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The object of the study is the process of treatment of sunflower phosphatide concentrate using sodium chloride solution.

Hydration is a stage of oil refining. The waste of the process is a phosphatide

concentrate, the disposal of which is dangerous to the environment. The concentrate contains valuable components – oil

and phosphatides. An important task is to separate these components for effective

phosphatide concentrate by hydration in

the presence of sodium chloride solution

was investigated. The influence of the con-

centrate treatment conditions on the oil

SOU 15.4-37-212:2004 (CAS 3436-44-0)

was used: mass fraction of moisture and

volatile substances -2.8 %, mass fraction of phosphatides -41.5 %. The concentrate

was treated with sodium chloride solution

with a concentration of (5-20) %. The

hydration time was 25 min., the temperature was 45 $^{\circ}$ C, and the mass ratio of the

sodium chloride solution to the concen-

ment were determined: the concentration

of sodium chloride solution was 15 %, set-

tling time was 5 hours. At the same time,

were determined: acid value 2.8 mg

KOH/g, peroxide value 3.2 ½ 0 mmol/kg,

mass fraction of moisture and volatile sub-

stances 0.12 %. According to these indica-

tors, the extracted oil corresponds to first-

grade unrefined unfrozen sunflower oil

according to DSTU 4492. The mass frac-

tion of phosphorus-containing substances

in terms of stearooleolecithin was 1.7 %,

which slightly exceeds the standard value.

to process hydration waste and obtain oil,

which is a raw material for the products of

many industries. This will help solve the

problem of disposal of environmentally hazardous waste and improve the state of

industry, phosphatide concentrate, sodi-

um chloride, rational hydration conditions

Keywords: waste of the oil and fat

The research results make it possible

the yield of oil was 86.9 %.

Conditions for the concentrate treat-

The parameters of the extracted oil

A sample of concentrate according to

The process of extracting oil from

use in various industries.

yield was determined.

trate was 1:1.

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DEVELOPMENT OF CONDITIONS FOR OBTAINING OIL FROM SUNFLOWER OIL HYDRATION WASTE

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1. Introduction

Hydration is a refining stage in which phosphatides, phosphatidoproteins and other hydrophilic impurities are removed from oil. This stage is necessary because during the storage of oils, phosphatides fall out in the form of sediment, which complicates a number of technological operations for oil processing. Vegetable oils contain a significant amount of phosphatides: sunflower -(0.2-1.4) %, cottonseed -(0.5-2.5) %, soybean - up to 3.5 %. The amount of phosphatides obtained can reach about 1 t per 100 t of unrefined sunflower oil produced [1].

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Phosphatides are organic substances that, by their chemical structure, are complex esters of glycerol and fatty acids, including phosphoric acid and nitrogen-containing substances. Vegetable oils contain: phosphatidylcholine, phosphatidylethanolamine, phosphatidyliositol, phosphatidylserine, phosphatidic acids.

During hydration, oils are treated with water or solutions of salts, acids, alkalis, tannin and some other substances. Water, interacting with hydrophilic groups of phosphatides, causes mass separation into a layer of acylglycerols and a layer of hydrated phosphatide molecules (hydration sediment). Thus, a phosphatide emulsion is obtained. The hydration sediment contains: phosphatides (8–2) %, oil – up to 20 %, protein substances – (50–60) %, moisture – about 14 %. A phosphatide concentrate is obtained from the emulsion – a product of dehydration of the emulsion at a temperature of (85–90) °C and a residual pressure of 15.0 kPa [2].

Hydration sediment does not allow for long-term storage and is usually disposed of. A significant amount of oil and fat production waste exacerbates the problem of environmental pollution [3]. When the waste comes into contact with oxygen, intense oxidation and heat release occur, which can cause spontaneous combustion and the formation of toxic substances [4].

The components of hydration waste are valuable industrial raw materials. Phosphatides and oils are used in the chemical, food, and paint industries, as well as in the production of alternative biofuels [2]. Phosphatides are a promising component of therapeutic and dietary products [5].

So, an urgent task is to develop and improve the technology for processing hydration waste in order to increase the concentration of phosphatides and extract oil. In particular, it is necessary to develop rational conditions for obtaining proper quality oil, which is a raw material for various industrial purposes.

2. Literature review and problem statement

There are various ways of separating phosphatides from oil in order to separately obtain concentrated phosphatides and oil.

Enzyme preparations are used to separate oil and phosphatides. For example, in [6], phospholipase treatment of sediment obtained after hydration of vegetable oils with water was considered. The use of an enzyme preparation made it possible to increase oil yield by converting phospholipids into diglycerides and reduce oil retention in the sediment. However, the economic feasibility of using the preparation (the cost of phospholipase reaches \$70/kg) and the physicochemical parameters of the obtained oil have not been shown.

The authors [7] investigated the treatment of soy lecitin by hydrolysis using phospholipase A1 in order to obtain L- α -glycerylphosphorylcholine (the yield was 95.8%). The technology for isolating individual phosphatides is important, but the effect of this method on the quality of the bulk of phosphatides and extracted oil has not been shown.

In [8], magnetically immobilized phospholipase A2 (PLA2) for isolating phosphatides from soybean oil was studied. The process was carried out for 5.0 hours with an enzyme dosage of 0.24 g/kg. The disadvantage of the work is the lack of data on the effect of this method on the quality of oil separated from phosphatides.

The authors [9] compared the effect of acid and enzymatic hydration on the preservation of bioactive components in hydrated oil. Phospholipase A1 in combination with phospholipase C provided better retention of bioactive components. But the effect of these methods on the efficiency of separating phosphatides and oil, as well as the quality indicators of oil after hydration, is not shown.

In order to intensify the separation of oil and phosphatides, chemical reagents and solvents are used. In [10], simultaneous hydration and neutralization of oils using the tetrabutylphosphonium phosphate IL reagent were studied. Optimal treatment conditions: soybean oil (3 % IL, $60 \degree$ C and 20 min), canola oil (2 % IL, $60 \degree$ C and 20 min), sunflower oil (1.5 % IL, $50 \degree$ C, $15 \min$). The content of free fatty acids and phosphorus in the oil reached 0.18 % and 3.5 mg/kg; 0.12 % and 3.8 mg/kg; and 0.17 % and 3.5 mg/kg, respectively. But the effect of this treatment method on other physicochemical indicators of oils is not shown.

The authors [11] investigated the acetone extraction of phosphatides from by-products of water hydration of rice bran oil. The yield was 4.77 % dry phosphatide. The disadvantage of the work is the use of an explosive and fire-hazardous solvent, the quality indicators of the oil separated from phosphatides are not shown.

The authors [12] studied the fractionation of phosphatide sediment obtained after acid hydration of rapeseed oil using ethanol. The ethanol-soluble fraction was rich in phosphatidylcholine (57 %), the insoluble fraction was rich in phosphatidylinositol and phosphatidic acid (37.4 %). But the effect of this method on the quality indicators of the oil formed after the extraction of phosphatides from the sediment is not shown.

The paper [13] considered the separation of phosphatides from unrefined soybean oil using cavitation reactors. The degree of hydration was 93.65 %. But the influence of process parameters on the quality indicators of the obtained oil is not shown.

The work [14] studied the hydration of rapeseed oil with water and an electrolyte. Compared to water hydration (86.3%), sodium chloride hydration demonstrated a higher completeness of phospholipid removal (90.7%). Therefore, the use of sodium chloride during hydration increases the efficiency of separating oil from phosphatides. However, the effect of sodium chloride concentration on the yield and quality indicators of the obtained oil is not shown.

The prospects of using sodium chloride in the extraction of oil and waxes from spent perlite are shown in [15]. The use of a 7.5 % sodium chloride solution intensifies the separation of individual layers of wax and oil. But the application of this method in separating oil from phosphatide concentrate is not shown.

In [16], the treatment of sunflower phosphatide concentrate with citric acid was studied. Rational conditions for concentrate treatment have been determined: duration (25 min.) and citric acid concentration relative to the mass of phosphatide concentrate (25 %). The oil yield was 76.1 %, the mass fraction of moisture in the oil was 18.6 %. But the use of citric acid complicates the production of food concentrate, requires additional processing of extracted oil and phosphatides.

Thus, existing studies [6–16] present data on various methods of separating oils from phosphatides. But there is not enough data on the impact of the considered methods and parameters of oil and hydration sediment treatment on the yield and quality indicators of extracted oils. Therefore, an urgent task is to develop rational conditions for obtaining oil from hydration waste using a safe and effective reagent. Such a promising reagent for the effective production of high-quality oil from hydration sediment is sodium chloride.

3. The aim and objectives of the study

The aim of the study is to develop conditions for obtaining oil from the waste of sunflower oil hydration – sunflower phosphatide concentrate. This will make it possible to effectively use oil hydration waste, reduce the negative impact of industry on the environment and obtain a valuable product – oil that has a wide range of uses.

To achieve the aim, the following objectives were accomplished:

– to determine the dependence of oil yield on the concentration of sodium chloride solution and mass settling time and determine the conditions of phosphatide concentrate treatment under which the oil yield is maximum;

 to determine the physicochemical indicators of the oil extracted under rational conditions.

4. Materials and methods

4. 1. The object and hypothesis of the study

The object of the study is the process of extracting oil from the waste of sunflower oil hydration – phosphatide concentrate. The main hypothesis of the study is that the concentration of sodium chloride solution and settling time of the obtained mass after treatment affect the yield of the extracted oil. The study suggested that increasing the concentration of sodium chloride solution would increase the efficiency of oil extraction from the phosphatide concentrate and reduce the settling time. A simplification is adopted that the amount of sodium chloride solution does not affect the efficiency of oil extraction (in the study, the mass ratio of sodium chloride solution to phosphatide concentrate is 1:1), but only the concentration is affected.

4.2. Examined materials and equipment used in the experiment

The following reagents and materials were used (CAS Number is a unique numerical identifier assigned to chemicals for a consistent method of identification by the Chemical Reference Service):

- sunflower phosphatide concentrate according to SOU 15.4-37-212:2004 (CAS 3436-44-0);

- sodium chloride according to DSTU 3583 (CAS Number 7647-14-5).

4.3. Method of determining the quality indicators of phosphatide concentrate

The quality indicators of phosphatide concentrate are determined by standard methods according to SOU 15.4-37-212:2004.

4.4. Method of phosphatide concentrate treatment with sodium chloride solution

A portion of phosphatide concentrate is placed in a heat-resistant beaker mounted on an electric stove. A stirrer and a thermometer are placed in a beaker. Sodium chloride solution of a given concentration (according to the experimental plan) is added. The hydration time is 25 minutes, the temperature is 45 °C, the mass ratio of sodium chloride solution to the concentrate is 1:1. After hydration, the mass is settled for the time set according to the experimental plan at a temperature of 45 °C. After that, the upper layer (oil), middle layer (phosphatides) and lower layer (aqueous compo-

nent – sodium chloride solution) are separated. The scheme of mass stratification is shown in Fig. 1.

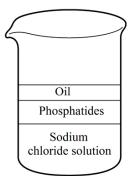


Fig. 1. Scheme of the mass stratification in a glass after settling

4. 5. Method of studying the quality indicators of extracted oil

The physicochemical indicators of the oil are determined by the following standard methods of analysis of oils and fats. The acid value, peroxide value, mass fraction of phosphorus-containing substances, mass fraction of moisture and volatile substances are determined by standard methods according to ISO 660, ISO 3960, ISO 10540-3, ISO 662, respectively.

4. 6. Planning of experimental studies and processing of data

A second-order full factorial experiment was used. Data processing and construction of graphical dependency were performed in the StatSoftStatistica v6.0 package environment (USA). Each experiment was repeated twice. Calculations were made using the "General Regression Models" module. Tabs used: Parameter Estimates (calculation of equation coefficients, standard error, 95 % confidence interval); "Observed, Predicted, and Residual Values" (determination of calculated values of the response function); "ANOVA" (analysis of variance).

5. Results of determining conditions for obtaining oil from phosphatide concentrate

5. 1. Determination of the dependence of the oil yield on the concentration of sodium chloride solution and mass settling time

Quality indicators of the test sample of sunflower phosphatide concentrate in comparison with standard indicators according to SOU 15.4-37-212:2004 are given in Table 1.

Table 1

Quality indicators of the phosphatide concentrate sample

	Characteristic	
Indicator	Test sample	Value according to SOU 15.4-37-212:2004 (SnK mark)
Mass fraction of moisture and volatile substances, %	2.8	3.0
Mass fraction of phosphatides, %	41.5	40.0
Mass fraction of oil, %	42.5	60.0
Mass fraction of substances insoluble in ethyl ether, %	3.8	5.0

So, the test sample corresponds to the phosphatide feed concentrate (SnK mark) according to SOU 15.4-37-212:2004.

The influence of the conditions of the phosphatide concentrate treatment with sodium chloride solution (hydration) on the oil yield was determined. Variation factors and intervals: $-x_1$ – concentration of sodium chloride solution: from 5 to 20 %;

 $-x_2$ – settling time: from 1 to 7 hours.

The response function y is the oil yield (as a percentage of the available oil content in the phosphatide concentrate). The results were processed using the StatSoftStatistica v6.0 (USA) package. The calculated regression dependence of oil yield on the treatment conditions of phosphatide concentrate in real variables is as follows:

$$y = -15.84 + 10.69 \cdot x_1 + 3.33 \cdot x_2 - -0.30 \cdot x_1^2 - 0.01 \cdot x_1 \cdot x_2 - 0.20 \cdot x_2^2.$$
(1)

The significance level of the regression equation coefficients (p>0.05) and the coefficient of determination (0.999) were determined. Table 2 shows the planning matrix, experimental and calculated values of the response function.

Graphically, the dependence of oil yield on hydration conditions is presented in Fig. 2.

Factors of variation		Oil yield, %	Oil viold %		
Experiment number	Concentration of sodi- um chloride solution, %	Settling time, hours	(experimental value)	Oil yield, % (calculated value)	
1	5	1	33.2	33.3	
2	5	3	37.1	38.1	
3	5	5	41.5	41.4	
4	5	7	42.6	42.9	
5	10	1	64.8	63.8	
6	10	3	68.9	68.7	
7	10	5	73.5	71.9	
8	10	7	74.5	73.5	
9	15	1	78.5	79.1	
10	15	3	82.3	84.0	
11	15	5	86.9	87.2	
12	15	7	87.4	88.8	
13	20	1	79.7	79.2	
14	20	3	84.1	84.1	
15	20	5	88.2	87.3	
16	20	7	88.7	88.9	

Planning matrix and response function values

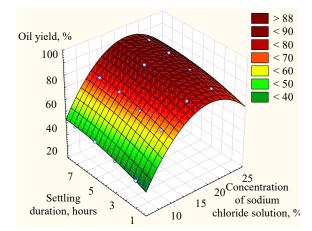


Fig. 2. Dependence of the oil yield on the concentration of sodium chloride solution and mass settling time

5.2. Determination of physicochemical indicators of oil extracted under rational conditions

The physicochemical indicators of the oil extracted from the phosphatide concentrate under rational conditions were determined. Before analysis, the oil was washed with water

Table 2

at a temperature of 45 °C to remove residual sodium chloride and dried in a vacuum at (85–90) °C. The results of the study are given in Table 3.

Table 3

Physicochemical indicators of oil extracted from phosphatide concentrate

Indicator	Characteristic	
Acid value, mg KOH/g	2.8	
Peroxide value, ½ O mmol/kg	3.2	
Mass fraction of phosphorus-containing substances, in terms of stearooleolecithin, %	1.7	
Mass fraction of moisture and volatile substances, %	0.12	

The obtained oil corresponds to first-grade unrefined unfrozen sunflower oil according to DSTU 4492. The mass fraction of phosphorus-containing substances was 1.7 %, which slightly exceeds the standard value. The oil can be used as a raw material

An increase in the concentration of sodium chloride solution has a greater effect on the oil yield than the settling time of the mass after hydration. But after increasing the concentration by more than 15 %, the increase in oil yield slows down and does not increase significantly. Therefore, it is effective to use the concentration of sodium chloride solution of 15 %. With a settling time of 3 hours, a significant increase in oil yield is observed, but only after 5 hours of settling, the oil yield practically does not increase. Therefore, it is advisable to use the mass settling time of 5 hours. in various industries, and after additional purification – for food purposes.

6. Discussion of the results of developing conditions for obtaining oil from phosphatide sunflower concentrate

The technology of processing waste from the hydration of sunflower oil (phosphatide concentrate) in order to extract a valuable product – oil was studied. Treatment of the concentrate involves hydration using a sodium chloride solution. According to equation (1), Table 2 and Fig. 2, conditions for phosphatide concentrate treatment (hydration) are determined: the concentration of sodium chloride solution is 15 %, settling time is 5 hours. At the same time, the oil yield was 86.9 %. The increase in the oil yield with an increase in the concentration of sodium chloride solution and mass settling time is due to an increase in the density of the lower aqueous phase, a greater intensity of separation of layers in the mass after hydration. Sodium chloride is an electrolyte that prevents emulsification in the mass, which intensifies the separation of phosphatides.

An increase in the concentration of sodium chloride solution has a greater effect on the oil yield than the settling time of the mass after hydration. For the settling time of 1 hour, with an increase in the concentration of sodium chloride solution from 5 to 10 %, the oil yield increases by 31.6 %. At the concentration of sodium chloride solution of 5 % when changing from the settling time of 1 to 3 hours the yield increases by only 3.9 %. According to equation (1), Table 2 and Fig. 2, it is effective to use the concentration of sodium chloride solution of 15 % and settling time of 5 hours.

The research results make it possible to rationally use the waste of the oil and fat industry to obtain a high-quality product – oil. This will reduce environmental pollution and increase the profitability of oil and fat enterprises due to the production of a valuable product.

The works [6–16] provide data on various methods of separating oil and phosphatides. For this purpose, enzyme preparations [6-9], special reagents and solvents [10-13] are used. However, enzyme preparations are costly, organic solvents are dangerous substances. An unsolved problem is the use of hazardous substances and the lack of correlation between the amount of reagents and process conditions for oil yield and quality. The method of extracting oil from the phosphatide concentrate studied in this work correlates with the method discussed in [14], where the sodium chloride solution was used for the hydration of rapeseed oil. Phosphatides isolated using sodium chloride have a higher emulsifying capacity. This indicates that the presence of an electrolyte intensifies the process of separating oil from phosphatides. However, there are no data on the effect of this method on the yield and quality indicators of oil.

The advantages of this study in comparison with similar known ones [6–16] are the use of a safe and affordable reagent (sodium chloride) in order to extract high-quality oil. The spent sodium chloride solution formed in the lower part of the mass can be used to extract sodium chloride, which can be reused. For this purpose, cleaning of the solution from mechanical impurities, evaporation, crystallization, separation of sodium chloride crystals (for example, by centrifugation) and drying are used.

The limitation of using the results of the work is the developed rational hydration conditions: the concentration of sodium chloride solution is 15 %, settling time is 5 hours. A decrease in the concentration of sodium chloride and mass settling time causes a decrease in oil yield and can affect the oil quality.

The disadvantage of the work is the study of the effect of hydration parameters only on oil yield. After all, phosphatides, from which oil has been extracted, are also a valuable product. It is appropriate to study the influence of hydration parameters on the yield and quality of phosphatides.

A promising area of work is to study the influence of hydration parameters on other indicators of the extracted oil, as well as on the parameters of the obtained phosphatides.

7. Conclusions

1. The dependence of the oil yield on the concentration of sodium chloride solution and the settling time of the mass after hydration of the phosphatide concentrate was determined. An increase in the concentration of sodium chloride solution has a greater effect on the oil yield than mass settling time. Thus, for the settling time of 1 hour, with an increase in the concentration of sodium chloride solution from 5 to 10 %, the oil yield increases by 31.6 %. At the concentration of sodium chloride solution of 5 % when changing from the settling time of 1 to 3 hours, the yield increases by only 3.9 %. Conditions for concentrate treatment were developed: the concentration of sodium chloride solution was 15 %, settling time was 5 hours. Under these conditions, the oil yield was 86.9 %.

2. The physicochemical indicators of the oil extracted under rational conditions were determined: acid value 2.8 mg KOH/g, peroxide value 3.2 O mmol/kg, mass fraction of moisture and volatile substances 0.12 %. According to these parameters, the extracted oil corresponds to first-grade unrefined unfrozen sunflower oil according to DSTU 4492. The mass fraction of phosphorus-containing substances in terms of stearooleolecithin was 1.7 %, which slightly exceeds the standard value. The obtained oil can be used as a raw material in productions of many industries, and after additional purification – directly for food purposes.

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.

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