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DEVELOPMENT OF SAFE TECHNOLOGY OF OBTAINING FATTY ACID MONOGLYCERIDES USING A NEW CATALYST

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Fatty acid monoglycerides are a valuable component of the products of various industries. The emulsifying ability of monoglycerides is used in cosmetic, pharmaceutical, and food production.

The process of fatty acid monoglycerides obtaining by the reaction of vegetable hydrogenated fat (salomas) with glycerol (glycerolysis method) has been studied. Potassium glycerate is used as a catalyst, which is characterized by high efficiency and safety of production and use.

A feature of the work is the study of the dependence of the yield and melting point of monoglycerides on the technological parameters of glycerolysis.

As a raw material, hydrogenated refined fat according to DSTU 5040 (CAS Number 68334-28-1) was used: melting point – 48 °C, mass fraction of moisture and volatile substances – 0.08 %, acid value – 0.25 mg KOH/g, peroxide value – 2.8 ½ O mmol/kg.

In all experiments, the glycerolysis temperature was 180 °C, the catalyst concentration – 0.5 % in terms of metal.

Rational conditions for glycerolysis were determined: duration (90 min.) and glycerol concentration (50 %). Under these conditions, the monoglycerides yield was 32.9 %, melting point – 61.5 °C. The mass fraction of free glycerol in monoglycerides was 1.0 %, acid value – 2.2 mg KOH/g.

The efficiency of monoglycerides obtaining using potassium hydroxide and glycerol mixture as a catalyst under certain rational conditions has been studied. The monoglycerides yield of 30.1 %, melting point of 59 °C were obtained. Therefore, the use of potassium glycerate catalyst is more efficient.

The results of the study make it possible to improve the technology for the production of fatty acid monoglycerides using a new catalyst and use resources rationally

Keywords: fatty acid monoglycerides, potassium glycerate, hydrogenated fat, glycerolysis, melting point

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1. Introduction

Partial fatty acid esters (mono-, diglycerides) are non-ionic surfactants that are used in various industries as plasticizers, emulsifiers, dispersants, stabilizers, etc. By stabilizing emulsions and effective homogenization, the rheological and cosmetic properties of creams and ointments are improved. In the food industry, they are used in bakery, confectionery, sugar, and alcoholic beverage industries. The part of monoglycerides in the total use of emulsifiers is 60 % [1].

Nonionic surfactants are becoming increasingly important. Their potential consumers are the oil, chemical and light industries. These substances are obtained from natural raw materials by chemical, microbiological and biochemical methods. However, the production of

nonionic surfactants is characterized by high resource consumption. The molecular distillation stage requires the use of vacuum (10^{-3} mmHg and below) requiring special equipment. Energy costs for the process of molecular distillation are up to 30 % higher than for the distillation process [2]. Typical process catalysts are alkoxides or oxides of lead, zinc and other metals, which have a number of disadvantages. In the production of monoglycerides, the processes of transesterification and alcoholysis are used. Common catalysts for these processes (metal alkoxides) are toxic, explosive, flammable substances that are harmful to the environment, and decompose to form toxic and volatile substances [3]. This exacerbates the problem of wastewater and soil pollution [4]. The self-flash temperature of sodium methylate is 80 °C, sodium ethylate – 50 °C. The use of such catalysts requires the value of the mass fraction of moisture in the fatty raw material, which is significantly lower than the standard value (the required value is 0.015 %, the standard value – 0.1 %) [2].

Thus, in order to improve the safety of the glycerolysis process, it is advisable to improve the technology for producing fatty acid monoglycerides using a new catalyst and reduce the negative impact of the process on the environment. This area of scientific research is important for the industry, since there is a need for affordable and safe emulsifiers in many industries.

2. Literature review and problem statement

Monoglycerides allow creating emulsions of fat and water, forming a crystalline structure of fats, in which liquid oils can be introduced, which increases the hardness of the fat product [2]. Distilled, acetylated, succinylated monoglycerides and monoglyceride esters of other acids and sucrose are used in the food industry [5].

Monoglyceride is a more high-melting substance than triglyceride. The melting point of monolaurin is (40–50) °C, monostearin – (57–61) °C, α-monopalmitin – 77 °C, β-monopalmitin – 69 °C. This allows reducing the amount of the component and provides the required hardness compared to triglycerides [6]. In the pharmaceutical industry, monoglycerides are used to increase the bioavailability of high-lipid and hydrophobic drugs. Their content in medicines reaches 10 %, in the shells of dosage forms – up to 40 % [7].

The development and improvement of technologies for monoglycerides production is an important scientific and practical direction.

There is a method of monoglycerides obtaining by enzyme technology at sufficiently low temperatures (30–46) °C in the presence of lipase enzymes [8]. However, there is a negative impact of the obtained products on the human body.

The raw materials for the production of distilled monoglycerides of saturated fatty acids are deeply hydrogenated vegetable oils (palm, sunflower, etc.) and animal fats (pork, beef). When using alkaline catalysts, the content of monoglycerides in the product is (45–55) %, when using sulfuric acid as a solvent – (85–92) % [6]. However, the effect of process conditions on the quality of monoglycerides has not been shown. The problem of production is the use of catalysts, among which there are explosive, flammable, toxic, ineffective substances. Thus, alkali metal alkoxides quickly lose their activity and require special conditions of production, use and storage.

Fractional crystallization and molecular distillation for the isolation of monoglycerides are considered in [9]. The first method involving polar solvents provides a monoglyceride concentration of (72–74) %, non-polar – 91 %. This indicator for molecular distillation is 99 %. However, there are no data on the influence of process parameters on the quality of monoglycerides, in particular, the melting point.

There is a method of monoglycerides obtaining using glycerolysis of triglycerides on the example of methyl stearate. The process is carried out in the presence of an alkaline catalyst (lithium, magnesium, aluminum oxide). A product with a monoglyceride content of at least 78 % is obtained [10]. The disadvantage of the study is the lack of data on the use of other raw materials (salomas, vegetable oils, etc.) and on the impact of process parameters on monoglycerides quality.

In the study [11], the method of monoglycerides obtaining involves the use of sunflower oil, glycerin and enzyme catalyst. Monoglycerides concentration in the product is (50–60) % with glycerolysis duration of (2–3) h and (70–80) % for the duration of (5–6) h. The disadvantage of this method is process duration, high cost of the catalyst ($ 600 kg).

There is a method [12] for monoglycerides obtaining using amidation of linseed oil with diethylenetriamine at temperatures of (413–453) K and different molar ratios of components. The disadvantage of this method is many stages, the need and complexity of mono- and diglycerides separation, the use of diethylenetriamine.

In [13], the possibility of obtaining lactic acid esters with mono-, diglycerides was investigated. A two-stage process was used: esterification of glycerol with lactic acid followed by transesterification of the resulting esters with triglycerides. The disadvantage of this method is the lack of data on the influence of process parameters on the yield and indicators of monoglycerides.

Alkali metal glycerates are effective catalysts for the transesterification of fats. Thus, the authors [14] showed the feasibility of using glycerates in the transesterification process to obtain methyl esters of fatty acids (biodiesel). But no other examples of using these catalysts are given, in particular, for the production of incomplete glycerides.

In [15], data on the high efficiency of alkali metal glycerates in the process of transesterification of palm olein are presented. Potassium glycerate can increase the melting point of test fat as a result of transesterification by more than 15 °C, and common in the industry sodium methylate – by 12 °C. This indicates a greater efficiency of potassium glycerate. It is possible to restore the properties of such a catalyst. However, the possibility of transesterification of fat mixtures and production of incomplete glycerides, which are important industrial processes, has not been shown.

In [16], the conditions and technology of potassium glycerate production were investigated. The possibility of catalyst production directly at the plant under rational conditions is shown: molar concentration of glycerol 60 %, heating duration 4 h. The disadvantage of the study is the lack of data on the use of the catalyst on specific examples (fats with the desired properties, monoglycerides, esters, etc.). The mechanism of transesterification and glycerolysis reactions is similar. Therefore, it is advisable to investigate glycerolysis with potassium glycerate catalyst, which is characterized by high activity, the possibility of production at the plant and safety of production and use.

The review of scientific studies [9–16] indicates that the transesterification process is promising in terms of monoglycerides obtaining. However, there is insufficient data on the impact of process parameters on the yield and quality of the product. The catalysts used (alkoxides, metal oxides, etc.) have a number of disadvantages. These substances are explosive, flammable, quickly and irreversibly lose their activity, require special hermetic storage conditions. The production of such catalysts is also dangerous and requires special equipment. Thus, improving the technology of monoglycerides using a new catalyst, which has advantages over existing catalysts, is an important task.

3. The aim and objectives of the study

The aim of the study is to determine the rational conditions for the production of fatty acid monoglycerides by glycerolysis using potassium glycerate as a catalyst. This will allow developing an advanced technology for the production of monoglycerides using a new catalyst (potassium glycerate). Potassium glycerate is characterized by increased efficiency in the process of transesterification of fats compared to other catalysts (in particular, sodium methylate). The production and use of this catalyst are safer because potassium glycerate is not an explosive or flammable substance and does not form volatile compounds during decomposition and storage.

To achieve the aim, the following objectives were accomplished:

– to investigate the quality of raw material – hydrogenated refined vegetable fat;

– to determine the dependence of the yield and melting point of monoglycerides on the technological parameters of the glycerolysis process and to determine the rational conditions of glycerolysis;

– to investigate the efficiency of monoglycerides obtaining using potassium hydroxide and glycerol mixture as a catalyst.

4. Materials and methods of research

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

The following reagents and materials were used:

– hydrogenated refined vegetable fat according to DSTU 5040 (CAS Number 68334-28-1);

– p.a.-grade potassium hydroxide (basic substance mass fraction not lower than 85.0 %) (CAS Number 1310-58-3);

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

4. 2. Method for determining the quality indicators of raw materials

The melting point is determined according to ISO 6321:2021. The mass fraction of moisture and volatile substances and acid value are determined according to DSTU 4463 (ISO 662, ISO 660, respectively). The peroxide value is determined according to DSTU 4570 (ISO 3960).

4. 3. Method of carrying out the glycerolysis process

A portion of hydrogenated fat was placed in a heat-resistant round-bottom flask mounted on an electric stove. A stirrer and a thermometer were placed in the flask. The potassium glycerate catalyst was added in the amount of 0.5 % in terms of metal and glycerin (in the amount provided in the experimental plan). The flask was connected to a vacuum pump. The process was carried out at a temperature of 180 °C under stirring conditions, in a vacuum. The duration of the process was set according to the experimental plan. After completion of the process, the mass was subjected to adsorption purification (amount of adsorbent 0.5 %, temperature 80 °C, duration 25 min.) and filtered on a paper filter. The mass obtained after filtration was washed with water and dried in a vacuum (90 °C).

4. 4. Method for determining the quality indicators of monoglycerides

The mass fraction of monoglycerides and free glycerol is determined according to DSTU ISO 7366:2014 (ISO 7366). The melting point and acid value are determined according to ISO 6321:2021 and DSTU 4463 (ISO 660), respectively.

4. 5. Research planning and processing of results

The complete second-order factor experiment was used for research planning and mathematical data processing. Processing of the obtained results, construction of graphical dependences were performed in the environment of the Stat Soft Statistica v6.0 package (USA). Two repetitions were performed in each experiment.

5. Results of determining the rational conditions for obtaining fatty acid monoglycerides by glycerolysis

5. 1. Research of quality indicators of hydrogenated refined vegetable fat

Physicochemical indicators of the experimental sample of hydrogenated refined vegetable fat are presented in Table 1.

Table 1

Physicochemical indicators of the experimental sample of fat

Indicator	Characteristic
Melting point, °C	48
Mass fraction of moisture and volatile substances, %	0.08
Acid value, mg KOH/g	0.25
Peroxide value, ½ O mmol/kg	2.8

Thus, hydrogenated refined vegetable fat meets the requirements of DSTU 5040 (CAS Number 68334-28-1).

5. 2. Determination of the dependence of the yield and melting point of monoglycerides on the technological parameters of glycerolysis

The glycerolysis reaction was used to obtain fatty acid monoglycerides. The reaction mass included hydrogenated refined vegetable fat, glycerol and the catalyst (potassium glycerate). Potassium glycerate is a true catalyst for transesterification, in which fatty acid residues are exchanged between glyceride molecules [15]. The study used glycerolysis, in which incomplete glycerides (monoglycerides) are formed. In these compounds, only one fatty acid residue is attached to the glycerol molecule.

The influence of technological parameters of glycerolysis on the yield and melting point of monoglycerides was determined. The following conditions of the factor experiment were used: number of factors – 2, number of levels of factor variation – 5, number of experiments – 25. Factors and intervals of variation:

– x_1 – glycerol concentration (in the reaction mass): from 10 to 50 %;

– x_2 – reaction duration of components of the reaction mixture: from 20 to 300 min.

The response functions are the yield and melting point of monoglycerides. Experimental data were processed using the Stat Soft Statistica v6.0 package (USA). The obtained regression dependence of the monoglycerides yield (y_1) on the technological parameters of the process in real variables has the form:

$$y_1 = 16.414 - 0.323 \cdot x_1 + 0.031 \cdot x_2 + 0.011 \cdot x_1^2. \quad (1)$$

The regression dependence of the melting point of monoglycerides (y_2) on the technological parameters of the process in real variables has the form:

$$y_2 = 54.087 - 0.409 \cdot x_1 + 0.001 \cdot x_2 + \\ + 0.010 \cdot x_1^2 + 0.004 \cdot x_1 \cdot x_2. \quad (2)$$

The significance level of the coefficients of regression equations (p>0.05) and the coefficients of determination, which were 0.969 and 0.972 for dependences (1), (2), respectively, were determined. According to equations (1), (2), the calculated values of the response functions are determined. For equation (1), the standard deviation (absolute conception) is 0.889 %, for equation (2), 0.711 °C. Table 2 presents the matrix of experiment planning, experimental and calculated values of response functions.

The dependences of the yield and melting point of monoglycerides on the technological parameters of the process are graphically presented in Fig. 1, 2.

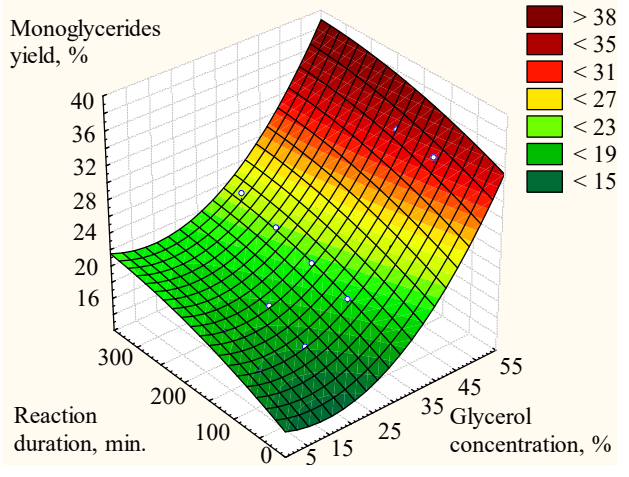

Fig. 1. Dependence of monoglycerides yield on reaction duration and glycerol concentration

According to Table 2, equation (1) and Fig. 1, it is determined that glycerol concentration has a more significant effect on increasing the values of the response function. Starting with a glycerin concentration of 35 %, there is a sharp increase in the monoglycerides yield. Maximum yield values are observed at the maximum concentration of glycerin (50 %).

Table 2

Planning matrix and values of response functions (experimental and calculated)

Experiment number	Factors of variation		Experimental values of response functions		Calculated values of response functions	
	Glycerol concentration, %	Reaction duration, min.	Monoglycerides yield, %	Melting point of monoglycerides, °C	Monoglycerides yield, %	Melting point of monoglycerides, °C
1	10	20	13.8	49.5	15.0	49.9
2	10	90	17.0	50.2	16.8	50.7
3	10	160	18.6	51.3	18.3	51.5
4	10	230	19.2	52.5	19.4	52.1
5	10	300	19.4	51.0	20.2	52.6
6	20	20	15.2	52.5	15.2	49.5
7	20	90	17.4	52.3	17.0	50.4
8	20	160	19.4	52.0	18.4	51.1
9	20	230	19.2	51.2	19.5	51.8
10	20	300	20.1	52.1	20.3	52.3
11	30	20	19.0	51.5	17.6	51.3
12	30	90	20.7	52.0	19.4	52.1
13	30	160	22.1	52.4	20.8	52.9
14	30	230	23.7	53.0	22.0	53.5
15	30	300	25.2	53.8	22.7	54.0
16	40	20	20.8	53.5	22.3	55.0
17	40	90	21.3	55.0	24.1	55.9
18	40	160	22.7	55.8	25.6	56.7
19	40	230	23.9	57.1	26.7	57.3
20	40	300	25.6	58.0	27.4	57.8
21	50	20	30.0	59.6	29.3	60.9
22	50	90	32.9	61.5	31.1	61.7
23	50	160	33.5	63.0	32.5	62.5
24	50	230	33.6	64.1	33.6	63.1
25	50	300	35.5	65.5	34.4	63.6

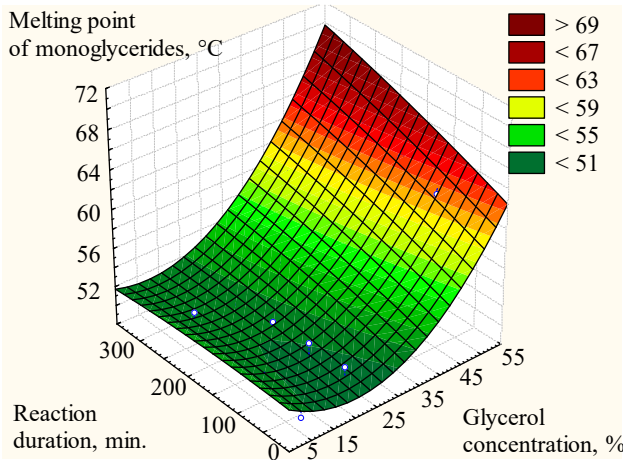

Fig. 2. Dependence of melting point of monoglycerides on reaction duration and glycerol concentration

Therefore, glycerol concentration also has the most significant effect on the melting point of monoglycerides, starting from 35 %. Up to 35 %, the duration has almost no effect on the melting point. The duration has a negligible effect on the response function. The maximum values of melting point are observed at a glycerol concentration of 50 %. According to Table 2, starting from the duration of 90 min., the maximum values of the response function are observed and the growth of these values slows down. Therefore, the rational conditions for glycerolysis are a glycerol concentration of 50 % and a process duration of 90 min. Under these conditions, the monoglycerides yield was 32.9 %, the melting point – 61.5 °C. The mass fraction of free glycerol in monoglycerides was 1.0 %, the acid value – 2.2 mg KOH/g.

5. 3. Investigation of the efficiency of monoglycerides obtaining using potassium hydroxide and glycerol mixture

The research process of glycerolysis under certain rational conditions was carried out, using potassium hydroxide and glycerol mixture as a catalyst. The amount of potassium hydroxide was 0.5 % in terms of metal (as a solution in glycerol with a concentration of 10 %). The monoglycerides yield was 30.1 %, melting point of monoglycerides – 59 °C. The use of potassium glycerate as a catalyst is more effective because it allows you to get a higher quality product.

6. Discussion of the results of studying the dependence of the yield and melting point of monoglycerides on the technological parameters of glycerolysis

The technology of fatty acid monoglycerides obtaining by glycerolysis, which involves the reaction of hydrogenated vegetable fat with glycerol in the presence of potassium glycerate, has been studied. According to Table 2, Fig. 1, 2 and equations (1), (2), the rational conditions of glycerolysis were determined: glycerol concentration 50 %, process duration 90 min. Under these conditions, the monoglycerides yield was 32.9 %, melting point – 61.5 °C.

During glycerolysis, fat molecules react with glycerol. This removes and redistributes fatty acid residues. If there is a sufficient amount of glycerol in the mass, the equilibrium of the process tends towards the formation of monoglycerides. The chemical nature of the glycerolysis mechanism is similar to the mechanism of transesterification. When using alkaline catalysts (in particular, potassium glycerate), glycerate anion is formed as an intermediate product, which is a true homogeneous catalyst for the exchange of fatty acid residues. Therefore, the introduction of potassium glycerate is appropriate and effective.

Increasing the glycerol concentration and process duration leads to an increase in the values of response functions. Glycerol concentration has a greater effect. The highest values of monoglycerides yield are observed at a glycerol concentration of 50 %. When increasing the duration from 20 to 300 min., the greatest increase in yield is only at the transition from 20 to 90 min. In the future, the growth of yield slows down. The maximum melting point also corresponds to a glycerol concentration of 50 %. Up to a glycerol concentration of 35 %, the reaction time has almost no effect on the melting point of monoglycerides (Fig. 2). At a glycerol concentration of 50 % with increasing duration from 90 to 300 min., the melting point increases by only 4 °C. Therefore, it is rational to use the maximum value (50 %). The rational process duration is 90 min.

The obtained results make it possible to predict the yield and melting point of the product depending on the technological parameters of glycerolysis.

The cost of the common catalyst sodium methylate is $ 9.5/kg, potassium glycerate – $ 4.3/kg. Therefore, the use of potassium glycerate will increase the profitability of the technological process.

Because potassium glycerate is not an explosive or flammable substance, does not form toxic dust and does not contain volatile components, the use of this catalyst will make the process safer. Catalyst production technology also does not provide explosion or fire hazards, unlike other catalysts (metal alkoxides), and does not require special complex equipment. Therefore, it is possible to manufacture the catalyst at the plant. After the loss of catalytic activity during storage, it is possible to restore the properties of the catalyst. Transesterification with fresh catalyst allows increasing the melting point of test fat by 17.4 °C, with regenerated catalyst – by 16.7 °C. The technology is simplified due to the fact that in this case there is no need for complex loading equipment for alkali metal alcoholates [15].

The limitation of using the results of the work is the need to determine the concentration of the basic substance in the catalyst. During storage, potassium glycerate decomposes into glycerol and potassium hydroxide, which reduces the efficiency of the catalyst.

The disadvantage of the study is taking into account the impact of only glycerol concentration and process duration on the yield and melting point of monoglycerides. But the process is also affected by temperature, catalyst concentration. It is also advisable to investigate these parameters.

Promising areas of research are the use of different types of raw materials (including production waste, of low quality), the study of various glycerolysis parameters in a wide range of variations. This will contribute to the innovative development of production technology for monoglycerides, the demand for which is consistently high.

7. Conclusions

1. As a result of research of raw materials quality (hydrogenated refined vegetable fat), fat indicators were de-

termined. The melting point was 48 °C, mass fraction of moisture and volatile substances – 0.08 %, acid value – 0.25 mg KOH/g, peroxide value – 2.8 ½ O mmol/kg. Raw materials meet the requirements of DSTU 5040 (CAS Number 68334-28-1).

2. On the basis of experimental research and processing of the obtained data, rational conditions of glycerolysis were determined: duration (90 min.) and glycerol concentration (50 %). Under these conditions, the monoglycerides yield was 32.9 %, melting point – 61.5 °C. The mass fraction of free glycerol in monoglycerides was 1.0 %, acid value – 2.2 mg KOH/g.

3. As a result of the study of monoglycerides obtaining efficiency using a mixture of potassium hydroxide and glycerol as a catalyst, it was found that the monoglycerides yield was 30.1 %, melting point – 59 °C. The use of potassium glycerate catalyst is more effective than potassium hydroxide solution in glycerol.

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