of methyl ethers (90.2–91.2) % was obtained. But the conditions of obtaining and quality indicators of the applied calcium glyceroxide are not shown.

In [14], it is shown that the synthesized calcium glyceroxide catalyst allows obtaining higher yields of methyl esters by transesterification (82.6 %) than a mixture of calcium oxide and glycerol (76.9 %). This is due to the fact that glyceroxide is a true catalyst of transesterification, which is formed in the reaction mass. The added glycerol can be used for other chemical processes, and the previously obtained glyceroxide directly catalyzes the transesterification process. But the work did not determine what conditions for the preliminary preparation of the catalyst are rational and what is the concentration of the catalyst in the product.

In [15], the production of calcium glyceroxide using calcium oxide, glycerol and methanol in the mass ratio of 1:1.6:13.4, respectively, was investigated. Carrying out the transesterification reaction in an ambient atmosphere for 2 hours at 333 K, using 0.4 % catalyst and the methanol to oil molar ratio of 7:1, a high-quality product was obtained using soybean oil and waste cooking oil. But no data are given on the composition of the obtained catalyst, the concentration of the main substance.

The authors of the paper [16] carried out the synthesis of calcium di glyceroxide, which was used as a catalyst for the methanolysis of sunflower oil. To obtain calcium glyceroxide, calcium oxide (500 mg) was mixed with methanol (100 cm^3) and glycerol (35 cm^3). Calcium glyceroxide made it possible to obtain a biodiesel yield of more than 80 %, while for calcium oxide this figure was 20 %. But the influence of the technological parameters of calcium glyceroxide obtaining on the quality and composition of the product has not been determined. These data are important during catalyst dosing, as well as for predicting the efficiency of glyceroxide-catalyzed processes.

Thus, high-quality metal glyceroxides are necessary for various industries, in particular, in the production of alternative fuels. Obtaining metal glyceroxides is an important issue of modern scientific research. But there is not enough data on the synthesis conditions of these substances, only the effectiveness and expediency of their use are shown. There is little data on the effect of glyceroxides production conditions on the composition, concentration and physicochemical properties of the obtained substances. Therefore, the research of rational conditions for obtaining alkali metal glyceroxides, in particular, the temperature and concentration of the applied alkali solution, remains an unsolved issue.

3. The aim and objectives of the study

The aim of the study is to determine rational conditions for obtaining sodium glyceroxide using glycerol and an aqueous solution of sodium hydroxide. This will make it possible to obtain high-quality sodium glyceroxide directly at the production and predict the concentration of the main substance in glyceroxide depending on the applied synthesis conditions. Sodium glyceroxide can be used as a catalyst in the production of transesterified fats and products based on them for a wide range of purposes. Unlike modern transesterification catalysts, sodium glyceroxide is safer to use.

To achieve the aim, the following objectives were set:

– to determine the quality indicators of the initial raw material – sodium hydroxide;

– to determine the relationship between reaction temperature, the concentration of the aqueous solution of sodium hydroxide and mass fraction of the main substance in the obtained product;

– to investigate the indicators of sodium glyceroxide: melting point and mass fraction of moisture;

– to check the catalytic activity of sodium glyceroxide in comparison with potassium glyceroxide in the process of transesterification of palm olein.

4. Materials and methods

4. 1. The object and hypothesis of the study

The object of the study is the technology of sodium glyceroxide obtaining from glycerol and an aqueous solution of sodium hydroxide. The main hypothesis of the study is the influence of the reaction temperature and the concentration of the aqueous sodium hydroxide solution on the mass fraction of the main substance in the obtained product. The study suggests that the temperature and concentration of the sodium hydroxide solution have a significant effect on the efficiency of glyceroxide synthesis. In the work, a simplification is adopted regarding the fact that foreign impurities present in the raw material do not affect the results of determining the dependence of the mass fraction of the main substance in the product on the technological parameters of the reaction process. The work uses standard and modified standard research methods.

4. 2. Examined materials and equipment used in the experiment

The following reagents and materials were used:

– p.a.-grade glycerol; concentration 99.5 % (CAS Number 56-81-5);

– p.a.-grade sodium hydroxide with the mass fraction of the main substance 98.0 % (CAS Number 1310-73-2);

– palm olein, refined, bleached, deodorized according to DSTU 4438 (CAS Number 93334-39-5).

4. 3. Methods for determining the quality indicators of the initial raw material – sodium hydroxide

The mass fraction of the main substance and the mass fraction of sodium carbonate in sodium hydroxide are determined by the standard method according to GOST 4328 (ISO 979 and ISO 3196, respectively). Permissible differences between the two parallel determinations did not exceed 0.3 %.

4. 4. Methods of sodium glyceroxide obtaining

Glycerol and an aqueous solution of sodium hydroxide (heated to 70 °C) with a concentration according to the experimental plan are placed in a heat-resistant flask, the mixture is thoroughly stirred with a glass rod. The molar concentration of glycerol in relation to sodium hydroxide is 60 %. The flask is fixed on a tripod and placed in a glycerol bath with a magnetic stirrer, the specified temperature is set (according to the experiment plan) using a thermostat, the flask is connected to a vacuum pump. The contents of the flask are stirred for 4 hours.

4. 5. Methods for determining the concentration of the main substance in sodium glyceroxide

The concentration of the main substance in sodium glyceroxide was determined using the potentiometric method given in [3].

4. 6. Methods of determining the quality indicators of sodium glyceroxide

The melting point of sodium glyceroxide was determined by the standard method according to EN ISO 3146, the mass fraction of moisture – by the method given in [3].

4. 7. Methods of palm olein transesterification

A weight of palm olein was placed in a heat-resistant round-bottomed flask installed on an electric plate. The catalyst in an amount of 0.3 % was added. The flask was connected to a vacuum pump. The process was carried out at a temperature of 85 °C for 1.5 hours, with stirring. The resulting mass was subjected to adsorption purification (amount of adsorbent 0.5 %, temperature 80 °C, duration 25 min.) and filtered on a paper filter. The melting point of palm olein was determined according to ISO 6321.

4. 8. Research planning and results processing

The second-order complete factorial experiment was used to plan the research and calculate the mathematical dependence. Processing of the results and construction of the graphic dependence were performed in the Stat Soft Statistica v6.0 package environment (USA). Each experiment was repeated twice.

5. Results of determining rational conditions for sodium glyceroxide obtaining

5. 1. Determination of the quality indicators of sodium hydroxide

When storing alkalis, absorption of moisture, sulfur dioxide, carbon dioxide from the air and the formation of impurities, such as sodium carbonate, can occur. In order to accurately dose raw materials, the mass fraction of the main substance in sodium hydroxide was determined – 98.0 %, as well as the mass fraction of sodium carbonate – 0.5 % according to the standard method.

5. 2. Determining the relationship between the reaction conditions and the mass fraction of the main substance in the product

The formation of glyceroxides of alkali metals occurs under the conditions of effective evaporation of moisture from the reaction mixture. Therefore, the process must be carried out at temperatures above 100 °C, under vacuum conditions. The amount of moisture in the reaction mass affects the rate of moisture evaporation, the rate of formation and the quality of the final product. Therefore, it is appropriate and necessary to determine how the temperature and concentration of an aqueous solution of sodium hydroxide affect the efficiency of the sodium glyceroxide formation process.

The influence of the technological parameters of sodium glyceroxide formation on the mass fraction of the main substance in the final product was determined. The following conditions of the second-order full factorial experiment were used: the number of factors – 2, the number of factor variation levels – 5, the number of experiments – 25. Factors and intervals of variation:

– x_1 – reaction temperature: from 100 to 160 °C;

– x_2 – concentration of sodium hydroxide solution: from 30 to 70 %.

The response function (y) – mass fraction of the main substance in the product, %. Processing of experimental data was performed using the Stat Soft Statistica v6.0 package (USA). The regression dependence of the mass fraction of the main substance in the product in real variables is as follows:

$$y = -217.40 + 3.17 \cdot x_1 + 0.48 \cdot x_2 - \\ -0.01 \cdot x_1^2 - 0.001 \cdot x_1 \cdot x_2 + 0.002 \cdot x_2^2. \quad (1)$$

The significance level of the coefficients of the regression equation (p>0.05) and the coefficient of determination (0.983) were determined. According to equation (1), the calculated values of the response function are determined. Table 1 shows the experiment planning matrix, experimental and calculated values of the response function.

Table 1

Planning matrix and response function values

Experiment No.	Factors of variation		Response function	
	Reaction temperature x_1, °C	Concentration of sodium hydroxide solution x_2, %	Mass fraction of the main substance in the product, **%** (experimental values)	Mass fraction of the main substance in the product, **%** (calculated values)
1	100	30	20.1	22.5
2	100	40	25.0	26.6
3	100	50	28.2	30.0
4	100	60	32.0	35.7
5	100	70	41.0	40.7
6	115	30	45.5	40.1
7	115	40	50.0	49.2
8	115	50	54.0	48.6
9	115	60	59.0	58.3
10	115	70	64.0	60.3
11	130	30	50.2	53.8
12	130	40	54.1	57.9
13	130	50	58.3	62.3
14	130	60	62.0	64.0
15	130	70	69.0	72.0
16	145	30	63.2	63.4
17	145	40	67.0	67.5
18	145	50	72.1	71.9
19	145	60	77.0	76.6
20	145	70	81.1	81.6
21	160	30	70.3	69.0
22	160	40	75.0	73.1
23	160	50	78.1	77.5
24	160	60	84.5	82.2
25	160	70	85.0	87.2

The dependence of the mass fraction of the main substance in sodium glyceroxide on the reaction temperature and concentration of the aqueous sodium hydroxide solution (response surface) is presented in Fig. 1.

The analysis of regression dependence (1), Table 1 and Fig. 1 revealed the following. As the reaction temperature and concentration of the sodium hydroxide solution increase, the mass fraction of the main substance in the obtained sodium glyceroxide increases. The response function is most significantly affected by the response temperature. The largest values of the response function are observed at a temperature from 130 °C and a concentration of sodium hydroxide solution from 60 %. This zone contains the response function values that are close to the maximum. Rational conditions for sodium glyceroxide obtaining are as follows: the concentration of sodium hydroxide solution is 65 %, the reaction temperature is 145 °C. The experimentally determined concentration of sodium glyceroxide in the product under these conditions is 80 %.

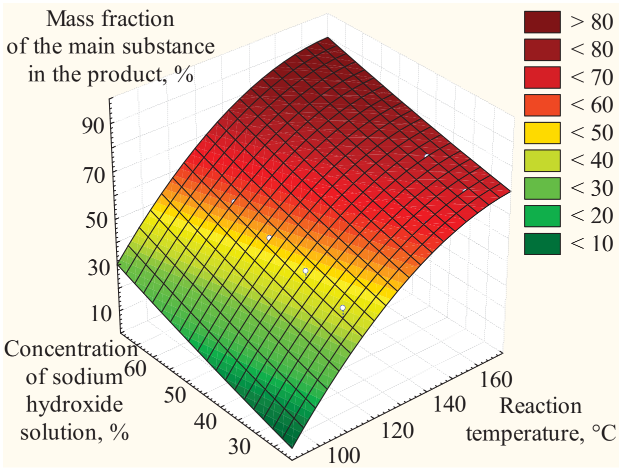

Fig. 1. Dependence of the mass fraction of the main substance in sodium glyceroxide on the reaction temperature and concentration of sodium hydroxide solution

5. 3. Study of sodium glyceroxide indicators

The following indicators of sodium glyceroxide obtained under rational conditions were determined: melting point (72 °C) and mass fraction of moisture (0.3 %). The paper [3] gives the results of the study on potassium glyceroxide obtaining at a concentration of potassium hydroxide solution of 50 %. Other parameters of glyceroxide obtaining were similar. The melting point of the product was 69 °C, the mass fraction of moisture was 0.8 %. The mass fraction of the main substance in the product was 75.77 %. Thus, sodium glyceroxide obtained under rational conditions has a higher quality and concentration of the main substance. The study of a wider set of parameters, the inclusion of the alkali solution concentration to the variation factors made it possible to obtain a high-quality product that can be used in various directions.

5. 4. Testing the catalytic activity of sodium glyceroxide compared to potassium glyceroxide in the process of palm olein transesterification

Experimental transesterification of palm olein with sodium glyceroxide, obtained under rational conditions, and with potassium glyceroxide, obtained in [3], was carried out. The increase in the melting point of palm olein as a result of transesterification when using sodium glyceroxide was 15 °C, potassium glyceroxide – 12.1 °C. Thus, sodium glyceroxide, obtained by the developed technology, exhibits greater catalytic activity.

6. Discussion of the results of studying rational technological parameters of the sodium glyceroxide synthesis process

The technology of sodium glyceroxide obtaining using glycerol and an aqueous solution of sodium hydroxide was studied. According to equation (1), Table 1 and Fig. 1, rational process conditions are determined: the concentration of sodium hydroxide solution (65 %), the reaction temperature (145 °C). The concentration of sodium glyceroxide in the product under these conditions is 80 %.

The formation of metal glyceroxides occurs under conditions of heating and effective evaporation of moisture. In the presence of moisture in glyceroxides, these substances decompose into initial components: glycerol and alkali, from which glyceroxide is formed again upon subsequent heating. This fact determines the economic and technological expediency of using glyceroxides as transesterification catalysts instead of alkoxides, which irreversibly lose their activity during storage and are dangerous toxic substances. The amount of moisture in the reaction mass during the formation of glyceroxides is essential, since an excess of moisture increases the duration and decreases the efficiency of the process.

An increase in the concentration of the sodium hydroxide solution and the reaction temperature leads to an increase in the mass fraction of the main substance in the product. The reaction temperature has a greater influence. The largest values of the response function are observed at a temperature from 130 °C and a concentration of the alkali solution from 60 %. The temperature has the maximum effect during the transition from 100 to 115 °C, then the growth slightly decreases. With an increase in the concentration of the alkali solution at the same temperature, the increase in the values of the response function is uniform. According to Table 1, the maximum value of the response function is observed at a temperature of 160 °C and a solution concentration of 70 %. But at the highest values of the response function, when the factors change from 145 to 160 °C and from 65 to 70 %, there is practically no increase in the response function. Therefore, it is rational to use the concentration of sodium hydroxide solution of 65 %, the reaction temperature of 145 °C.

The obtained scientific data on the dependence of the concentration of the main substance on the conditions of sodium glyceroxide synthesis will allow predicting the quality of the end product depending on the process parameters used. In case of inappropriate quality or long-term storage of the product, it is possible to re-process the mass to obtain glyceroxide. The cost of potassium hydroxide ($4.59/kg) is higher than that of sodium hydroxide ($2.43/kg). Therefore, the use of a rational and effective technology for obtaining high-quality sodium glyceroxide will contribute to the growth of the company's profitability.

The works [1, 8, 9] present data on the production of alkali metal glyceroxides. Thus, the authors [1] investigated the production of potassium glyceroxide and determined a rational molar ratio of glycerol:potassium hydroxide 2:1 and the process temperature of 130 °C. It is shown that the evaporation of moisture from the mass in this case is as effective as possible. In [8], hydroxide-free sodium glyceroxide was obtained by mixing 18.4 g of glycerol (0.2 mol) and 16.0 g of sodium hydroxide in the form of a 50 % aqueous solution (0.2 mol). Moisture from the mass was removed by rotary evaporation. In [9], the catalyst (a mixture of lithium glyceroxide and lithium hydroxide) was obtained by heating a 10 % aqueous solution of lithium hydroxide and glycerol in a vacuum in three molar ratios of lithium to glycerol (1:1, 2:1, and 3:1). As a response function, water loss was determined by weighing. So, the principle for obtaining glyceroxides of alkali metals, applied in modern research, is correlated with the one used in this work. Upon the contact of alkalis and glycerol during evaporation of moisture, the target product is obtained. But there are no data on the influence of the conditions of obtaining glyceroxides on the quality, composition and physicochemical properties of products. This issue, along with determining rational conditions, is solved in the work.

The limitation of using the research results is the need to take into account the real content of the main substance in the applied sodium hydroxide and glycerol. This indicator must be used when calculating the dosage of the components of the reaction mixture, as it can affect the efficiency of the synthesis process.

The drawback of the study is considering only the mass fraction of the main substance in the product as a response function. But melting temperatures, mass fraction of moisture in the product, etc. are also of scientific and practical interest. These data are also necessary for enterprises that need glyceroxides. For example, the lack of data on moisture content will make it impossible to use the product in processes with a strict limitation on the moisture content of the reaction mass or the end product.

Promising areas of work on this topic are the study of the obtained samples of sodium glyceroxide in relation to temperatures, thermal effects of melting and crystallization, mass fractions of the solid phase and degree of purity using instrumental studies. This will allow a more prompt assessment of the product quality, the required dosage and the expected result from the use of the substance.

7. Conclusions

1. Based on the quality analysis of the raw materials, the parameters of sodium hydroxide (CAS Number 1310-73-2) were determined: the mass fraction of the main substance is 98.0 %, the mass fraction of sodium carbonate is 0.5 %. Sodium hydroxide corresponds to the p. a. grade.

2. As a result of experimental research and data processing, rational conditions for sodium glyceroxide obtaining were determined: temperature (145 °C) and concentration of sodium hydroxide solution (65 %). Under these conditions, the mass fraction of the main substance in the product was 80 %.

3. From the quality studies of the obtained product, the following indicators of sodium glyceroxide were determined: melting point (72 °C) and mass fraction of moisture (0.3 %).

4. By checking the catalytic activity of sodium glyceroxide in comparison with potassium glyceroxide in the palm olein transesterification, it was found that sodium glyceroxide is more effective. The increase in the melting point of palm olein as a result of transesterification when using sodium glyceroxide was 15 °C, potassium glyceroxide – 12.1 °C.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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