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ESTABLISHING THE INFLUENCE OF SUNFLOWER OIL QUALITY INDICATORS ON THE EFFICIENCY OF THE TRANSESTERIFICATION CATALYST

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The object of the study is the process of chemical transesterification of sunflower oil with increased mass fraction of moisture and acid value.

Transesterification technology allows obtaining fats with the necessary physico-chemical properties without changing the fatty acid composition. The problem of using alkaline catalysts is rapid deactivation under the influence of impurities present in the fatty raw material. The greatest influence is exerted by moisture and fatty acids.

Transesterification of sunflower oil with increased mass fraction of moisture and acid value in the presence of potassium glyceroxide, which is more resistant to deactivating impurities, was studied. Oil parameters: peroxide value 0.18 ½ O mmol/kg, acid value 0.08 mg KOH/g, mass fraction of moisture 0.05%, crystallization temperature –18.38°C.

Oil samples with mass fractions of moisture from 0.1 to 0.75% (obtained by adding water) were stored for two months under the following conditions: temperature (20±2)°C, air humidity not more than 70%. After storage, the acid value was measured in each sample and transesterification was performed. The efficiency of the process was assessed by the oil crystallization temperature.

The maximum values of oil indicators at which the potassium glyceroxide catalyst is effective were established: mass fraction of moisture 0.4%, acid value 0.28 mg KOH/g. At the same time, the crystallization temperature of sunflower oil is –3.95°C, which corresponds to the maximum increase in the crystallization temperature of oil as a result of transesterification.

The results obtained make it possible to effectively transesterify sunflower and other types of non-standard quality oils without preliminary treatment. This will reduce the cost of production and increase the profitability of enterprises (depending on the indicators of non-standard raw materials)

Keywords: transesterification catalyst, potassium glyceroxide, chemical transesterification, sunflower oil, crystallization temperature

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1. Introduction

Transesterification (randomization) is the exchange of fatty acid radicals in glyceride molecules. This process allows to obtain surfactants, esters of fatty acids and low molecular weight alcohols (the basis of biodiesel), components of lubricants, etc. [1]. The most widely used chemical catalysts for transesterification are alkaline catalysts – alkoxides (sodium methoxide and sodium ethoxide), which allow to reduce the process temperature from 250°C and above to (50–120)°C [2]. The initial raw material must be refined and deodorized to free it from fatty acids, moisture and peroxides. The greatest influence on the deactivation of catalysts is the presence of moisture and free fatty acids. The presence of 0.01% moisture in fatty raw materials leads to the deactivation of sodium methoxide in the amount of 0.3 kg per 1 ton of fat. The presence of 0.05% fatty acids leads to the deactivation of sodium methoxide in the amount of 0.1 kg per 1 ton of fat [3]. New alternatives have been investigated, such as the use of a homogeneous acid catalyst instead of homogeneous alkalis. However, inorganic acids require a long reaction time, which negatively affects the economic component of the process [4].

For transesterification, fatty raw materials with a mass fraction of moisture and volatile substances of no more than 0.015%, free fatty acids – no more than 0.05% are used. For example, the standard value of the mass fraction of moisture and volatile substances for refined, deodorized, frozen sunflower oil is no more than 0.1%. That is, this indicator for oil sent for transesterification should be 6.7 times lower than the standard [2]. Alternative, more stable transesterification catalysts are alkali metal glyceroxides [5]. Potassium glyceroxide is effective in the process of transesterification of palm olein with increased oxidation rates (peroxide value 12.7 ½ O mmol/kg; anisidine value 10.4 c. u.) [6].

An important issue is the industrial use of fatty raw materials of substandard quality (with increased spoilage rates): waste oils and fats, waste from oil and fat production, etc. [7]. Utilization of such components is unprofitable and causes environmental pollution. Alkoxides for transesterification of such raw materials are ineffective. At the same time, the choice of raw materials has a significant impact, for example, on the production of biodiesel, since the initial raw materials account for 70% of the product cost [3].

Thus, the use of alkali metal alkoxides as transesterification catalysts involves thorough purification of fatty raw materials from impurities. Research into methods for effective transesterification of fatty raw materials of substandard quality is relevant. This makes it possible to reduce the amount of waste and increase the profitability of enterprises. A way to solve the problem of using non-standard raw materials for transesterification is to use alkali metal glyceroxides as catalysts that are resistant to foreign impurities in fats. Promising are studies of transesterification of oils, in particular, sunflower oil, with an increased mass fraction of moisture and acid value using glyceroxides.

2. Literature review and problem statement

The current problem for oil and fat enterprises is the use of fatty raw materials of non-standard quality (waste, used oils and fats, etc.) for the production of various types of products, in particular, in transesterification processes. Modern research involves the search for ways to effectively use raw materials with non-standard indicators for the production of high-quality transesterification products, in particular, by developing and implementing new catalysts. Thus, in the work [8], the transesterification reaction of used cooking oil with methanol in the presence of the K_2CO_3-Al_2O_3 catalyst was considered. The optimal reaction conditions were determined (the product yield was 98.7%): catalyst loading of 5.8% at 25°C for 120 min. with a molar ratio of alcohol:fat raw materials of 9:1. However, the influence of moisture content and free fatty acids in the raw materials on the efficiency of the process was not shown. The objective reason for this could be the lack of a number of samples of the same oil with different moisture content and free fatty acids.

In the work [4], two different methods of transesterification of used cooking oil with a high content of free fatty acids were compared. The first method was transesterification using sulfuric acid as a catalyst. The optimal conditions were the molar ratio of methanol to oil of 52:1 and a catalyst loading of 3.5%; the degree of conversion was 96.2% after 1 h at 65°C. The second method was heterogeneously catalyzed transesterification using cement dust. In this case, the optimal conditions were: time 6 h, molar ratio of methanol to oil of 18:1, catalyst loading of 2% at 65°C. This resulted in the degree of conversion of 98.8%. The comparison of the two methods showed that the use of cement dust is more economically feasible than the homogeneous catalyst. But there is no data on the influence of the content of free fatty acids and moisture on the completeness of the process. This is due to the fact that the fat waste was used, which contained various foreign impurities, and it is difficult to vary only one parameter, for example, the content of free fatty acids.

The authors of [9] presented data on the transesterification of soybean oil with a high content of free fatty acids (80.65%) with methanol. Acid oils are promising as raw materials for the synthesis of methyl esters of fatty acids. The maximum degree of conversion was 98.06%. But no results are given on the dependence of the efficiency of the transesterification process, the degree of conversion on the content of free fatty acids and moisture in the initial fatty raw material. One of the objective reasons may be the duration of the study when obtaining the same sample of oil with different contents of fatty acids (subjecting the oil to hydrolytic spoilage).

In the work [10], the transesterification of used cooking sunflower oil with methanol using hazelnut shell ash as a catalyst was investigated. The highest content of the main product (98%) was achieved at catalyst loading of 5%, molar ratio of methanol to oil of 12:1, and reaction time of 10 min. However, the influence of the quality indicators of the starting raw materials on the activity of the catalyst and the efficiency of the process was not presented. This is due to the fact that the aim of the work was primarily to establish the efficiency of the new catalyst, without considering additional conditions for its use.

The process of transesterification of palm oil with oleic acid (to simulate sour oil) with methanol in the presence of an acid-base bifunctional catalyst based on CaO from solid waste from blast furnace dust was investigated [11]. Optimal experimental conditions: methanol to oil ratio 15:24, catalyst dosage 7.96%, reaction temperature 148.95°C, reaction duration 2 h, with the addition of oleic acid 4%. The product yield was 87.67%. However, there is no data on how different concentrations of added oleic acid affect the catalyst activity and the yield of the final product. The reason for this may be the difficulty of operationally assessing the efficiency of the transesterification process in the presence of the applied catalyst.

A highly active bifunctional catalytic system for the conversion of oilseeds with a high content of free fatty acids and waste cooking oil into fatty acid methyl esters has been described [12]. The CaO/tungsten-based hydroxyapatite catalyst containing basic and acidic sites was prepared by adding 12-phosphotungstic acid to powdered bovine bone followed by calcination at 750°C. Thus, the process of obtaining esters from oilseeds with a high content of free fatty acids (24.38 mg KOH/g oil) was catalyzed. It was found that the conversion of waste cooking oil into esters by simultaneous transesterification/esterification can be achieved in 5 h at 100°C and methanol:oil ratio of 6:1. However, there is no data on the effect of the acid value of the starting oil on the activity of the catalyst. In addition, non-standard and waste oils may contain an increased concentration of moisture, which is also not considered in this work. The reason for this is, first of all, the development and testing of a new catalyst, the preparation of which is quite difficult. Such studies require identical catalyst samples and a long time.

In the work [13], the transesterification of sour soybean oil was investigated using lithium metasilicate and its mixtures with ABW zeolite as catalysts. Lithium metasilicate (Li_2SiO_3) and ABW zeolite were synthesized hydrothermally at 150°C for one day using diatomite and LiOH as starting materials. These solid catalysts were successfully used to catalyze the transesterification of sour soybean oil in excess methanol at 60°C for the production of esters. Soybean oil was enriched with oleic acid to obtain an acid value of 0.54 mg KOH/g of oil. When loading the catalyst into the oil to 30% by weight, the product yield reached 95% in 1 h at 60°C. However, there is no data on the dependence of the activity of the developed catalysts on the acid value of the initial oil. These studies require a long time and stability of the composition and properties of the catalysts. Another disadvantage is the high catalyst loading rate.

The production of ethyl esters from high-acid waste cooking oil containing 18% free fatty acids was investigated. A simultaneous transesterification and esterification process was applied using a number of heterogeneous bifunctional catalysts containing strontium, zinc, and aluminum [14]. It was found that the catalyst with strontium to zinc ratio of 2.6:1 performed best in the transesterification of glycerides and esterification of fatty acids in waste oil simultaneously. The catalyst demonstrated higher catalytic activity with high-acid waste cooking oil, with conversion of 95.7%. The optimal conditions were: molar ratio of ethanol to waste oil of 10:1, catalyst dosage of 15% and reaction time of 5 h at reaction temperature of 75°C. However, the efficiency of the process and the degree of conversion under conditions of different concentrations of free fatty acids and moisture in the initial oil were not shown. The reason for this is the complexity and time required to obtain samples of the same oil with different values of free fatty acid content. A reliable operational method for assessing the transesterification efficiency is also needed.

In the work [15], the transesterification of waste cottonseed oil with ethanol was investigated. The oil was subjected to ultrasound esterification and transesterification in the presence of immobilized lipase Thermomyces lanuginosus as a catalyst. The effect of various parameters (molar ratio of ethanol to oil, reaction temperature, catalyst concentration, reaction time, ultrasound amplitude, frequency and duty cycle) on the efficiency of the process was investigated. However, the effect of oil quality parameters on catalyst activity and conversion degree was not shown. The reason for this is the difficulty of obtaining samples of the same oil with different acid values, as well as the focus of the study on the principle possibility of using waste oil in the transesterification reaction with an enzyme catalyst.

The production of esters from waste cooking oil using a magnetic nanocatalyst (CaO/HAP/MnFe@K) obtained from eggshell/chicken bones was investigated [16]. The highest degree of conversion using the catalyst was 99.10%, which was achieved at catalyst content of 2.97%, reaction time of 175.72 min. and temperature of 67.72°C (optimal conditions). However, there is no data on the influence of oil quality parameters, in particular, moisture content and free fatty acids, on the efficiency of the catalyst used. The reason for this is that the study was aimed at proving the efficiency of the newly developed catalyst, without taking into account the composition and quality of the initial fatty raw material.

Transesterification of waste lard in supercritical alcohols was studied [17]. The ratio of waste lard to alcohol (wt/wt) was changed from 1:1.5 to 1:2.5, the temperature was considered from 220 to 290°C with an interval of 10°C. The products were analyzed using gas chromatography with mass spectrometry. It was found that the apparent activation energy for supercritical ethanol was lower than that for supercritical methanol. However, there is no data on how the parameters of lard, in particular, moisture content and acid value, affect the efficiency, process speed and degree of conversion during the reaction. The reason for this is the complexity of the operational assessment of the process efficiency and obtaining fat samples with different values of quality indicators.

In the work [18], data are provided on increasing the efficiency of the transesterification process of waste oil with methyl acetate. Potassium methoxide was used as a catalyst. The process temperature range (30–50) °C, molar ratios of oil to methyl acetate from 1:4 to 1:14, and catalyst concentration (0.5–1.5) % were investigated. However, there is no data on the influence of the quality indicators of the initial oil on the activity of the catalyst and the efficiency of transesterification. The reason for this is the complexity of analyzing the final product and operational control of the completeness of the reaction.

The efficiency of the potassium glyceroxide catalyst in the transesterification reaction of palm olein with increased oxidation rates was investigated [6]. Olein was heated at temperature of 90°C in order to increase the oxidation rates, after which the corresponding samples were subjected to transesterification. The difference in melting temperatures of the initial and transesterified palm olein was used as a parameter of the efficiency of the process. The maximum limit values of the oxidation rates at which the process is effective were established: peroxide value 12.7 ½ O mmol/kg, anisidine value 10.4 c. u. The difference in melting temperatures of olein in this case is 12.1°C, which indicates the efficiency of the process. The qualitative indicators of the obtained interesterified olein indicate compliance with DSTU 4336 (CAS Number 97593-46-9): melting point 34.5°C, peroxide value 1.2 ½ O mmol/kg, anisidine value 1.0 c. u. But the greatest influence on the activity of alkaline catalysts is exerted by moisture and free fatty acids present in the initial fat. Therefore, it is advisable to investigate the influence of these impurities on the efficiency of the interesterification process.

Thus, existing studies have considered the processes of transesterification of oils and fats of non-standard quality, in particular, with an increased content of free fatty acids and moisture. An important task is to find opportunities for using such raw materials in the production of fatty acid esters for obtaining biodiesel fuel. To this end, existing studies have developed a number of new multicomponent catalysts, the preparation of which is complex and multistage, and established optimal process parameters for different types of raw materials and catalysts. But the influence of the mass fraction of moisture and acid value of the initial raw material on the efficiency of transesterification processes remains an unresolved issue. In addition, it is necessary to develop a technology for transesterification of raw materials of non-standard quality using a safe, effective catalyst, the preparation of which does not require complex equipment and special conditions. Therefore, it is advisable to establish the influence of the mass fraction of moisture and acid value of oil, in particular, sunflower oil, on the efficiency of a promising, resistant to the presence of impurities, potassium glyceroxide catalyst.

3. The aim and objectives of the study

The aim of the study is to determine the influence of the mass fraction of moisture and acid value of sunflower oil on the efficiency of the potassium glyceroxide transesterification catalyst. This will allow predicting the completeness of the transesterification process depending on the initial values of the mass fraction of moisture and acid value in the initial fatty raw material.

To achieve this aim, the following tasks were solved:

– to establish the maximum values of the mass fraction of moisture and acid value of sunflower oil at which the transesterification catalyst potassium glyceroxide is effective;

– to determine the quality indicators of sunflower oil, transesterified under the conditions of specified maximum values of mass fraction of moisture and acid value.

4. Materials and methods

4. 1. The object and hypothesis of the study

The object of the study is the process of chemical transesterification of sunflower oil with increased mass fraction of moisture and acid value. The main hypothesis of the study is that the influence of the mass fraction of moisture and acid value of the initial sunflower oil on the activity of the potassium glyceroxide catalyst is characterized by the change in the crystallization temperature of the transesterified oil.

It is assumed that an increase in the mass fraction of moisture and acid value in sunflower oil reduces the activity of the potassium glyceroxide transesterification catalyst. The following simplification is adopted – other indicators of deterioration of sunflower oil (in particular, the content of oxidation products) are not taken into account when conducting research according to the experimental plan. In this work, standard methods for studying oils and fats are used.

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

The following materials and equipment were used during the experimental research:

– refined, deodorized, frozen sunflower oil, according to DSTU 4492 (CAS Number 8001-21-6);

– differential scanning calorimeter DSC, model Q20 (USA).

4. 3. Methods for determining the quality indicators of experimental sunflower oil

Sunflower oil indicators were determined according to standard methods used for oils and fats: mass fraction of moisture and volatile matter – ISO 662:2016; acid value – ISO 660:2020; peroxide value – ISO 3960:2017. Crystallization temperature was determined by differential scanning calorimetry (DSC) according to ISO 11357-1:2023.

4. 4. Sunflower oil transesterification methods

Transesterification of sunflower oil samples was carried out according to [19] under the following conditions: temperature 110°C, duration 1.5 h, catalyst potassium glyceroxide concentration 0.45% by weight of oil.

4. 5. Planning and processing of research results

The work involved processing the data set regarding different mass fractions of moisture and corresponding values of acid value in sunflower oil samples, as well as crystallization temperatures of each interesterified oil sample. The processing of research results, calculation of the regression equation, and construction of graphical dependence were carried out using the Stat Soft Statistica v6.0 package (USA) (Basic Statistics module). In each experiment, two repetitions were performed.

5. Results of establishing the influence of sunflower oil indicators on the transesterification catalyst efficiency

5. 1. Establishing the maximum values of sunflower oil indicators at which the catalyst is effective

The physico-chemical indicators of the initial sample of refined, deodorized, frozen sunflower oil were determined (Table 1).

Table 1

Physico-chemical indicators of the initial sample of sunflower oil

Name of indicator	Characteristics	Norm according to DSTU 4492
Moisture and volatile matter mass fraction, %	0.05	0.10
Acid value, mg KOH/g	0.08	0.5
Peroxide value, ½ O mmol/kg	0.18	10.0

The crystallization temperature of sunflower oil by the DSC method is –18.38°C. Thus, the experimental sunflower oil meets the requirements of DSTU 4492 for this type of oil.

The influence of the mass fraction of moisture and the acid value of sunflower oil on the efficiency of the transesterification process using potassium glyceroxide as a catalyst was determined. In the work [19], it was established that an increase in the crystallization temperature of sunflower oil as a result of transesterification from –18.38°C to –4.1°C (i.e. by 14.28°C) corresponds to the change in the triglyceride composition of the oil outside the measurement error. In 12 out of 14 detected triglycerides, the difference between the content in the initial and transesterified sunflower oil was greater in modulus than the absolute measurement error. Thus, as a criterion for the efficiency of the transesterification process (response function), the crystallization temperature of transesterified sunflower oil (y, °C) was considered. Thus, the influence of the mass fraction of moisture and the acid value of the initial sunflower oil on the crystallization temperature of the transesterified oil was determined. Samples weighing 50 g each were taken from the experimental sunflower oil. Water was added to the oil samples until the mass fraction of moisture was reached from 0.1 to 0.75%. The oil samples were stored for two months under standard laboratory conditions: temperature (20±2)°C, air humidity not more than 70%, in the glass, non-hermetically sealed container, in a darkened place. After storage, the acid values in the samples increased, which was determined by analysis, and each sample was subjected to transesterification.

Ranges of variation of input variables: mass fraction of moisture (x_1): (0.05 – 0.75) %; acid value (x_2): (0.11 – 0.57) mg KOH/g.

The results were processed using the Stat Soft Statistica v6.0 package (USA) using the Basic Statistics module.

Table 2 shows the values of mass fraction of moisture and acid value for all points of the experiment, as well as the experimental and calculated values of the response function.

Table 2

Values of mass fraction of moisture and acid value for all points of the experiment, as well as experimental and calculated values of the response function

Experiment No.	Values of sunflower oil indicators		Crystallization temperature of sunflower oil, °C	
	Mass fraction of moisture (x_1), %	Acid value (x_2), mg KOH/g	Experimental values (y_e)	Calculated values (y_c)
1	0.05	0.11	–3.41	–3.35
2	0.1	0.13	–3.02	–3.22
3	0.15	0.14	–3.07	–2.98
4	0.2	0.17	–3.12	–3.12
5	0.25	0.19	–3.25	–3.16
6	0.3	0.23	–3.81	–3.67
7	0.35	0.24	–3.91	–3.63
8	0.4	0.28	–3.95	–4.33
9	0.45	0.33	–4.95	–5.38
10	0.5	0.35	–5.96	–5.79
11	0.55	0.39	–6.53	–6.81
12	0.6	0.45	–8.97	–8.53
13	0.65	0.48	–9.83	–9.50
14	0.7	0.52	–10.75	–10.87
15	0.75	0.57	–12.52	–12.70

The results of the analysis of variance, which confirmed the adequacy of the calculated mathematical model, are given in the Table 3.

The dependence of the crystallization temperature of interesterified sunflower oil (y, °C) on the mass fraction of moisture (x_1, %) and acid value (x_2, mg KOH/g) of the initial oil, in natural variables, has the form

$$y = -2.431 + 11.823 \cdot x_1 - 12.323 \cdot x_2 - 28.329 \cdot x_1 \cdot x_2. \quad (1)$$

Based on the regression analysis (1), in the environment of the StatSoftStatistica v6.0 package (USA), the maximum values of the oil indicators at which the potassium glyceroxide catalyst is effective were established: mass fraction of moisture 0.4%, acid value 0.28 mg KOH/g. At the same time, the crystallization temperature of sunflower oil is –3.95°C, i.e. the difference between the crystallization temperatures of the initial and transesterified oil is 14.43°C. These oil parameters are the maximum under which the activity of potassium glyceroxide is preserved. A further increase in the mass fraction of moisture and the acid value of the oil leads to a lower efficiency of the process, which is reflected in a less intense increase in the crystallization temperature of the transesterified oil. In the work [19], the change in the triglyceride composition of sunflower oil beyond the measurement error as a result of transesterification was confirmed, which corresponds to the increase in the crystallization temperature of the transesterified oil to –4.1°C, i.e. by 14.28°C. Therefore, the transesterification process is effective, as a result of which the crystallization temperature of the oil increases to this level.

The graphical dependence of the crystallization temperature of transesterified sunflower oil on the mass fraction of moisture and acid value in the initial oil in the form of a response surface is shown in the Fig. 1.

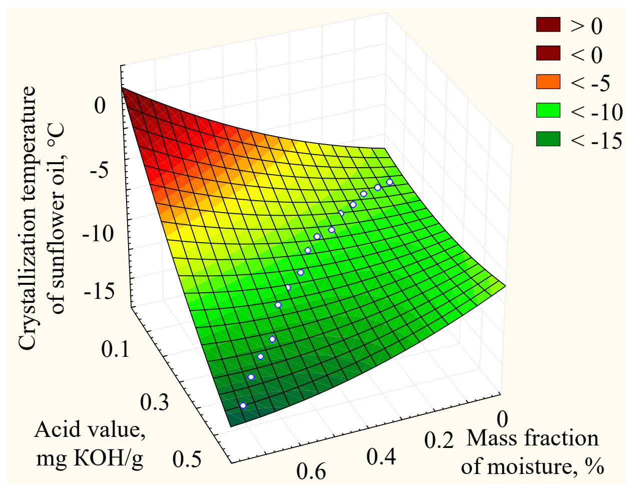

Fig. 1. Dependence of the crystallization temperature of interesterified sunflower oil on the mass fraction of moisture and acid value in the initial oil

Table 3

Results of analysis of variance

Factor	Sum of squares, SS	Degrees of freedom, df	Mean square, MS	F-value	Significance level, p-value
Free term of the equation	0.4512	1	0.4512	5.2684	0.042381
Mass fraction of moisture, %	0.6368	1	0.6368	7.4354	0.019691
Acid value, mg KOH/g	0.0878	1	0.0878	1.0251	0.033050
Mass fraction of moisture, %·Acid value, mg KOH/g	1.3745	1	1.3745	16.0482	0.002065
Error	0.9421	11	0.0856	–	–
Coefficient of determination $R^2 = 0{,}993$. Adjusted coefficient of determination $R^2_{adj} = 0{,}992$.					

Based on equation (1), Table 2 and Fig. 1, it was found that increasing the mass fraction of moisture and acid value of the initial oil reduces the efficiency of the transesterification process using potassium glyceroxide as a catalyst. At the same time, the increase in the crystallization temperature of the transesterified oil becomes less significant. Under the conditions of mass fraction of moisture of 0.4% and acid value of 0.28 mg KOH/g, transesterification of the oil is effective, which is characterized by an increase in the crystallization temperature of the transesterified oil to –3.95°C (by 14.43°C). The results obtained confirm the high efficiency of potassium glyceroxide as a catalyst for the transesterification of oils with non-standard quality indicators.

5. 2. Determination of quality indicators of interesterified sunflower oil

The physico-chemical indicators of the oil transesterified with the potassium glyceroxide catalyst were determined. The oil before transesterification had the determined maximum values of the mass fraction of moisture (0.4%) and acid value (0.28 mg KOH/g), at which the catalyst is effective. The obtained research results are presented in the Table 3.

Table 3

Physico-chemical indicators of interesterified sunflower oil

Name of indicator	Characteristics
Moisture and volatile matter mass fraction, %	0.06
Acid value, mg KOH/g	0.05
Peroxide value, ½ O mmol/kg	0.15

Thus, the transesterified sunflower oil meets the requirements of DSTU 4492 for refined, deodorized, frozen sunflower oil (Table 1). Therefore, transesterification with an alkaline catalyst potassium glyceroxide allowed for an effective transesterification process and a reduction in the mass fraction of moisture and acid value of the oil.

6. Discussion of the results of establishing the influence of oil indicators on the efficiency of the transesterification catalyst

The technology of transesterification of sunflower oil with increased mass fraction of moisture and acid value using potassium glyceroxide catalyst was investigated. In the experimental transesterification processes, oil samples with values of mass fraction of moisture from 0.1 to 0.75%, acid value – from 0.11 to 0.57 mg KOH/g were used. As a parameter of process efficiency, the crystallization temperature of transesterified sunflower oil was investigated.

By analyzing the dependence (1), Table 2 and Fig. 1, the maximum values of the initial sunflower oil indicators were established, at which the potassium glyceroxide catalyst is effective: mass fraction of moisture 0.4%, acid value 0.28 mg KOH/g. At the same time, the crystallization temperature of transesterified sunflower oil is -3.95°C, i.e. there was an increase of 14.43°C, which indicates the efficiency of the transesterification process.

The physico-chemical indicators of the oil, transesterified with the potassium glyceroxide catalyst, were determined. The oil before transesterification had the specified maximum values of the mass fraction of moisture (0.4%) and acid value (0.28 mg KOH/g), at which potassium glyceroxide retains activity. The mass fraction of moisture and volatile substances of the transesterified oil was 0.06%, the acid value was 0.05 mg KOH/g, and the peroxide value was 0.15 ½ O mmol/kg (Table 3). Therefore, the obtained transesterified oil meets the requirements of DSTU 4492 for refined, deodorized, frozen sunflower oil.

Foreign impurities present in the initial fatty raw material subjected to transesterification deactivate the catalysts, which leads to a decrease in the efficiency of the process and the formation of by-products. For transesterification in the presence of industrial alkoxide-based catalysts (sodium methoxide, sodium ethoxide), raw materials with a mass fraction of moisture and acid value significantly lower than standard values are used. Alternative transesterification catalysts that are more resistant to the presence of impurities are alkali metal glyceroxides.

Increasing the mass fraction of moisture and acid value in the initial sunflower oil slows down the increase in the crystallization temperature of the transesterified oil. This is explained by the decomposition of the potassium glyceroxide catalyst under the action of moisture and free fatty acids, and, accordingly, a decrease in the efficiency of the process. Under the action of moisture, potassium glyceroxide decomposes into alkali and glycerin, under the action of free fatty acids – into soap and glycerin.

The obtained scientific results will allow predicting the efficiency of using the potassium glyceroxide catalyst and the completeness of the transesterification process depending on the indicators of the initial fatty raw material (Table 2, equation (1), Fig. 1). The obtained dependencies can be used in the production processes of edible fats for various purposes, fatty acid esters as a basis for biodiesel fuel, surfactants, etc.

In the works [4, 6, 8–18] data on the processes of transesterification of fatty raw materials with non-standard indicators, low quality, in particular, with an increased content of moisture and free fatty acids are presented. In order to effectively use such raw materials, a number of catalysts of multicomponent composition have been developed, effective under the conditions of using non-standard raw materials. Thus, existing studies have confirmed that the increased content of moisture and free fatty acids causes deactivation of alkaline catalysts, and new technologies are needed for processing such raw materials. In the work [6], the efficiency of the potassium glyceroxide catalyst in the transesterification reaction of palm olein with increased oxidation indicators (peroxide value 12.7 ½ O mmol/kg, anisidine value 10.4 c. u.) was established. Thus, the feasibility of using this catalyst for transesterification of raw materials with non-standard indicators has been experimentally confirmed. But there is not enough data on the dependence of the activity of transesterification catalysts on the moisture content and free fatty acids in the raw material. After all, these impurities have the greatest impact on the deactivation of the catalyst. In addition, there is a need to develop new transesterification catalysts that are resistant to impurities and have a simple production technology.

The limitation of the application of the obtained results is that only one type of oil was considered – sunflower. The obtained data can be extended to other types of oils that are close in triglyceride composition to sunflower oil. But the use of oils and fats with other properties (for example, refractory oils, animal fats, etc.) requires additional research. Also, the use of the research results is limited by the intervals of the values of the input variables (mass fraction of moisture and acid number of the initial oil).

The disadvantage of the work is the study of only the mass fraction of moisture and acid value of the oil, without taking into account the oxidation indicators, which also increase during oil storage and affect the deactivation of the catalyst. A promising direction of work is the study of the processes of transesterification in the presence of potassium glyceroxide of other oil and fat raw materials with increased spoilage rates, for example, solid vegetable oils, animal fats, etc. After all, the use of oils and fats with different compositions and properties to obtain transesterified fat systems is of great importance for industry.

7. Conclusions

1. The maximum values of the initial sunflower oil indicators at which the potassium glyceroxide transesterification catalyst is effective were established: mass fraction of moisture 0.4%, acid value 0.28 mg KOH/g. The efficiency of transesterification was assessed by the crystallization temperature of the oil. In this case, the crystallization temperature of sunflower oil was –3.95°C, i.e. there was an increase of 14.43°C, which indicates a high activity of the catalyst in this process.

2. The qualitative indicators of the transesterified sunflower oil were determined, which before transesterification had the specified maximum values of the mass fraction of moisture and acid value. The mass fraction of moisture and volatile substances was 0.06%, the acid value was 0.05 mg KOH/g, and the peroxide value was 0.15 ½ O mmol/kg. Therefore, the obtained transesterified oil meets the requirements of DSTU 4492 for refined, deodorized, frozen sunflower oil. Transesterification in the presence of an alkaline catalyst of potassium glyceroxide allowed to effectively carry out the transesterification process and reduce the mass fraction of moisture and acid value of the oil.

Conflict of interest

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

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The study was conducted without financial support.

Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

Authors' contributions

Mykola Korchak: Resources, Conceptualization, Data Curation, Project administration. **Vladyslav Knysh:** Methodology, Data Curation, Validation. **Anatolii Shostia:** Resources, Investigation, Supervision. **Svitlana Usenko:** Conceptualization, Investigation, Writing – original draft. **Yevgenia Hmelnitska:** Formal analysis, Writing – review & editing. **Zoja Rachynska:** Writing – review & editing, Validation. **Volodymyr Viaskov:** Methodology, Formal analysis. **Ivan Kostenko:** Visualization, Software. **Vita Glavatchuk:** Writing – original draft, Visualization. **Sergii Zygin:** Software, Formal analysis.

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