The object of research is the process of chemical transesterification of palm olein with increased oxidation indicators in the presence of potassium glyceroxide catalyst.

Transesterification is an important method of fat modification. The use of fats with increased oxidation indicators leads to the deactivation of common catalysts and a decrease in the efficiency of the process. There is a need to increase the dosage of catalysts, increase the process temperature, which negatively affects the product quality.

An alternative transesterification catalyst (potassium glyceroxide) was used for the transesterification of palm olein with increased oxidation indicators.

Palm olein (CAS Number 93334-39-5) with standard indicators was used: melting point 22.4 °C, peroxide value 0.8 ½ O mmol/kg, anisidine value 0.3 c. u. Olein was subjected to heating at a temperature of 90 °C in order to increase oxidation indicators, after which it underwent transesterification. The difference in melting points of the initial and transesterified palm olein was used as a parameter of process efficiency.

The maximum limit values of the oxidation indicators at which the process is effective are: peroxide value 12.7 ½ O mmol/kg, anisidine value 10.4 c. u. The difference in melting points is 12.1 °C, which indicates the efficiency of the process. The qualitative indicators of the obtained transesterified fat 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.

The results of the research make it possible to use fat with increased oxidation indicators without pretreatment and predict the efficiency of transesterification depending on the fat indicators. This will increase profitability and reduce production waste

Keywords: chemical modification of fats, catalytic transesterification, potassium glyceroxide, catalyst of fat transesterification

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DEVELOPMENT OF TRANSESTERIFICATION MODEL FOR SAFE TECHNOLOGY OF CHEMICAL MODIFICATION OF OXIDIZED FATS

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1. Introduction

Transesterification is a redistribution of fatty acid residues between triacylglycerol molecules of the same type of fat or in a mixture of fats, which leads to changes in the physicochemical and structural-mechanical properties of fats [1]. Transesterification is an important tool for obtaining modified fats with properties necessary for various industries: chemical, pharmaceutical, cosmetic, food, surfactant production, etc. [2]. An important direction is the use of the transesterification reaction to obtain fatty acid esters [3]. Fatty acid esters of low molecular weight alcohols are the basis of biodiesel [4]. Transesterification makes it possible to obtain fats with specified parameters: plasticity, melting and crystallization points, hardness, etc.

A feature of fats and products based on them is their ability to oxidize. During oxidation, triacylglycerol breakdown products are accumulated in fats: free fatty acids, peroxides,

hydroperoxides, aldehydes, ketones, oxypolymers, etc. These products change the organoleptic and physicochemical properties of fat: a rancid taste appears, density increases, smoke formation temperatures decrease, etc. Oxidation products have a toxic effect on the human body if eaten. At the same time, an urgent issue is the use of waste and non-standard oils and fats as raw materials for various types of products. For example, the cost of edible vegetable oils as a raw material is (60–80) % of the total cost of biodiesel production. To overcome this problem, pretreated waste oils with increased degradation rates are used, or increased dosages of reagents are applied for processing such oils [5].

Oxidation products have a deactivating effect on common transesterification catalysts (alkoxides): sodium methoxide and ethoxide. At the same time, alkoxides are irreversibly destroyed. Fat peroxide value of 1.0 ½ O mmol/kg deactivates 0.054 kg of sodium methoxide for every 1 ton of fat. In industry, fats with a peroxide value of no more than 0.25 ½ O mmol/kg are used for transesterification. The standard value of this indicator for fats is no more than 10.0 ½ O mmol/kg. Therefore, the fat should have a peroxide value that is 40 times lower than the standard value [1].

Alkoxides are toxic, explosive, fire-hazardous compounds, harmful to the environment [6]. The use of these substances exacerbates the problem of wastewater and soil pollution [7]. The problem of fire safety and environmental pollution is important for industry and society as a whole [8]. The reduction of fire danger in premises, the study of fire hazards and the development of improved fire automation systems are urgent directions of scientific research [9]. Thus, there are problems associated with the use of transesterification catalysts that are widely used in industry.

Developments related to new catalysts for the transesterification of fats based on metal glyceroxides are promising. These catalysts show high efficiency and are safer, more stable compounds than alkoxides, and are not explosive or fire-hazardous [2]. The technology for obtaining glyceroxides is available for implementation at a fat processing enterprise and does not require non-standard equipment, unlike alkoxides [10]. For example, calcium diglyceroxide makes it possible to obtain a yield of methyl esters in the methanolysis of sunflower oil of more than 80 %, while for calcium oxide this indicator is 20 % [11]. It is known that the use of potassium glyceroxide increases the melting point of palm olein with standard values by 17.4 °C, while the effective redistribution of fatty acid residues corresponds to an increase in the melting point of palm olein by 12.0 °C [12].

Thus, the process of transesterification in the presence of alkoxides requires special precautions and a significant reduction in fat oxidation rates. Research on the transesterification of fats with standard and increased oxidation rates is important, as this will allow processing waste and non-standard fats. At the same time, catalysts based on metal glyceroxides are promising, which are safer, more resistant to external factors and impurities present in fats. In particular, the study of the transesterification of palm olein with increased peroxide and anisidine values in the presence of potassium glyceroxide is relevant.

2. Literature review and problem statement

An important issue is the processing of oils and fats of non-standard quality, including waste, by-products, substandard raw materials of the oil and fat industry, as well as used oils with increased deterioration rates.

The work [12] investigated the production of biodiesel fuel using the transesterification reaction of waste edible oil with non-standard quality indicators and methanol. The acid value of the experimental oil reached 35.4 mg KOH/g, the mass fraction of moisture was 0.136 %. The highest yield of methyl esters (97.5 %) was observed when potassium hydroxide was used. But the influence of oxidation indicators on the efficiency of methanolysis is not shown. After all, oxidation indicators lead to significant losses of the catalyst.

The authors [13] presented data on the production of methyl esters from fat waste, the acid value of which reached 7.59 mg KOH/g. The disadvantage of the work is the lack of dependence of yield and indicators of esters on peroxide and anisidine values, which also significantly affect the process of methanolysis.

In [14], the intensification of the transesterification reaction of waste oil with methyl acetate was carried out using potassium methoxide as a catalyst. Experiments were performed at different temperatures (30–50 °C), molar ratios of oil to methyl acetate (from 1:4 to 1:14), catalyst concentrations (0.5–1.5 %). However, there is no data on the effect of oil parameters on the efficiency of the process, the quality and quantity of the obtained product.

The authors [15] used spent vegetable oil for the production of biodiesel in the presence of an alkaline catalyst. The free fatty acid content of the oil before transesterification was reduced from 5 mg KOH/g to 1 mg KOH/g. The yield of biodiesel was 97.96 %. Thus, the quality of non-standard oil was increased before use, because the oil parameters affect the consumption of the catalyst, yield and quality of the final products. But it is not shown how the indicators of oxidative deterioration changed in the process of oil preparation and how they affected the quality of the product.

The work [16] investigated the transesterification of spent corn oil using sulfonated coal as a catalyst. However, there are no data on the dependence of the catalyst dosage, the quality of the reaction product on the indicators of oxidative and hydrolytic deterioration of the oil.

The authors of the paper [17] studied used oil for obtaining esters, the basis of biodiesel. Due to the high content of free fatty acids (2.43 %) in the oil, a two-stage transesterification process was used. In the first stage, a homogeneous catalyst was used to reduce the content of fatty acids, and then in the second stage, heterogeneous catalysts were used, which included MgO, K_2CO_3, to obtain biodiesel. It is shown that for an effective transesterification process, it is necessary to reduce the acid value of the oil. But the drawback of the work is the lack of data on the influence of oxidation indicators on the yield and quality of the product.

The work [18] describes the transesterification of waste vegetable oil with methanol in the presence of 1 % sodium hydroxide catalyst at different temperatures (40–65) °C. Methanol and oil were taken in a ratio of 6:1. The maximum yield of the obtained biodiesel was 90 % at 60 °C. The disadvantage of the study is the lack of data on the influence of the characteristics of oxidative and hydrolytic deterioration on the efficiency of the process.

Thus, the process of transesterification of fats with increased deterioration rates is an urgent issue, especially in order to obtain fatty acid esters as the basis of biofuel. But there is not enough data on the relationship between the values of the fat deterioration indicators, the consumption

of catalysts and other components of the reaction mixture on the efficiency of transesterification, the yield and quality of the product. In some studies, for example, in [12, 13, 15], attention is paid to the acid value of fat, but there are also no data on the effect of this indicator on the result of transesterification. At the same time, the study of the dependence of the transesterification process efficiency on the indicators of fat oxidation – peroxide and anisidine values – remains an unsolved issue.

3. The aim and objectives of the study

The aim of the study is to determine the relationship between the peroxide and anisidine values of the initial palm olein and the melting point of the olein transesterified in the presence of potassium glyceroxide. This will make it possible to effectively carry out the process of chemical transesterification for fats with increased indicators of oxidative deterioration. Potassium glyceroxide is an effective and safe transesterification catalyst that can be used in the production of modified fats for various purposes.

To achieve the aim, the following objectives were set:

– to determine the quality indicators of the experimental fat – palm olein;

– to determine the maximum limit peroxide and anisidine values of palm olein, under which the transesterification process in the presence of potassium glyceroxide is effective;

– to investigate the quality indicators of palm olein, transesterified under the conditions of the maximum limit values of the oxidation indicators.

4. Materials and methods

4. 1. The object and hypothesis of the research

The object of research is the technology of chemical transesterification of palm olein in the presence of a potassium glyceroxide catalyst. The main hypothesis of the research is the influence of indicators of oxidative deterioration of palm olein on the efficiency of the transesterification process, which is estimated by changes in the melting point of palm olein as a result of transesterification. The study suggested that the peroxide and anisidine values of palm olein affect the efficiency of the transesterification process due to the deactivation of the potassium glyceroxide catalyst. In the work, a simplification is adopted regarding the fact that other indicators of palm olein (acid value, mass fraction of moisture and volatile substances, etc.) remain unchanged and do not affect the change in the melting point of palm olein as a result of transesterification. The work uses standard research methods.

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

The following reagents and materials were used: palm olein, refined, bleached, deodorized according to DSTU 4438 (CAS Number 93334-39-5).

4. 3. Methodology for determining the quality indicators of palm olein

Melting point is determined according to ISO 6321, mass fraction of moisture and volatile substances – according to ISO 662, acid value – according to ISO 660, peroxide value – according to ISO 3960, anisidine value – according to ISO 6885.

4. 4. Methods of palm olein transesterification

A weight of palm olein was placed in a heat-resistant round-bottomed flask installed on an electric plate. The potassium glyceroxide catalyst was added in an amount of 0.45 % by weight of olein. The flask was connected to a vacuum pump. The process was carried out at the temperature of 100 °C for 1 hour under stirring conditions. The resulting mass was subjected to adsorption purification (amount of adsorbent 0.5 %, temperature 80 °C, duration 25 min) and filtered on a paper filter.

4. 5. Research planning and results processing

The research results are a set of data on the oxidation rates of palm olein and the corresponding differences in the melting points of the initial and transesterified olein. Processing of results, calculation of mathematical dependence, construction of graphical dependence were performed in the Stat Soft Statistica v6.0 package (USA) environment. The Basic Statistics module is applied to process statistical data. The significance level of the coefficients of the regression equation and the coefficient of determination of the mathematical model were calculated. Each experiment was repeated twice.

5. Results of determining the dependence of transesterification efficiency on palm olein oxidation indicators

5. 1. Determination of quality indicators of initial palm olein

Physicochemical parameters of the initial palm olein were determined. The results of the research are presented in Table 1.

Table 1

Physicochemical parameters of the initial palm olein

Indicator	Characteristic	Standard according to DSTU 4438
Melting point, °C	22.4	18–24
Mass fraction of moisture and volatile substances, %	0.02	0.1
Acid value, mg KOH/g	0.14	0.2
Peroxide value, ½ O mmol/kg	0.8	10.0
Anisidine value, c. u.	0.3	5.0

Thus, refined bleached deodorized palm olein meets the requirements of DSTU 4438 (CAS Number 93334-39-5).

5. 2. Determination of the limit maximum values of palm olein oxidation indicators for effective transesterification

An increase in the content of oxidation products in fat leads to the deactivation of the catalyst and, accordingly, to a decrease in the efficiency of the transesterification process. Therefore, it is expedient and necessary to determine the effect of the peroxide and anisidine values on changes in the melting point of palm olein as a result of transesterification. In [12], it was found that an increase in the melting point of palm olein by 12.0 °C as a result of transesterification

corresponds to a change in the triacylglycerol composition beyond the margin of error. Therefore, as a parameter of the transesterification process efficiency, it is advisable to use the difference in the melting points of the initial and transesterified palm olein.

The influence of the peroxide and anisidine values of the initial palm olein on changes in the melting point of palm olein as a result of transesterification was determined. Palm olein samples, which were kept in a drying cabinet at a temperature of 90 °C for 15 hours were used for experimental transesterification processess. Every 1 hour, a sample was taken in which the peroxide and anisidine values were determined and used for the corresponding transesterification experiment.

Processing of experimental data was performed using the Stat Soft Statistica v6.0 package (USA) using the Basic Statistics module. The dependence of the difference in the melting point of the initial and transesterified palm olein (y) on the peroxide (x_1) and anisidine (x_2) values has the following form:

$$y = 17.75 - 0.55 \cdot x_1 + 0.33 \cdot x_2 + \\ + 0.02 \cdot x_1^2 - 0.04 \cdot x_1 \cdot x_2 - 0.01 \cdot x_2^2. \quad (1)$$

Variation ranges of input variables: peroxide value (x_1): (1.3–24.6) ½ O mmol/kg, anisidine value (x_2): (1.0–6.9) c. u.

The significance level of the coefficients of the regression equation was determined (p>0.05). The adequacy of the obtained model was checked by the coefficient of determination (0.975).

According to equation (1), the estimated values of the difference in the melting points of the initial and transesterified palm olein are determined. Table 2 presents the peroxide and anisidine values for each point of the experiment, as well as experimental and calculated values of the difference in melting points.

Table 2

Peroxide and anisidine values for each point of the experiment, experimental and calculated values of the difference in melting points

Experiment No.	Values of palm olein oxidation indicators		Difference in melting points of initial and transesterified palm olein, °C	
	Peroxide value, ½ O mmol/kg	Anisidine value, c. u.	Experimental values	Calculated values
1	1.3	1.0	17.0	17.3
2	3.8	1.6	17.0	16.7
3	5.0	2.3	16.1	16.2
4	5.7	3.7	15.0	15.4
5	7.5	4.5	14.6	14.8
6	8.5	5.0	14.1	14.4
7	10.8	7.6	13.5	12.9
8	11.7	8.7	12.7	12.2
9	12.7	10.4	12.1	12.0
10	14.1	12.0	10.6	10.3
11	17.1	11.5	8.7	9.2
12	21.9	10.3	9.5	10.2
13	23.8	9.6	10.6	10.3
14	23.8	7.5	11.4	11.3
15	24.6	6.9	11.8	11.5

The dependence of the difference in the melting points of initial and transesterified palm olein on the oxidation indicators of initial palm olein is shown in Fig. 1.

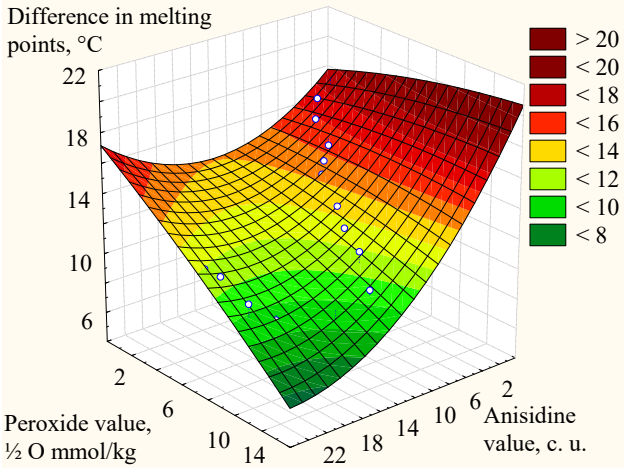

Fig. 1. Dependence of the difference in melting points of initial and transesterified palm olein on the peroxide and anisidine values of initial palm olein

By analyzing the obtained data, equation (1), Table 1 and Fig. 1, the following is found. An increase in the peroxide and anisidine values in palm olein gradually reduces the activity of the catalyst and, as a result, reduces the difference in the melting points of the original and transesterified palm olein. When samples of palm olein are kept at a temperature of 90 °C, a uniform increase in the peroxide and anisidine values is observed, accompanied by a decrease in the difference in melting points. But after 10 hours of keeping, the anisidine value begins to gradually decrease. After 11 hours of keeping against the background of a decrease in the anisidine number, a slight increase in the difference in melting points is observed. Up to 9 hours (including) of keeping olein at an elevated temperature, which corresponds to the peroxide value of 12.7 ½ O mmol/kg, the anisidine value – 10.4 c. u., a difference in melting points of more than 12.0 °C is observed. Thus, the peroxide value of 12.7 ½ O mmol/kg, the anisidine value of 10.4 c. u. are the maximum limiting values at which the transesterification process is efficient. The obtained data indicate the high efficiency of the potassium glyceroxide catalyst under the values of fat oxidation indicators above standard.

5. 3. Study of the quality indicators of palm olein, transesterified under the conditions of the limit maximum values of oxidation indicators

The physicochemical parameters of palm olein transesterified under the conditions of the limit values of oxidation indicators were determined: peroxide value 12.7 ½ O mmol/kg, anisidine value 10.4 c. u. The results of the research in comparison with the indicators for transesterified fat of the M1 brand according to DSTU 4336 are presented in Table 3.

Thus, transesterified palm olein meets the requirements of DSTU 4336 (CAS Number 97593-46-9) for transesterified fat of the M1 brand.

Table 3

Physicochemical parameters of transesterified palm olein

Indicator	Characteristic	Indicators according to DSTU 4336 for transesterified fat brand M1
Melting point, °C	34.5	27–39
Mass fraction of moisture and volatile substances, %	0.1	0.2
Acid value, mg KOH/g	0.09	0.5
Peroxide value, ½ O mmol/kg	1.2	10.0
Anisidine value, c. u.	1.0	5.0

6. Discussion of the results of studying the dependence of transesterification efficiency on the fat oxidation indicators

The technology of chemical transesterification of palm olein in the presence of potassium glyceroxide was investigated. Samples of palm olein with increased values of oxidation indicators were used for transesterification experiments. According to equation (1), Table 1 and Fig. 1, the maximum limit values of olein oxidation indicators under which transesterification is effective are determined: peroxide value 12.7 ½ O mmol/kg, anisidine value 10.4 c. u. Under these conditions, the experimental value of the difference in melting points of the initial and transesterified palm olein is 12.1 °C, which indicates the effective redistribution of fatty acid residues in the fat triacylglycerols.

There is a problem of deactivation of transesterification catalysts by oxidation products contained in fat. Therefore, fats subject to transesterification must undergo additional pre-treatment in order to reduce the peroxide value as much as possible (no more than 0.25 ½ O mmol/kg). The amount of oxidation products in the fat has a significant impact on the efficiency of transesterification.

An increase in the peroxide and anisidine values in palm olein reduces the difference in the melting points of the initial and transesterified palm olein due to the partial deactivation of the catalyst. When the olein samples were previously kept at a temperature of 90 °C, a decrease in the anisidine value was observed. This is explained by the advantage of the formation of secondary oxidation products (aldehydes, ketones, etc.), which are measured precisely by the anisidine value. At the same time, the rate of formation of primary products (peroxides, hydroperoxides), the amount of which is measured by the peroxide value, decreases. With a decrease in the anisidine value, a slight increase in the difference in melting points is observed.

The obtained scientific data on the dependence of the difference in the melting point of the initial and transesterified palm olein on oxidation indicators will allow obtaining high-quality modified fats from raw materials with increased oxidation indicators. The calculated mathematical dependence (1) and graphic representation (Fig. 1) allow predicting the efficiency of transesterification depending on the indicators of the initial raw materials.

The works [12, 13, 15] provide data on the transesterification of fats with increased indicators of hydrolytic deterioration (acid value up to 35.4 mg KOH/g), which also significantly affects the transesterification efficiency. So, scientific studies have confirmed that deterioration indicators affect the efficiency of the transesterification process due to catalyst deactivation. Fat subjected to this process requires a preliminary reduction of the corresponding indicators. In [15], it was shown that the acid value of fat must be reduced to 1.0 KOH/g for effective transesterification. But there is not enough data to consider the influence of oxidation indicators on the process of transesterification of fats.

The limitation of using the results of the work is the use of fat with stable indicators of acid value and moisture content. If these indicators exceed the values considered in this work, the dosage of the catalyst should be increased.

The disadvantage of the study is that only oxidation indicators are considered as factors affecting the transesterification process. Usually, during storage, fats are also subject to various other types of deterioration (hydrolytic, microbiological, etc.). In order to substantiate the possibility of using the potassium glyceroxide catalyst for non-standard, waste fats, these indicators should be taken into account as well.

Promising areas of work on this topic are the study of the influence of oxidation indicators of various types of fats and their mixtures on the results of transesterification. For example, it is important to select a fat recipe for specific purposes from used or non-standard fatty raw materials. This will make it possible to effectively use the potassium glyceroxide catalyst for fatty raw materials of different quality.

7. Conclusions

1. Based on the study of the quality of fatty raw materials, the indicators of palm olein (CAS Number 93334-39-5) were determined. The melting point is 22.4 °C, the peroxide value is 0.8 ½ O mmol/kg, the acid value is 0.14 mg KOH/g, the anisidine value is 0.3 c. u. Palm olein corresponds to refined deodorized bleached palm olein according to DSTU 4438.

2. As a result of experimental studies and mathematical processing of the obtained data, the maximum limit values of olein oxidation indicators were determined, under which transesterification in the presence of potassium glyceroxide is effective. The peroxide value is 12.7 ½ O mmol/kg, the anisidine value is 10.4 c. u. Under these conditions, the difference in the melting point of the initial and transesterified palm olein is 12.1 °C.

3. On the basis of studies of the quality of palm olein transesterified under the conditions of the limit maximum values of oxidation indicators, the following indicators were determined. The melting point was 34.5 °C, the peroxide value was 1.2 ½ O mmol/kg, the acid value was 0.09 mg KOH/g, the anisidine value was 1.0 c. u. Transesterified palm olein meets the requirements of DSTU 4336 (CAS Number 97593-46-9) for transesterified fat of the M1 brand.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this study, 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

Manuscript has no associated data.

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