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IMPROVEMENT OF THE TECHNOLOGY OF FATTY ACIDS OBTAINING FROM OIL AND FAT PRODUCTION WASTE

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Fatty acids are an important component in the pharmaceutical, food, chemical industries. The production of various types of products requires a certain purity and quality of fatty acids. To obtain these compounds, it is promising to use soapstocks, which are waste products of alkaline refining of oils.

The peculiarity of the work lies in determining the effect of the process parameters of soapstock decomposition with sulfuric acid on the saponification number, which is an important production characteristic of fatty acids.

The study used sunflower soapstock according to DSTU 5033 (CAS 68952-95-4) with a mass fraction of total fat of 69.5 %, fatty acids – 64.5 %. The soapstock was treated with a sulfuric acid solution at a temperature of 90 °C, the process duration was 40 min. Rational parameters of soapstock treatment were determined: concentration of sulfuric acid in the reaction mass is 80 %, concentration of an aqueous solution of sulfuric acid – 50 %. In the experiment interval, the settling duration of the reaction mass does not affect the saponification number of fatty acids. The settling time of 1 hour is effective for the isolation of fatty acids. Under these conditions, the saponification number of fatty acids was 186.4 mg KOH/g. The acids correspond to fatty acids of the first grade according to DSTU 4860 (CAS 61788-66-7): mass fraction of moisture and volatile substances – 1.2 %, mass fraction of total fat – 97.5 %, cleavage depth – 95.0 % oleic acid.

The obtained data allow rational and most efficient use of the reagent – sulfuric acid. The results of the work make it possible to reduce the duration of fatty acids obtaining from soapstocks, since the efficiency of the process with the minimum duration of mass settling has been confirmed. The improved technology of soapstock decomposition makes it possible to obtain a valuable product – high-quality fatty acids under rational conditions

Keywords: fatty acids, sunflower soapstock, sunflower oil, saponification number, alkaline neutralization

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1. Introduction

The oil and fat industry is one of the most important sources of food and technical products. Along with the problem of manufacturing environmentally friendly products, the issue of the negative environmental impact of the industry enterprises is constantly growing. More than 276 billion tons of industrial and household waste are generated annually [1]. A large number of by-products and wastewater are formed at oil and fat enterprises. Incineration, burial at special landfills and dumping of these products are dangerous for the ecological state of an area. Modern economic conditions require a more rational approach to industrial waste utilization, the development of innovative technologies and special equipment for recycling waste and by-products [2]. Production waste plays an important role as a secondary raw material resource.

The by-product of vegetable oils refining is soapstock, a product of alkaline neutralization of free fatty acids. The amount of soapstock can be up to 2 % by weight of oil [3]. The chemical diversity of soapstock composition, the presence of carboxyl, carbonyl, hydroxyl groups allow the synthesis of new types of competitive products. Modern methods of studying the crystal structure and composition of fats and fatty acids make it possible to determine the purity, nature and feasibility of using the obtained compounds in different areas [4].

Decomposition of soapstocks with sulfuric acid is the main method to obtain a valuable product – fatty acids. Thus, the improvement of this technology will allow reducing production waste, optimizing processing modes and using material resources responsibly. An important issue in fatty acids production is the rational use of reagents and materials, in particular sulfuric acid.

Thus, to optimize and reduce the waste of oil and fat production, the technology of extraction of high-quality fatty acids from soapstocks should be improved. The results of the study are important due to the need for high-quality fatty acids in the pharmaceutical, food and chemical industries.

2. Literature review and problem statement

By-products and waste of the oil and fat industry are valuable raw materials for the production of fatty acids. These compounds are used in the construction, pharmaceutical, paint, mining, oil and gas and other industries.

Soapstocks formed during vegetable oils refining consist of (30–40) % triacylglycerols, (40–50) % fatty acids sodium salts. Other substances of lipid and non-lipid nature (tocopherols, phosphatides, proteins, carbohydrates, sterols, pigments) make up (8–20) %. The amount of soapstock formed depends on the type of oil being processed and the content of free fatty acids in it [5].

The water content in soapstocks is (40–60) %. In [6], soapstock processing by concentration due to water removal was investigated. However, this technology does not involve the extraction of pure mixtures of fatty acids.

In [7], soapstock concentration with dry sodium chloride was proposed. In this way, the soapstock concentration due to water separation was (45–50) %. But the study does not provide for the production of high-purity fatty acids, gives no data on the effect of soapstock treatment parameters on fatty acids indicators.

Enzymatic hydrolysis of fats and fatty waste is promising. However, the disadvantage of this process is the high cost of enzyme preparations (up to $ 400/kg). The work [8] shows that in the process of fat hydrolysis using lipase, the acid number of fat increased from 60 to (170–180) mg KOH/g. The obtained fatty acids are used as a raw material for technical soap, salt drying oil, anti-adhesive oil, etc. [9]. The disadvantage of the study is the use of a costly enzyme, the lack of data on the effect of hydrolysis process parameters on acids quality.

Soapstocks are also useful as raw materials for other products, in particular hydrocarbons. The work [10] considers soapstock processing by cracking and decarboxylation of fatty acid molecules at (440–460) °C. Liquid hydrocarbons suitable for use as motor fuel are obtained. There are no data on the production of fatty acids, which are valuable raw materials for many products and can also be used in fuel production.

The technology of fatty acids production from soapstock by soap decomposition under the action of carbon dioxide has been developed [11]. The disadvantage of this technology is that the process is carried out under high pressure.

In [12], the process of soapstock decomposition with sulfuric acid to isolate fatty acids was studied. The yield of the concentrate was 50 %. The disadvantage of this technology is the lack of data on the quality of fatty acids.

The work [13] shows a method for isolating fatty acids from soapstock by hydrolysis to produce biofuels. The disadvantage of the study is the lack of data on the quality of fatty acids, in particular, neutralization number, saponification number, mass fraction of moisture and so on.

The work [14] proposes a technology of soapstock processing, which includes saponification with sodium hydroxide solution. Then treatment with sulfuric acid to isolate fatty acids is carried out. The disadvantage of the study is the lack of data on the effect of the concentration and amount of sulfuric acid on the efficiency of fatty acid extraction.

In [15], the process of soapstock decomposition with sulfuric acid to obtain fatty acids was studied. Rational process parameters have been determined: temperature (90–95) °C, duration 40 min. Under these conditions, the fatty acids yield is 79.0 %, neutralization number – 180.0 mg KOH/g. However, the work does not show the effect of the amount and concentration of sulfuric acid on the efficiency of fatty acid extraction. This is an important issue because sulfuric acid is a hazardous reagent, and special conditions are required to use concentrated acid solutions. Rational conditions should be determined to use this component. In addition, the effect of the process conditions on the saponification number of fatty acids is not shown. Saponification number is an important parameter necessary for technological calculations, determining the ratio of reagents, analyzing the molecular weight of fatty acids, predicting the product yield, etc.

Thus, there are little data on the effect of sulfuric acid treatment conditions on the saponification number and other quality indicators of fatty acids. There are no data on the effect of reagent characteristics (in particular, sulfuric acid concentration) on the quality of fatty acids. So, it is relevant to improve the technology of soapstock processing to obtain fatty acids as a marketable product. It is important to determine rational values of the concentration of sulfuric acid in the reaction mass and sulfuric acid solution. This will reduce the environmental burden and make rational use of reagents.

3. The aim and objectives of the study

The aim of the study is to improve the technology of soapstock decomposition with sulfuric acid to obtain fatty acids with a high saponification number. This will allow rational and efficient use of sulfuric acid during the process and using rational duration of reaction mass settling.

To achieve the aim, the following objectives were set:

– to investigate the quality indicators of the sunflower soapstock sample;

– to determine the dependence of the saponification number of fatty acids on the concentration of sulfuric acid in the reaction mass, concentration of an aqueous solution of sulfuric acid and duration of reaction mass settling;

– to determine the organoleptic, physicochemical parameters and average molecular weight of fatty acids extracted.

4. Materials and methods of research

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

The following reagents and materials were used:

– soapstock obtained after alkaline neutralization of sunflower oil, according to DSTU 5033 (CAS 68952-95-4);

– p.a.-grade sulfuric acid, according to GOST 4204 (CAS 7664-93-9).

4. 2. Method for determining sunflower soapstock indicators

The organoleptic indicators of sunflower soapstock were determined by standard method according to DSTU 5033:2008 (Method for determining color, consistency and odor). International normative documents for determining organoleptic parameters: color – ISO 15305, consistency – AOCS Method Cc 16-60, odor – AOCS Cg 2-83. The mass fraction of moisture, mass fraction of total fat and fatty acids were determined by standard methods according to DSTU 4603:2006 (ISO 662) and DSTU 5033:2008 (ISO 17189, IDF 194), respectively.

4. 3. Method of soapstock decomposition with sulfuric acid

Treatment of soapstock with sulfuric acid was performed according to [15]. The treatment temperature was 90 °C, process duration – 40 minutes.

4. 4. Method for determining the organoleptic, physicochemical indicators and average molecular weight of fatty acids

The organoleptic indicators, mass fraction of total fat, cleavage depth, presence of mineral acids were determined by standard methods according to DSTU 4860:2007, mass fraction of moisture – according to DSTU 4603:2006, saponification number – according to DSTU ISO 3657:2004 (ISO 3657:2002, IDT).

The average molecular weight of fatty acids (Ma) was determined by the formula:

$$Ma = \frac{56{,}110}{SN} - 12.67, \quad (1)$$

where SN is the saponification number of fatty acids, mg KOH/g.

4. 5. Research planning and results processing

A second-order full factorial experiment with three factors was used in the work. Each factor has three levels of variation. Processing of the obtained data and construction of graphic dependences were performed using the Stat Soft Statistica v6.0 package (USA). Two repetitions were performed in each experiment.

5. Results of studies of the dependence of the saponification number of fatty acids on the process parameters of soapstock decomposition

5. 1. Determination of quality indicators of the sunflower soapstock sample

The organoleptic and physicochemical indicators of the sunflower soapstock sample were determined. The results of the study are presented in Table 1.

Table 1

Organoleptic and physicochemical indicators of the soapstock sample

Indicator	Characteristic
Organoleptic indicators	
Color	Brown
Consistency at 20 °C	Oily
Odor	Inherent in sunflower oil soapstock
Physicochemical indicators	
Mass fraction of moisture and volatile substances, %	12.1
Mass fraction of total fat, %	69.5
Mass fraction of fatty acids, %	64.5
Mass fraction of neutral fat, %	5.0

Thus, the experimental sample of sunflower soapstock meets the standards set out in DSTU 5033 (CAS 68952-95-4).

5. 2. Determination of the dependence of the saponification number of fatty acids on the process parameters of soapstock decomposition

Saponification number is an important parameter, as it determines the actual amount of the lipid part in the product (fats and fatty acids in the free and bound state). Saponification number characterizes the average molecular weight of fatty acids in the sample. These data are important for many areas that use fatty acids. For example, the production of soap and detergents, fatty acid esters, production of high-purity fatty acids for pharmaceutical purposes, chemical synthesis of various compounds, etc.

Therefore, to improve the technology of soapstock decomposition with sulfuric acid, the dependence of fatty acids saponification number (y) on the following factors was studied:

– concentration of sulfuric acid in the reaction mass (x_1): from 20 to 100 %;

– concentration of an aqueous solution of sulfuric acid (x_2): from 20 to 80 %;

– duration of reaction mass settling (x_3): from 1 to 7 h.

The response function is the saponification number of fatty acids. The results of the study were processed in the Stat Soft Statistica v6.0 package (USA). Fig. 1 shows the Pareto chart constructed to test the significance of the regression equation coefficients. The Pareto chart shows standardized coefficients sorted by absolute values. The most influential effects and their interactions were selected by the t-criterion. The columns are arranged from top to bottom in descending order of the significance of the effects and their interactions. The coefficients related to the settling duration of the reaction mass (x_3) were found to be insignificant. All other effects evaluation columns cross the vertical line representing a 95 % confidence probability.

The regression dependence of the saponification number of fatty acids (y) on the concentration of sulfuric acid in the reaction mass (x_1) and the concentration of an aqueous solution of sulfuric acid (x_2) in real variables is as follows:

$$y = 98.014 - 0.963 \cdot x_1 + \\ +1.788 \cdot x_2 + 0.013x_1^2 - 0.016x_2^2. \qquad (2)$$

The adequacy of the obtained model was tested by the absence of consistency loss (significance level p is less than 0.05 and is $7.237 \cdot 10^{-12}$) and the determination coefficient (close to one and equal to 0.974).

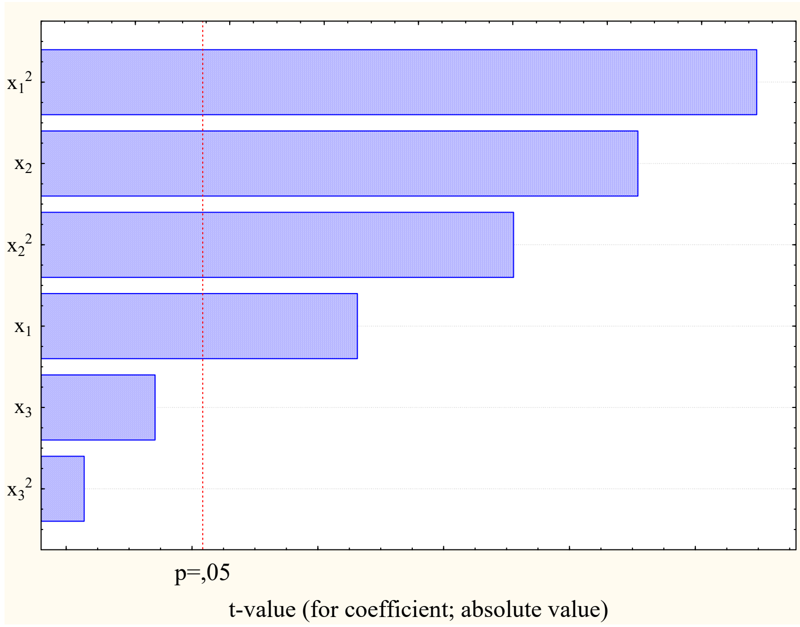

Fig. 1. Pareto chart

Table 2 shows the experiment planning matrix, experimental and calculated values of the response function (saponification numbers of fatty acids). The calculated values of the response function were obtained by regression equation (2).

The response surface, which is the dependence of saponification number on the concentration of sulfuric acid in the reaction mass and the concentration of sulfuric acid solution, is presented in Fig. 2.

Table 2

Experiment planning matrix and response function values

Experiment number	Factors of variation			Response function	
	Concentration of sulfuric acid in the reaction mass x_1, %	Concentration of sulfuric acid solution x_2, %	Duration of mass settling x_3, h	Saponification number y, mg KOH/g (experimental values)	Saponification number y, mg KOH/g (calculated values)
1	20	20	1	111.6	104.4
2	20	80	7	134.8	137.2
3	20	20	7	117.9	114.6
4	20	80	1	131.5	127.0
5	20	80	4	134.2	135.7
6	20	20	4	114.5	113.1
7	20	50	7	132.5	140.3
8	20	50	1	127.1	130.1
9	20	50	4	137.0	138.8
10	60	20	1	117.5	113.4
11	60	80	7	139.7	146.2
12	60	20	7	129.1	123.6
13	60	80	1	132.3	136.0
14	60	80	4	137.8	144.7
15	60	20	4	119.0	122.1
16	60	50	7	139.6	149.3
17	60	50	1	147.9	139.1
18	60	50	4	159.5	147.9
19	100	20	1	143.8	163.6
20	100	80	7	207.9	196.4
21	100	20	7	177.1	173.8
22	100	80	1	190.2	186.2
23	100	80	4	195.8	194.9
24	100	20	4	170.2	172.3
25	100	50	7	202.2	199.5
26	100	50	1	187.1	189.3
27	100	50	4	199.5	198.0

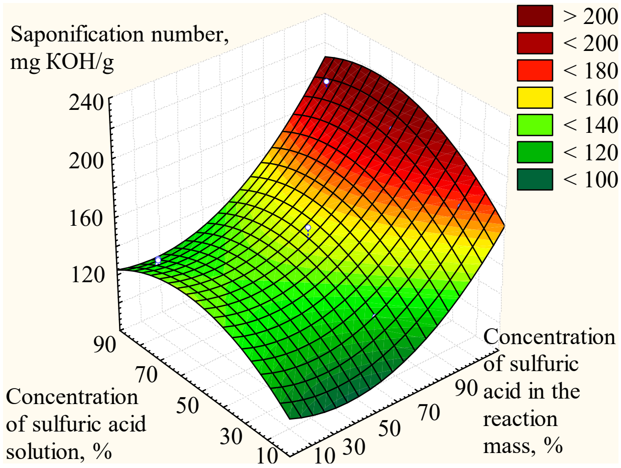

Fig. 2. Dependence of the saponification number of fatty acids on the concentration of sulfuric acid in the reaction mass and the concentration of sulfuric acid solution

Therefore, with increasing concentration of sulfuric acid solution, there is an increase and then a decrease in the values of the response function. The maximum values of the saponification number correspond to the concentration of sulfuric acid solution of 50 %. As the concentration of sulfuric acid in the reaction mass increases, the response function has minimal values at a concentration of (30–50) %. After that, the values of the response function increase. Analyzing the data in Table 2, equation (2) and Fig. 2, it is revealed that an increase in the concentration of sulfuric acid in the reaction mass of more than 80 % does not lead to a significant increase in the saponification number. Thus, the rational conditions for soapstock decomposition with sulfuric acid to obtain the maximum saponification number are as follows. The concentration of sulfuric acid in the reaction mass is 80 %, the concentration of an aqueous solution of sulfuric acid – 50 %. The settling duration in the experiment does not affect the saponification number of fatty acids. Therefore, in this case, the minimum value of settling duration of 1 hour is rational. Under these conditions, the saponification number of fatty acids was 186.4 mg KOH/g.

5. 3. Determination of the organoleptic, physicochemical indicators and average molecular weight of fatty acids

The quality indicators of fatty acids extracted under rational conditions were determined. The results of the study are shown in Table 3.

Table 3

Organoleptic and physicochemical indicators of extracted fatty acids

Indicator	Characteristic
Color at 20 °C	Brown
Odor	Specific for fatty acids
Mass fraction of moisture and volatile substances, %	1.4
Mass fraction of total fat, %	98.1
Cleavage depth, % oleic acid	66.7
Presence of mineral acids	None

Thus, under the determined rational conditions, high-quality fatty acids were obtained corresponding to the fatty acids of light oils and modified fats obtained without saponification, first grade, according to DSTU 4860 (CAS 61788-66-7).

The average molecular weight of fatty acids calculated by formula (1) was 288.3 g/mol, which correlates with the reference value for fatty acids of sunflower oil – 283 g/mol [10].

The use of rational values of sulfuric acid concentration in the reaction mass and concentration of sulfuric acid solution allowed obtaining fatty acids with improved parameters. Thus, the mass fraction of moisture and volatile substances decreased, the mass fraction of total fat and cleavage depth increased compared to fatty acids obtained without taking into account the effects of sulfuric acid. The following rational parameters of soapstock treatment with sulfuric acid were determined in [15]: temperature (90–95) °C, duration 40 min. Under these conditions, the mass fraction of moisture and volatile substances in fatty acids was 1.8 %, mass fraction of total fat – 97.0 %, cleavage depth – 64.5 %. However, the concentrations of sulfuric acid in the solution and in the reaction mass were not taken into account. Therefore, the application of the determined rational conditions can improve the product quality.

6. Discussion of the results of studying the dependence of the saponification number of fatty acids on the process conditions of soapstock decomposition

The improved technology of soapstock decomposition with sulfuric acid, which involves the use of rational values of sulfuric acid concentration in the reaction mass and concentration of sulfuric acid solution, has been studied. This allows you to predict the value of the saponification number of fatty acids and obtain fatty acids with a high value of this indicator. The influence of the process parameters of soapstock treatment on the saponification number of fatty acids was determined. According to Table 2, Fig. 2 and equation (2), the rational conditions for soapstock decomposition were determined: concentration of sulfuric acid in the reaction mass is 80 %, concentration of an aqueous solution of sulfuric acid – 50 %. In the conditions of the experiment, the settling duration of the reaction mass has no effect. Therefore, the minimum duration of 1 hour is rational in this case. Under these conditions, the saponification number of fatty acids was 186.4 mg KOH/g.

The work differs from the existing scientific research [7–15] by determining the rational values of sulfuric acid concentration in the reaction mass and sulfuric acid solution concentration in order to improve the quality of fatty acids. The developed advanced technology can improve the quality of fatty acids. The mass fraction of moisture and volatile substances decreases (by 22 % relative to the technology without taking into account sulfuric acid concentration). The mass fraction of total fat and cleavage depth increase (by 1.1 % and 3.4 %) [15]. The use of soapstock decomposition technology is more rational given the concentration of sulfuric acid solution and the amount of acid in the mass. This technology allows an efficient and rational use of the reagent (sulfuric acid). In addition, the process duration can be significantly reduced, as it was experimentally confirmed that the settling duration (from 1 to 7 hours) does not affect the saponification number.

With increasing concentration of sulfuric acid solution, there is an increase and then a decrease in the response function values. The maximum values of the saponification number correspond to the concentration of sulfuric acid solution of 50 %. As the concentration of sulfuric acid in the reaction mass increases, the response function increases after the minimum values at a concentration of (30–50) %. As shown in Table 2, equation (2) and Fig. 2, an increase in the concentration of sulfuric acid in the reaction mass of more than 80 % does not lead to a significant increase in the saponification number. Therefore, the rational conditions for soapstock decomposition are as follows: concentration of sulfuric acid in the reaction mass is 80 %, concentration of an aqueous solution of sulfuric acid – 50 %. The minimum value of the settling duration of 1 hour is rational.

Increasing the concentration of sulfuric acid in the mass has a more significant effect on the increase in the saponification number than the concentration of sulfuric acid solution. Thus, at a concentration of sulfuric acid in the mass of 100 % when changing the acid solution concentration from 20 to 80 %, the saponification number increases 1.17 times. At a concentration of sulfuric acid solution of 80 %, when changing the acid concentration in the mass from 20 to 100 %, the saponification number increases 1.54 times.

In general, increasing the acid concentration in the mass contributes to a faster and more efficient mass transfer, a more complete response of the components and, consequently, an increase in the saponification number. Increasing the concentration of the acid solution also increases the amount of acid per unit mass of soapstock, while reducing the amount of water in the mass.

The results of the work allow a more rational, effective and fast process of soapstock decomposition with sulfuric acid. The determined dependence of the saponification number of acids on the process conditions allows assessing the quality and feasibility of using acids for different purposes. These data are necessary for calculating the amount of reagents and auxiliary components in production processes where the raw material is fatty acids (various types of soaps, biodiesel, pharmaceutical compounds, etc.). The obtained data are a scientific achievement that will contribute to the rational use of multicomponent and environmentally hazardous waste of oil and fat production.

The limitation of using the research results is that the work used the initial soapstock with certain indicators. If soapstock with higher moisture values is to be used, these data must be taken into account when administering an aqueous solution of sulfuric acid in order to maintain rational acid concentrations.

The disadvantage of the study is the lack of data on the effect of sulfuric acid concentrations on the fatty acids yield. After all, in addition to quality indicators, the amount of the obtained product is essential.

Promising areas of research on soapstock treatment to isolate fatty acids is determining a wider range of fatty acid indicators at the points of the experiment (i.e., composition, mass fraction of total fat, etc.). This will allow a comprehensive analysis of the possibility of using the acids obtained in this way in various spheres.

7. Conclusions

1. The quality indicators of the sunflower soapstock sample were studied: mass fraction of moisture and volatile substances – 12.1 %, total fat – 69.5 %, fatty acids – 64.5 %, neutral fat – 5.0 %. Sunflower soapstock meets the requirements of DSTU 5033 (CAS 68952-95-4).

2. The dependence of the saponification number of fatty acids on the parameters of soapstock treatment was determined. Rational values of the parameters: concentration of sulfuric acid in the reaction mass – 80 %, concentration of an aqueous solution of sulfuric acid – 50 %. In the experimental interval, the settling duration of the reaction mass does not affect the saponification number of fatty acids. Settling duration of 1 hour is effective for the isolation of fatty acids. Under these conditions, the saponification number of fatty acids was 186.4 mg KOH/g.

3. In terms of quality, the fatty acids correspond to those of light oils and modified fats obtained without saponification, first grade, according to DSTU 4860 (CAS 61788-66-7). Characteristics of the acids: mass fraction of moisture and volatile substances – 1.2 %, mass fraction of total fat – 97.5 %, cleavage depth – 95.0 % oleic acid. The average molecular weight of fatty acids was 288.3 g/mol.

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