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The object of research is the process of fat glycerolysis in order to obtain fatty acid monoglycerides.

Monoglycerides are an important component of chemical, pharmaceutical, cosmetic, and food industry products. These substances are used as emulsifiers, structure formers, complex formers, etc. The industrial production of monoglycerides involves the use of complex technologies, as well as dangerous and unstable catalysts. An urgent task is to develop new catalysts and improve technologies for monoglycerides obtaining.

The technology for the synthesis of monoglycerides by the glycerolysis method, which involves the reaction of vegetable hydrogenated fat with glycerol, was studied. Potassium glyceroxide was used as a catalyst, which is effective and safe in terms of production and use.

Hydrogenated unrefined fat according to DSTU 5040 (CAS Number 68334-28-1) was used. The fat has non-standard parameters: the melting point is 51 °C, the mass fraction of moisture and volatile substances is 0.3 %, the acid value is 3.2 mg KOH/g, the peroxide value is 7.6 ½ 0 mmol/kg.

The process duration was 90 minutes, the glycerol concentration was 50 %. Rational conditions for glycerolysis were determined: catalyst concentration (1.5 %) and temperature (140 °C). Under these conditions, the product ensured the stability of the “water – sunflower oil” emulsion of 96.8 %, the concentration of monoglycerides in the system was 0.1 %. Product parameters: mass fraction of monoglycerides – 72.5 %, free glycerol – 1.5 %, acid value – 1.7 mg KOH/g.

The research results make it possible to improve the glycerolysis process using a new catalyst and obtain monoglycerides with high emulsifying ability. This will increase the profitability of the enterprise and increase the volume of production of high-quality monoglycerides for various industries

Keywords: fatty acid monoglycerides, chemical transesterification catalyst, emulsion stability, potassium glyceroxide

IMPROVING THE TECHNOLOGY OF THE SYNTHESIS OF FATTY ACID MONOGLYCERIDES USING THE GLYCEROLYSIS REACTION

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1. Introduction

Fatty acid monoglycerides are nonionic surfactants that have a wide range of applications. These substances are used as stabilizers, emulsifiers, plasticizers, defoamers, etc. [1, 2].

In cosmetic production, monoglycerides are used in the production of creams, shampoos, balms, in the chemical industry – for the production of technological and automotive fluids, alcohol, and packaging materials [3, 4]. In the food industry, monoglycerides are used in the production of

canned food, emulsion products, etc. Among the emulsifiers used in industry, the share of monoglycerides is 60 % [5]. Thermodynamic properties of fatty acid esters, including monoglycerides, are also used in some biodiesel fuels [6, 7].

The traditional method of producing monoglycerides using glycerolysis is complex and multi-stage [8]. It involves obtaining a mixture of mono-, di-, triglycerides and glycerol, which requires further isolation of monoglycerides by centrifugation and molecular distillation [9]. This process is time-consuming and has a complex hardware design. Energy costs for the molecular distillation process are 30 % higher than for the distillation process. The addition of glycerol to the reaction mixture in an amount of at least 20 % changes the balance of the process towards the formation of monoglycerides [10].

During the synthesis of monoglycerides, transesterification and alcoholysis processes are used. Alcoholysis using glycerol (glycerolysis) is carried out in the presence of alkaline and acid catalysts.

The most common alkaline catalysts (metal alkoxides) are toxic, caustic, explosive, and fire-hazardous substances [11]. Alkoxides are capable of self-ignition and form explosive mixtures with air, which exacerbates the problem of industrial hazards [12, 13]. The high reactivity of alkoxides causes the reduction of the main substance, the loss of catalytic activity and the formation of explosive components [14].

Alkoxides are destroyed by moisture, acids, peroxides, carbon dioxide, oxygen, etc. Fat subjected to glycerolysis in the presence of these catalysts should have a mass fraction of moisture $<0.015\%$ (standard value – no more than 0.1%), peroxide value – $<0.25 \frac{1}{2} \text{ O mmol/kg}$ (standard value – no more than $10.0 \frac{1}{2} \text{ O mmol/kg}$) [15, 16].

Alkoxides have a polluting effect on soils and wastewater. At the same time, it is important to use innovative water purification technologies [17, 18] and technological solutions at enterprises that consume a large amount of water resources [19, 20]. There are many clear and reliable methods for detecting threats or changes in the environment [21, 22], determining the risk to human health [23, 24] and timely preventing or eliminating emergency situations [25, 26]. However, constant monitoring must be carried out to determine the impact of nanoproductions on the environment and human health for the safe and productive use of innovative technologies [27].

Thus, the problem of finding new environmentally safe ways to produce monoglycerides using new catalysts, which are safer and more resistant to the influence of foreign impurities present in fats, is urgent. At the same time, an important task is to obtain high-quality monoglycerides that have a high emulsifying capacity with minimal negative effects on human health and the environment. This will meet the needs of various industries in high-quality emulsifiers.

2. Literature review and problem statement

Monoglycerides are highly effective as emulsifiers, can also act as stabilizers, defoamers, structure formers, complex formers, etc. Researching the technology of monoglycerides obtaining is an important task. The main method of synthesis of monoglycerides is glycerolysis of fats. There are different options for carrying out this process. For example, the process of enzymatic glycerolysis for the production of monoglycerides was studied in [28]. The optimal amount of Fermase CALB 10000 enzyme preparation was 15 % by

weight, the molar ratio of oil and glycerol was optimized at 1:5. The disadvantage of the work is the high cost of the catalyst (about \$ 600/kg). Acid and alkaline catalysts are also used in the glycerolysis process. The authors [29] considered the use of waste and non-standard raw materials in the glycerolysis process catalyzed by an alkaline catalyst. However, glycerolysis is effective only under the conditions of pre-treatment of raw materials with a decrease in the content of free fatty acids from 2.82 and 2 % to 1 %. Catalysts used in industry are not effective for non-standard raw materials. This is a disadvantage of using such catalysts, as it is promising to use waste and spent fats for industrial processing to obtain valuable products.

In addition, scientific research is devoted to obtaining monoglycerides from synthesized complex esters of the same type. In this case, monoglycerides with an ether group in the specified positions are obtained. For example, the authors of [30] investigated the glycerolysis of methyl oleate, which resulted in glycerol monooleate, a monoglyceride with an ether fragment in one of the terminal positions of the glycerol molecule. Magnesium oxide was used as a catalyst. But the rational conditions of glycerolysis and the influence of the process conditions on the parameters and properties of the product are not shown.

The relevant direction is the intensification of the glycerolysis process by the action of microwave radiation, which was demonstrated in [31]. As a result of microwave radiation, the reaction time was reduced from 240 to 25 min., the optimal temperature was reduced from 200 to 105°C . The optimal molar ratio of oil to glycerol was 1:2, the catalyst concentration was 0.1 %. But it is not shown how the process conditions affect the quality, yield and emulsifying properties of the product.

An important direction is the use of waste from the oil and fat industry for the production of valuable products. The work [32] presents the results of processing soapstock into fatty acids, which can be used for the synthesis of monoglycerides. As a result of alkaline neutralization of oils, a significant amount of soapstock is formed, the disposal of which creates an environmental and economic problem. But the influence of the reaction conditions of glycerol and fatty acids on the quality and quantity of monoglycerides is not shown.

Effective catalysts of glycerolysis are metals. In particular, in [33], monoglycerides were obtained by the transesterification reaction of methyl stearate in the presence of metals (lithium, potassium, or cesium). The highest yield of monoglycerides (about 78 %) was observed in the presence of lithium. But the use of alkali metals as catalysts has disadvantages: the high reactivity of metals leads to a rapid loss of catalytic activity.

A wide range of oils, fats and compositions based on them are used as raw materials for obtaining monoglycerides. For example, the study [34] considered the production of monoglycerides from castor oil. Glycerolysis of castor oil gave the highest yield of products at 180°C and 30 min (50.4 % monoglycerides and 35 % diglycerides). The disadvantage of the work is the lack of data on the emulsifying properties and melting point of the obtained product. In [35], the melting point of monoglycerides obtained as a result of glycerolysis of hydrogenated fat was investigated. Rational conditions for glycerolysis were determined: duration (90 min.) and glycerol concentration (50 %). Under these conditions, the yield of monoglycerides was 32.9 %, the melting point was 61.5°C . But the effect of synthesis conditions on the emulsifying ability of the obtained product is not shown.

Therefore, it is urgent to improve the technology of glycerolysis of fats using a new catalyst, as well as non-standard or used raw materials. It is necessary to determine the effect of synthesis conditions on the properties of monoglycerides. After all, it is the emulsifying ability that is key to the use of monoglycerides in various directions. Scientific research is devoted to the study of various methods of glycerolysis, various process conditions and catalysts are considered. But the use of non-standard fats for the glycerolysis reaction, the use of stable, effective and safe catalysts remains an unsolved issue. In addition, there is insufficient data on the properties of the reaction products obtained. Therefore, it is advisable to determine a relationship between the production conditions and the emulsifying ability of monoglycerides.

3. The aim and objectives of the study

The aim of the study is to determine rational conditions for the synthesis of fatty acid monoglycerides through the glycerolysis reaction using non-standard raw materials. This will improve the technology of monoglycerides obtaining using an effective catalyst of potassium glyceroxide and fatty raw materials with increased indicators of oxidative, hydrolytic deterioration and moisture content. The production and use of the applied catalyst are safer compared to common alkaline catalysts of transesterification and alcoholysis processes.

To achieve the aim, the following objectives were set:

- to determine the quality indicators of fatty raw materials – unrefined hydrogenated vegetable fat;
- to determine the dependence of the emulsifying ability of monoglycerides on the production conditions: catalyst concentration and glycerolysis temperature and to determine rational conditions for the synthesis of monoglycerides;
- to investigate the physico-chemical parameters of monoglycerides obtained under rational conditions (catalyst concentration and glycerolysis temperature, under which the greatest stability of the emulsion in the presence of monoglycerides is observed).

4. Materials and methods of research

4.1. The object and hypothesis of the research

The object of research is the process of glycerolysis of hydrogenated vegetable fat in the presence of a potassium glyceroxide catalyst.

The main research hypothesis is that catalyst concentration and glycerolysis temperature affect the emulsifying ability of the obtained monoglycerides. The study suggests that increasing the concentration of the potassium glyceroxide catalyst in the reaction mixture increases the emulsifying ability of the obtained monoglycerides, and also provides an opportunity to lower the process temperature. A simplification is adopted that the initial indicators of fatty raw materials do not affect the effectiveness of the catalyst.

4.2. Researched materials and equipment used in the experiment

The following reagents and materials were used:

- unrefined hydrogenated vegetable fat according to DSTU 5040 (CAS Number 68334-28-1);
- p. a.-grade potassium hydroxide (mass fraction of the main substance not less than 85.0 %) (CAS Number 1310-58-3);

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

4.3. Methods for determining the quality indicators of fatty raw materials

Physico-chemical indicators were determined using generally accepted methods presented in international documentation on the analysis of oils and fats. The melting point was determined according to ISO 6321. The mass fraction of moisture and volatile substances was determined according to the standard method intended for animal or vegetable fats and oils according to ISO 662. Method A was used, which is used for liquid and solid fats and oils (with different acid values). The acid value and peroxide value were determined according to international standards ISO 660 and ISO 3960, respectively.

4.4. Methods of carrying out the glycerolysis process

The process of glycerolysis of experimental fat was carried out in laboratory conditions according to the method given in [35]. In all experiments, the process duration was 90 minutes, the glycerol concentration was 50 %.

4.4. Methods for determining the quality indicators of the obtained monoglycerides

The emulsifying ability of monoglycerides was determined as follows. 50 g of distilled water is added to the beaker of the homogenizer, to which 50 g of sunflower oil is added with an amount of monoglycerides dissolved in it (0.1 % by weight of the water and oil mixture). Homogenization is carried out for 300 s at a speed of 16 s⁻¹. The emulsion is kept for 600 s, after which the stability of the emulsion is determined by centrifugation with an acceleration of 210 g for 300 s. The stability of the emulsion is calculated by the formula:

$$C = \frac{E_n \cdot 100}{E_0}, \quad (1)$$

where C is emulsion stability, %; E_n is the amount of unstratified emulsion; E_0 is the total amount of emulsion.

The analysis of the physicochemical indicators of monoglycerides was carried out using generally accepted methods according to international documentation on the analysis of oils and fats. The mass fraction of monoglycerides and free glycerol was determined according to DSTU ISO 7366:2014 (ISO 7366). The melting point and acid value were determined according to ISO 6321 and ISO 660, respectively.

4.5. Planning of experimental studies and processing of results

A second-order full factorial experiment was used for research planning and mathematical processing of the obtained data. Data processing and construction of graphical dependence were performed in the StatSoftStatistica v6.0 (USA) package environment. Each experiment was repeated twice.

In the Statistica package, calculations were made using the “General Regression Models” module, which combines the necessary procedures for data processing. Thus, the following tabs were applied:

- parameter estimates (calculation of equation coefficients, standard error, 95 % confidence interval);
- “Observed, Predicted, and Residual Values” (determination of calculated values of the response function);
- “ANOVA” (analysis of variance).

5. Results of research on determining rational conditions for the synthesis of fatty acid monoglycerides

5.1. Determination of quality indicators of fatty raw materials

Hydrogenated unrefined fat according to DSTU 5040 (CAS Number 68334-28-1) was used as the initial raw material. Table 1 presents the physico-chemical parameters of the experimental fat and the corresponding standards according to DSTU 5040 (for M6 fat intended for the production of surfactants).

Table 1

Physico-chemical parameters of experimental fat

Indicator	Characteristic	Standard according to DSTU 5040
Melting point, °C	51	not lower than 53
Mass fraction of moisture and volatile substances, %	0.3	0.2
Acid value, mg KOH/g	3.2	3.0
Peroxide value, ½ O mmol/kg	7.6	– during the release: 3; – at the end of storage: 10

So, the experimental fat has non-standard quality indicators: increased hydrolytic and oxidative deterioration, as well as mass fraction of moisture and volatile substances. The melting point of fat is lower than standard.

5.2. Determination of the dependence of the emulsifying capacity of monoglycerides on the catalyst concentration and glycerolysis temperature

To obtain fatty acid monoglycerides, the glycerolysis process was used, which was carried out in the presence of a potassium glyceroxide catalyst. Potassium glyceroxide is characterized by high efficiency and is a true catalyst in the processes of transesterification and alcoholysis of fats [35]. The effect of glycerolysis conditions on the main quality indicator of monoglycerides – emulsifying ability – was determined. A second-order full factorial experiment was applied. Factors and intervals of variation:

- x_1 – catalyst concentration: from 0.1 to 1.5 %;
- x_2 – process temperature: from 80 to 200 °C.

The response function is the stability of the “water – sunflower oil” emulsion (y), which was determined under the conditions of a monoglyceride concentration of 0.1 wt%. The results were processed using the StatSoft Statistica v6.0 (USA) package. The calculated regression dependence of the stability of the “water – sunflower oil” emulsion in the presence of the obtained monoglycerides (y) on the glycerolysis conditions in real variables is as follows:

$$y = 17,083 + 8,459 \cdot x_1 + 13,776 \cdot x_1^2 + 0,385 \cdot x_2 - 0,001 \cdot x_2^2 \quad (2)$$

The level of significance of the coefficients of the regression equation ($p > 0.05$) and the coefficient of determination, which was 0.993, were determined. Table 2 presents the experiment planning matrix, experimental and calculated values of the response function (emulsion stability).

Table 2

Planning matrix and response function values

Experiment number	Factors of variation		Emulsion stability, % (experimental value)	Emulsion stability, % (calculated value)
	Catalyst concentration, %	Process temperature, °C		
1	0.1	80	41.5	43.0
2	0.1	140	55.8	54.0
3	0.1	200	58.1	58.4
4	0.8	80	57.1	57.6
5	0.8	140	66.7	68.6
6	0.8	200	75.4	73.0
7	1.5	80	87.7	85.7
8	1.5	140	96.8	96.7
9	1.5	200	99.0	101.1

In graphic form, the dependence of the stability of the “water – sunflower oil” emulsion in the presence of the obtained monoglycerides on the glycerolysis conditions is presented in Fig. 1.

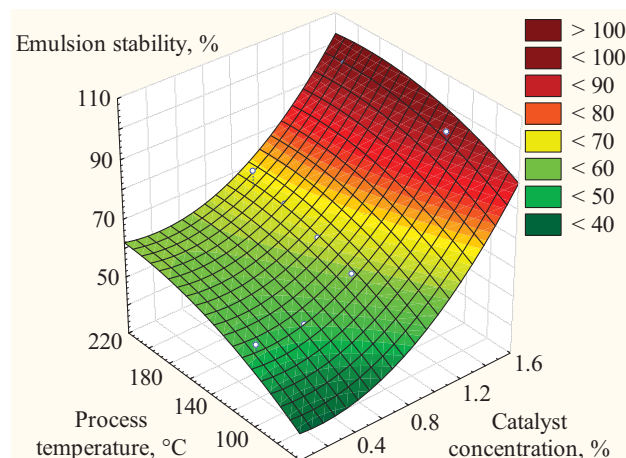


Fig. 1. Dependence of emulsion stability on glycerolysis conditions: catalyst concentration and process temperature

Increasing the glycerolysis temperature from 80 to 200 °C uniformly increases the emulsion stability provided by monoglycerides. Starting with a catalyst concentration of 1.15 %, there is a sharp increase in the values of the response function. The maximum values of the response function are observed at the maximum catalyst concentration (1.5 %). Significant increases in the values of the response function are observed starting from the process temperature of 140 °C.

Therefore, in order to achieve the maximum values of emulsion stability, it is advisable to use a catalyst concentration of 1.5 % and a process temperature of 140 °C. At the same time, the stability of the “water – sunflower oil” emulsion in the presence of the obtained monoglycerides was 96.8 %.

5.3. Research of physicochemical indicators of monoglycerides obtained under rational conditions

The product obtained under certain rational conditions was analyzed. The physicochemical parameters of monoglycerides are presented in Table 3.

Table 3
Physicochemical indicators of monoglycerides

Indicator	Characteristic
Mass fraction of monoglycerides, %	72.5
Melting point, °C	62.7
Mass fraction of free glycerol, %	1.5
Acid value, mg KOH/g	1.7

Therefore, the developed rational conditions of glycerolysis (catalyst concentration 1.5 %, process temperature 140 °C), which involve the use of a lower temperature, increase the quality of monoglycerides. Thus, the melting point increases and the acid value decreases, even if non-standard fatty raw materials are used.

6. Discussion of the results of studying the dependence of the emulsifying ability of monoglycerides on the glycerolysis process conditions

The technology for the synthesis of monoglycerides of fatty acids has been improved by determining rational conditions for the glycerolysis of non-standard fat in the presence of a potassium glyceroxide catalyst. According to equation (2), Table 2 and Fig. 1, the following rational conditions for glycerolysis are determined: catalyst concentration 1.5 %, process temperature 140 °C. Under these conditions, the stability of the “water – sunflower oil” emulsion in the presence of the obtained monoglycerides was 96.8 %.

By chemical nature, the mechanism of glycerolysis is similar to that of hydrolysis and transesterification. In the presence of alkaline catalysts (including potassium glyceroxide), a glyceroxide anion is formed in the reaction mass, which is a true homogeneous catalyst of the process. Therefore, the use of metal glyceroxides is effective and appropriate. Potassium glyceroxide is more resistant to destruction by moisture, acids and other compounds, and therefore is effective when using raw materials with a high content of peroxides, free fatty acids, and moisture.

Analysis of equation (2), Table 2 and Fig. 1 shows the following. An increase in the catalyst concentration has a more significant effect on the value of the response function than the glycerolysis temperature.

Starting from a concentration of 1.15 %, there is a sharp increase in emulsion stability in the presence of monoglycerides. In particular, when going from a concentration of 1.15 to 1.5 % at a process temperature of 80 °C, the stability of the emulsion increases by 24.2 %. The increase in the values of the response function when the process temperature is increased is more uniform and when going from 80 to 200 °C, it is 11.3–18.3 %. Therefore, it is most effective to use the maximum catalyst concentration (1.5 %) and the glycerolysis temperature of 140 °C, from which significant increases in the values of the response function begin.

Lowering the glycerolysis temperature is expedient, since elevated temperatures deteriorate the quality of the finished product, intensify the processes of oxidation and polymerization of fats, the formation of by-products, and require substantial energy consumption.

The spontaneous process of glycerolysis even at temperatures above 250 °C occurs at a rate that is insufficient for practical needs. Alkaline and acid catalysts are used to reduce the activation energy and increase the reaction rate, among which the most effective are alkaline alkoxides. But these catalysts

are toxic, quickly break down and lose activity under the influence of air moisture and impurities present in fats. The potassium glyceroxide catalyst is highly effective in transesterification processes, and therefore its use is appropriate in the synthesis of monoglycerides by the glycerolysis method.

The quality indicators of monoglycerides obtained under rational conditions were studied. A comparative analysis of the data with monoglycerides presented in [35] was performed, where the glycerolysis temperature was 180 °C, the catalyst concentration was 0.5 %. Standard hydrogenated refined vegetable fat was used as raw material. At the same time, the melting point of monoglycerides was 61.5 °C, the acid value was 2.2 mg KOH/g. The improved technology with a decrease in process temperature allows obtaining higher quality monoglycerides (melting point 62.7 °C, acid number 1.7 mg KOH/g).

The results of research on determining rational conditions for glycerolysis make it possible to obtain high-quality monoglycerides. At the same time, the process of glycerolysis of non-standard fatty raw materials was applied using potassium glyceroxide, which is a safer and more accessible catalyst compared to alkoxides. Catalyst production technology does not require complex equipment and is not explosive or fire-hazardous.

The works [28–31, 33, 34] provide data on obtaining fatty acid monoglycerides from various raw materials, using various catalysts and glycerolysis conditions. Thus, the work [29] states that effective glycerolysis of non-standard fatty raw materials is possible only under the conditions of pretreatment with a decrease in the content of free fatty acids. The authors [34] showed the production of monoglycerides from castor oil by the glycerolysis method. So, the main method of synthesis of monoglycerides is glycerolysis, which correlates with the method considered in this paper. But there is not enough data on the rational conditions of glycerolysis, as well as the quality indicators of the obtained products, in particular, the most important functional characteristic – emulsifying ability. This problem is solved in this work.

A limitation for the use of research results is the need to check the quality of the components for the preparation of the catalyst (potassium hydroxide and glycerol). Thus, a decrease in the concentration of the main substance in potassium hydroxide can lead to a decrease in the concentration of potassium glyceroxide in the product and a decrease in catalytic activity. Limitations are also the ranges of variation of input variables: catalyst concentration (from 0.1 to 1.5 %) and glycerolysis temperature (from 80 to 200 °C). The obtained results refer to these ranges of variation. When going beyond these ranges, additional research is needed.

The drawback of the study is that the emulsifying ability of monoglycerides is checked only in the “water – sunflower oil” system. It would be appropriate to test the effectiveness of monoglycerides in a multicomponent system, for example, in a cream, process or automotive fluid, etc.

A promising area of work is the use of various types of fatty raw materials and other catalysts (alkaline and acid) in order to obtain monoglycerides. This will help to increase the production of monoglycerides, which are valuable production components in various industries.

7. Conclusions

1. The physico-chemical parameters of the initial raw material used in the study – unrefined hydrogenated vegetable

fat according to DSTU 5040 (CAS Number 68334-28-1) were determined. The melting point of fat was 51 °C, the mass fraction of moisture and volatile substances was 0.3 %, the acid value was 3.2 mg KOH/g, the peroxide value was 7.6 ½ O mmol/kg. Therefore, fatty raw materials are non-standard – they have increased indicators of oxidative and hydrolytic deterioration and moisture content.

2. As a result of experimental studies, the dependence of the emulsifying ability of monoglycerides on the conditions of the glycerolysis process was determined in the form of a second-degree regression equation. From the obtained equation, it was found that increasing the catalyst concentration has a more significant effect on the value of the response function than the glycerolysis temperature. Starting from a concentration of 1.15 %, there is a sharp increase in emulsion stability in the presence of monoglycerides. In particular, when going from a concentration of 1.15 to 1.5 % at a process temperature of 80 °C, the stability of the emulsion increases by 24.2 %. The increase in the values of the response function when the process temperature is increased is more uniform and when going from 80 to 200 °C, it is 11.3–18.3 %. Rational conditions for glycerolysis were determined: catalyst concentration (1.5 %) and temperature (140 °C). Under these conditions, the product ensured the stability of the “water – sunflower oil” emulsion of 96.8 %, the concentration of monoglycerides in the system was 0.1 %.

3. The quality indicators of the finished product obtained as a result of research – monoglycerides obtained

under rational conditions – were determined. The melting point of monoglycerides was 62.7 °C, the mass fraction of monoglycerides was 72.5 %, free glycerol was 1.5 %, and the acid value was 1.7 mg KOH/g. The developed rational conditions made it possible to obtain monoglycerides of higher quality than in the case of glycerolysis temperature of 180 °C, catalyst concentration of 0.5 %. At the same time, the melting point of monoglycerides was 61.5 °C, the acid value was 2.2 mg KOH/g.

Conflict of interest

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

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Data availability

The manuscript has no associated data.

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