

IMPROVEMENT OF THE HYDRATION TECHNOLOGY OF WALNUT OIL WITH NON-STANDARD QUALITY INDICATORS

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The object of the study is the hydration process of walnut oil with non-standard quality indicators.

Walnut oil is used in the food, cosmetic industry, and for technical purposes. Phosphorus-containing substances worsen the consumer and technological properties of oils, complicate the purification processes and use in chemical reactions. Hydration allows to remove phosphorus-containing substances by treating with water, acids, or enzyme preparations.

The hydration of walnut oil with an increased mass fraction of phosphorus-containing substances, as well as moisture and volatile substances was studied. The qualitative indicators of the initial oil: mass fraction of phosphorus-containing substances (in terms of stearooleolecthin) 0.56%, mass fraction of moisture and volatile substances 0.4%, peroxide value 3.2 $\frac{1}{2}$ O mmol/kg, acid value 2.5 mg KOH/g.

The oil was previously subjected to water hydration under the following conditions: temperature 55°C, added water concentration 1.0%, settling time 2 h. The mass fraction of phosphorus-containing substances decreased to 0.49%.

Further reduction of the mass fraction of phosphorus-containing substances was carried out using citric acid (50% solution). Rational conditions for acid hydration were established: temperature 60°C, citric acid concentration to the mass of oil 0.3%. After that, the mass fraction of phosphorus-containing substances in the hydrated oil was 0.07%.

The hydrated oil was subjected to drying under vacuum conditions (95°C, 1 h). The mass fraction of moisture and volatile substances in the dried hydrated oil was 0.1%.

Effective extraction of phosphorus-containing substances from the oil was achieved by gradually conducting water and acid hydration and establishing rational conditions for acid hydration.

The results of the work make it possible to effectively carry out hydration of walnut oil and other oils with an increased content of phosphorus-containing substances

Keywords: walnut oil, water hydration, acid hydration, content of phosphorus-containing substances

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1. Introduction

Walnut (*Juglans regia*) is oil crop that is characterized by high yield – 122 kg per tree. The plant is characterized

by a significant kernel content – (53–47.1)%, fruit weight – (17.6–10.8) g, ease of separation of the kernel from the shell, and resistance of fruits and leaves to disease [1]. The kernels contain up to 75% fatty oil, up to 6000 mg% amino acids,

vitamins E and C, β -carotene, salts K, Ca, Mg, S, P. The fatty acid composition of walnut oil includes: saturated fatty acids – 12.0%; monounsaturated – 24.0%; polyunsaturated – 64.0% [2]. Nut kernels contain iron – (5.1 ± 1.3) mg/100 g, zinc – (3.2 ± 0.9) mg/100 g, copper – (1.0 ± 0.26) mg/100 g, manganese – (3.9 ± 1.1) mg/100 g, cobalt – (7.5 ± 2.5) μ g/100 g, chromium – (7.0 ± 0.88) μ g/100 g [3, 4].

In many industries, there is a need for refined oil from which impurities have been removed. Oil refining includes hydration, neutralization, bleaching and deodorization. Hydration (with the addition of water and/or citric, phosphoric acid) is the first stage of refining, during which phospholipids, proteins and mucous substances are removed from the oil. These impurities complicate the further transportation, storage and use of the oil. Thus, during chemical reactions, such impurities reduce the efficiency of processes and worsen the quality of the product. For example, in the production of biodiesel fuel, esters of low molecular weight alcohols are obtained from oils when heated. And such impurities contribute to the formation of undesirable products, worsen the color, composition of the product, and reduce the shelf life. The ingress of the product with phosphatides into engines causes increased carbon deposits, clogging of injectors and filters [5].

During hydration, the mixture of oil and water is treated very carefully to prevent the formation of an emulsion. A certain amount of non-hydrated phospholipids (calcium and magnesium salts of phosphatidic acids and phosphatidylethanolamine) remains in the hydrated oil, since only hydrated phospholipids are removed during hydration. Non-hydrated phospholipids are converted to the hydrated form using acid.

Walnut oil is used in cosmetic products (creams, lotions, hair care products). In the food industry, refined walnut oil is used as an ingredient in dressings, sauces and cooking oils due to its neutral taste and high smoking point [6]. The oil is used for technical and industrial purposes (e.g., in lubricants, for pharmaceutical purposes, etc.) [7].

Thus, obtaining oil from walnuts is promising. The oil has a valuable fatty acid composition and is used in various industries. An important stage of oil processing is the first stage of refining – hydration, which allows obtaining the product for further use in various industries. The problem of oil purification during hydration is the presence of non-hydrated phospholipids. Therefore, research on the effective hydration of walnut oil with the production of high-quality hydrated oil with a minimum content of phosphorus-containing substances is relevant.

2. Literature review and problem statement

Existing study is aimed at developing and improving hydration methods, in particular, for processing crude oils with a high content of phosphorus-containing substances. A separate issue is increasing the efficiency of hydration of walnut oil. Thus, in the work [8], hydration of crude wild walnut oil was carried out at different levels of concentrations of phosphoric acid, alkali and bleaching earth, and temperature. In the experiment, the phosphorus content was controlled as a response function. The phosphorus content in the oil ranged from 2.7 mg/kg to 14.18 mg/kg during the experiments. The maximum phosphorus content was observed at low doses of phosphoric acid (1.0%), bleaching earth (1.0%) and temperature of 70°C. The minimum was observed at the dosage of

phosphoric acid of 1.25%, bleaching earth of 1.5% and temperature of 85°C. However, the use of phosphoric acid makes the measurement results less accurate due to the additional phosphorus content in the oil. The reason for this is, first of all, the comparison of the influence of different conditions of oil hydration on phosphorus content, without considering the rational management of the process and the maximum removal of phosphorus-containing substances in general.

Changes in physicochemical properties (acid value, peroxide value), the content of tocopherols, polyphenols and phytosterols in cold-pressed walnut oil as a result of refining were studied [1]. Hydration with water was the most suitable for preserving bioactive compounds. For example, the content of tocopherols was 259.40 mg/kg, polyphenols – 44.54 mg/kg, and phytosterols – 987.32 mg/kg. However, the oxidation stability of the obtained oil (3.09 h) was lower than that of molecularly distilled oil (4.18 h). The initial physicochemical properties, especially the peroxide value, had a significant impact on the oxidation stability. It is necessary to find a compromise between the preservation of nutrients and an increase in the shelf life, which indicates the need to develop appropriate refining methods. The disadvantage of the work is the lack of data on the influence of technological parameters of hydration on the content of phosphorus-containing substances in the oil. The objective reason for this is the long process of determining the content of such substances and the need to conduct a series of studies for this indicator, and the presented study is general for different oil indicators.

Technological parameters of the walnut oil refining process were investigated [9]. Rational hydration parameters were established: mixing speed 60 rpm, added water concentration 3%, temperature 60°C. For alkaline neutralization: mixing speed 50 rpm, alkali concentration 10%, temperature 55°C. For adsorption purification: adsorbent concentration 1%, mixing speed 55 rpm, temperature 65°C. When conducting processes under these conditions, the oil yield was 85.1%. The qualitative indicators of refined oil were determined: the concentration of unsaturated fatty acids was 92.7%, vitamin E was 28.4 mg/100 g. However, the influence of hydration parameters on the content of phosphorus-containing substances in the oil was not shown. This is due to the fact that the priority indicator was the oil yield, and the determination of the content of phosphorus-containing substances requires a separate series of studies of the hydration process itself and is quite lengthy.

In the work [10], the process of refining walnut oil was investigated. The neutralization rate was 46.75% for adsorption deoxidation (AD), 94.06% for chemical deoxidation (CD) and 66.05% for molecular distillation deoxidation (MDD). Both CD and MDD reduced the content of α -linolenic acid compared to AD. All methods affected the total content of phenols, tocopherols and phytosterols; in particular, CD and MDD reduced the total phenol content by 24.38% and 54.13%, while AD retained it up to 95.79%. Regarding the retention of tocopherols: the MDD method showed the lowest result – 57.75%, AD – the highest – 74.62%. Therefore, the adsorption deoxidation (AD) process is considered the most acceptable method of deoxidation of walnut oil due to its advantages in preserving nutritional components and antioxidant capacity of the oil. The disadvantage of the work is the lack of data on the influence of phosphorus-containing substances in the oil on the neutralization efficiency. It is not shown which preliminary hydration treatment is effective for the further implementation of the considered refining

methods. The reason for this is the fact that it is difficult to vary several parameters related to different stages of refining simultaneously.

Oil refining leads to the decrease in the content of vitamins and natural antioxidants in oils. The effect of hydration (compared to other stages of refining) on the physicochemical properties, content of trace elements and oxidative stability of vegetable oils was studied [11]. The level of losses of total phytosterols in oil during neutralization (9.23–7.3%) and deodorization (9.97–8.27%) was higher than during hydration (3.01–0.87%) and bleaching (2.75–1.18%). Deodorization had the greatest impact on the total content of tocopherols, hydration had the least. The authors noted that the obtained data can be used to find optimal conditions to meet the basic requirements for oil and minimize the loss of trace elements. However, the effect of hydration parameters on the content of phosphorus-containing substances, as well as other quality indicators of oil, was not shown. Such data would be useful for optimizing the hydration process of oils with a high content of phosphorus-containing substances. One of the objective reasons may be the duration and complexity of analyzing all oil samples in the experimental plan for this indicator.

An important direction is the development of new hydration methods for the most complete removal of phosphorus-containing substances, in particular, during the processing of oils of substandard quality.

In the work [12], data are provided on the development of a highly efficient hybrid hydration process for vegetable oils, which combines chelation with citric acid with subsequent flocculation with sodium hydroxide (NaOH). The optimal conditions were determined: 339 mg/kg citric acid, 167 mg/kg NaOH, 1.76% water at 70°C, which reduced the residual phosphorus content to 0.22 mg/kg. However, there is no data on the influence of hydration parameters on the content of phosphorus-containing substances in the experimental oils. The reason is to consider the simultaneous effect of citric acid and alkali on the quality of the obtained hydrated oils and the complexity of studying the effect of citric acid alone.

A new process for hydration of oils based on polymer inclusion membranes (PIM) made with polyvinyl alcohol (PVA) as a carrier and tributyl phosphate (TBP) as a plasticizer was investigated [13]. A number of selective extraction agents were also used: cholic acid (CA), resorcinol (RES), β -cyclodextrin (β -CD), dibenzo-18-crown-6 ether (CRW), gluconic acid (GA), and EDTA. These membranes provide facilitated transport of transition metal ions (Ca^{2+} , Mg^{2+} , Fe^{3+}), while mediating controlled water penetration into the oil phase. This promotes hydration of phospholipids and the formation of liposomes for removal by centrifugation or decantation. The disadvantage is the use of a large number of reagents. The influence of hydration parameters on the content of phosphorus-containing substances in the studied oils was also not shown. The reason is that the developed membranes were tested without taking into account the changes in the hydration process parameters.

Simultaneous hydration and neutralization of edible oils using tetrabutylphosphonium phosphate IL [14] were studied. Soybean oil (3 wt.% IL, 60°C and 20 min), rapeseed oil (2 wt.% IL, 60°C and 20 min) and sunflower oil (1.5 wt.% IL, 50°C, 15 min) were treated. The content of free fatty acids (FFA) and phosphorus reached 0.18% and 3.5 mg/kg; 0.12% and 3.8 mg/kg; and 0.17% and 3.5 mg/kg,

respectively. The water washing stage was excluded, and no wastewater was generated. However, the disadvantage is the lack of data on the influence of processing conditions on the content of phosphorus-containing substances in oils. The reason is the simultaneous hydration and neutralization of oils without taking into account the efficiency of hydration separately.

In the work [15], a strategy for enzymatic degumming for various vegetable oils is described by combining phospholipase A1 with monoacylglycerol lipase. The combination of the two enzymes significantly reduced the phosphorus content, which indicates better hydrolysis of phospholipids. Enzyme preparations have a higher cost (up to 2 thousand USD/kg) than conventional reagents for hydration (for example, the cost of citric acid is 2 USD/kg). However, enzyme preparations were used in the work to demonstrate the advantages of this hydration method, its speed and efficiency, without taking into account the economic component.

The effect of different hydration processes on the physicochemical properties of crude sunflower oil was investigated: phosphorus content, acid value, peroxide value, phospholipids, iron, moisture, unsaponifiable matter, viscosity, density and color [16]. Three different hydration processes were evaluated: with phospholipase A1, with phospholipase A2 and with water. Enzymatic hydration tests were performed at 50°C, pH 5 and an enzyme dose of 200 units/kg oil for 180 minutes with both enzymes. Water treatment was performed with a water/oil ratio of 3% and a temperature of 65°C. The phosphorus content decreased from 544.51 to 3.02 mg/kg and 5.81 mg/kg with phospholipase A1 and A2, respectively. Enzymatically hydrated oils had better physicochemical parameters than untreated and water-hydrated oils. The disadvantage is the high cost of enzyme preparations. However, the paper highlights the existing advantages of this method of oil hydration, without taking into account the cost of such a preparation per unit of oil mass. In order to more completely remove phosphorus-containing substances from oils, a polypropylene-based membrane was developed [17]. Scanning electron microscopy, Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy, and atomic force microscopy were used to characterize the developed membrane. The optimized membrane demonstrated a permeability of pure n-hexane of 35 l/m² h·bar, a tensile strength of 112 MPa, an oil wetting angle of 30° and a water wetting angle of 106°. The process of hydration of oils using a membrane demonstrated 36% oil rejection, 42% free fatty acid rejection and 97% phospholipid rejection. Thus, the membrane proved to be effective for oil hydration. The disadvantage is the use of several reagents, the need for preliminary preparation of the membrane. Also, the influence of oil processing parameters on the content of phosphorus-containing substances has not been shown. The reason for this may be the instability of the membrane properties and the need for a separate long-term series of studies.

Thus, in existing works [1, 8–10], the hydration processes of walnut oil have been investigated. However, there is no data on a clear relationship between hydration conditions and the content of phosphorus-containing substances in the oil. To intensify the process of hydration of oils, a number of innovative methods have been developed that involve the use of certain, in particular, multicomponent, reagents, as well as preliminary preparation of these substances [11–17]. At the same time, there is not enough data on the use of classical hydration agents (water, citric, phosphoric acid) for process-

ing walnut oil, including those with high initial levels of phosphorus-containing substances. The influence of walnut oil hydration parameters on the content of phosphorus-containing substances remains an unresolved issue. All this allows to argue that it is advisable to conduct a study devoted to identifying the relationship between the hydration parameters of walnut oil and the content of phosphorus-containing substances. It is also advisable to determine the rational conditions for hydration of oil with a high initial level of this indicator.

3. The aim and objectives of the study

The aim of the study is to improve the technology of hydration of walnut oil with non-standard quality indicators. This will allow for efficient hydration of walnut oil and other oils with high initial levels of phosphorus. It will also allow for the prediction of the effect of hydration conditions (citric acid concentration and temperature) on the mass fraction of this indicator in the hydrated oil.

To achieve the aim, the following objectives were solved:

- to investigate the effect of preliminary aqueous hydration of walnut oil on the mass fraction of phosphorus-containing substances;
- to determine the effect of citric acid concentration and hydration temperature of walnut oil on the mass fraction of phosphorus-containing substances and to establish the values of these parameters corresponding to the maximum removal of phosphorus-containing substances;
- to determine the quality indicators of walnut oil hydrated under established rational conditions.

4. Materials and methods

4.1. The object and hypothesis of the study

The object of the study is the hydration process of walnut oil with non-standard quality indicators.

The main hypothesis of the study is that the intensity of the extraction of phosphorus-containing substances from walnut oil depends on the concentration of citric acid and the hydration temperature.

The work assumes that the residual mass fraction of phosphorus-containing substances in hydrated walnut oil decreases with increasing citric acid concentration and hydration temperature.

The adopted simplification is that other hydration parameters that may affect the result (duration, concentration of the added citric acid solution, etc.) are not considered. The study used standard methods for studying the quality indicators of oils and fats.

4.2. Examined materials and equipment used in the experiment

During the experimental studies, the following materials and equipment were used:

- walnut oil (DSTU 6047, CAS Number 8024-09-7);
- citric acid monohydrate food grade (CAS Number 5949-29-1);
- softened drinking water (DSTU 7525, CAS Number 7732-18-5).

Walnut oil with increased mass fraction of phosphorus-containing substances (0.56% in terms of stearooleoleci-

thin), as well as moisture and volatile substances (0.4%) was used. The standard limit values of these indicators according to DSTU 6047 are 0.4% and 0.2%, respectively. This oil was chosen for the study, since there is a problem of the most complete extraction of phosphorus-containing substances from oils, in particular, with high values of the content of these substances. It is also necessary to take into account the increased moisture content in the starting raw materials.

4.3. Methods for researching the quality indicators of initial and hydrated walnut oil

The parameters of walnut oil were tested using standard methods generally accepted for oils and fats. The mass fraction of moisture and volatile substances was tested according to ISO 662:2016; acid value – ISO 660:2020; peroxide value – ISO 3960:2017; mass fraction of phosphorus-containing substances – DSTU 7082:2009 (weight method).

4.4. Methods of experimental walnut oil hydration processes

Preliminary aqueous hydration is carried out as follows. A portion of walnut oil is placed in a heat-resistant beaker placed on an electric hotplate. A stirrer and a thermometer are placed in the beaker. Water is added to the portion of oil in the amount of 1.0%. Hydration is carried out at the temperature of 55°C. The duration of hydration is 25 min. After the end of the process, the mass is settled for 2 h. After that, the upper layer (hydrated oil), the middle layer (mainly phosphatides) and the lower aqueous layer are separated.

Hydration using citric acid is carried out similarly. The 50% solution of citric acid is added to the portion of oil. The amount of solution is calculated according to the required amount of citric acid in each experiment of the experiment. The hydration temperature is maintained for each experiment according to the experimental plan. The duration of hydration is 25 min. After the end of the process, the mass is settled for 2 h. After that, the top layer (hydrated oil), middle layer (mainly phosphatides), and bottom layer (aqueous layer – citric acid solution) are separated.

4.5. Research planning and processing of results

The full factorial experiment of the second order was used. The processing of the results of the study was performed in the StatSoftStatistica v6.0 (USA) environment – the “General Regression Models” module. The coefficients of the regression equation, the standard error, the estimated values of the response function were calculated, and the analysis of variance was performed. Two repetitions were performed in each experiment.

5. Results of establishing the rational concentration of citric acid and the oil hydration temperature

5.1. Study of the effect of preliminary aqueous hydration of walnut oil on the mass fraction of phosphorus-containing substances

The study used walnut oil (raw, obtained by walnuts pressing) with the physicochemical indicators given in the Table 1.

Thus, in the studied oil sample, the mass fraction of phosphorus-containing substances, mass fraction of moisture and volatile substances exceed the standards according to DSTU 6047. The oil corresponds to the first grade in terms of acid value.

Table 1

Physicochemical indicators of the original walnut oil

Name of indicator	Characteristics	Norm according to DSTU 6047	
		Higher grade	First grade
Moisture and volatile matter mass fraction, %	0.4	0.2	
Acid value, mg KOH/g	2.5	1.5	4.0
Peroxide value, 1/2 O mmol/kg	3.2	10.0	
Mass fraction of phosphorus-containing substances (calculated as steorooleocithin), %	0.56	0.4	

Preliminary water hydration of the oil was carried out (according to point 4.4) and its effect on the mass fraction of phosphorus-containing substances was established. Water hydration was carried out under the following conditions: temperature 55°C, concentration of added water 1.0%, settling time 2 hours. As a result of hydration, the mass fraction of phosphorus-containing substances decreased from 0.56 to 0.49%.

During the interaction of oil with water, small flakes of hydration sediment were formed, which are difficult to settle and remove from the oil. This fact indicates that the oil contains mainly phospholipids that do not hydrate.

Considering the mass fraction of phosphorus-containing substances in the oil after hydration, the mass fraction of phosphorus-containing substances that are not hydrated is up to 87.5% of the total content of phospholipids. Therefore, it is advisable to carry out acid hydration.

5.2. Determination of the influence of walnut oil hydration conditions on the mass fraction of phosphorus-containing substances

The influence of the conditions of acid hydration of the walnut oil on the mass fraction of phosphorus-containing substances (response function) was established. The following factors and variation intervals were investigated:

- x_1 – acid hydration temperature: from 40 to 60°C;
- x_2 – citric acid concentration (to the weight of oil): from 0.1 to 0.5%.

Citric acid was added to the oil in the form of a 50% solution. Acid hydration was carried out according to the point 4.4. The results were processed using the Stat Soft Statistica v6.0 package (USA).

Table 2 presents the experimental research planning matrix, experimental (y_e) and calculated (y_c) values of the mass fraction of phosphorus-containing substances in the hydrated oil.

The dependence of the mass fraction of phosphorus-containing substances in hydrated oil (Y , %) on the hydration temperature (x_1 , °C) and the concentration of citric acid (x_2 , %), in natural variables, has the form

$$Y = 1.187 - 0.017 \cdot x_1 - 0.596 \cdot x_2 + 0.006 \cdot x_1 \cdot x_2. \quad (1)$$

The results of the analysis of variance, which confirmed the adequacy of the calculated mathematical model and the significance of the coefficients of the regression equation, are shown in the Table 3.

By analyzing equation (1) in the environment of the Stat Soft Statistica v6.0 package (USA), the conditions of acid hydration of oil were determined, which correspond to the maximum removal of phosphorus-containing substances: temperature 60°C, citric acid concentration 0.3%.

Table 2

Experimental research planning matrix, experimental (y_e) and calculated (y_c) values of the mass fraction of phosphorus-containing substances in hydrated oil

Experiment No.	Variation factors				Mass fraction of phosphorus-containing substances y_e , % (experimental value)	Mass fraction of phosphorus-containing substances y_c , % (calculated value)
	Temperature, x_1		Citric acid concentration, x_2			
	Coded level	°C	Coded level	%		
1	-1	40	-1	0.1	0.45	0.46
2	-1	40	0	0.3	0.39	0.39
3	-1	40	+1	0.5	0.33	0.32
4	0	50	-1	0.1	0.3	0.29
5	0	50	0	0.3	0.24	0.23
6	0	50	+1	0.5	0.15	0.17
7	+1	60	-1	0.1	0.12	0.12
8	+1	60	0	0.3	0.07	0.08
9	+1	60	+1	0.5	0.05	0.03

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	0.0753	1	0.0753	277.1595	0.000014
Temperature, °C	0.0414	1	0.0414	152.4005	0.000062
Citric acid concentration (to oil weight), %	0.0022	1	0.0022	8.1464	0.035649
Temperature, °C· Citric acid concentration, %	0.0006	1	0.0006	2.3006	0.048976
Error	0.0014	5	0.0003	-	-

Coefficient of determination $R^2 = 0.996$. Adjusted coefficient of determination $R_{adj}^2 = 0.992$

As a result of oil treatment under these conditions, the mass fraction of phosphorus-containing substances in the hydrated oil was 0.07%. Further increase in the citric acid concentration does not lead to a significant decrease in the value of the response function.

The graphical dependence of the mass fraction of phosphorus-containing substances in hydrated walnut oil on the hydration temperature and citric acid concentration is shown in the Fig. 1.

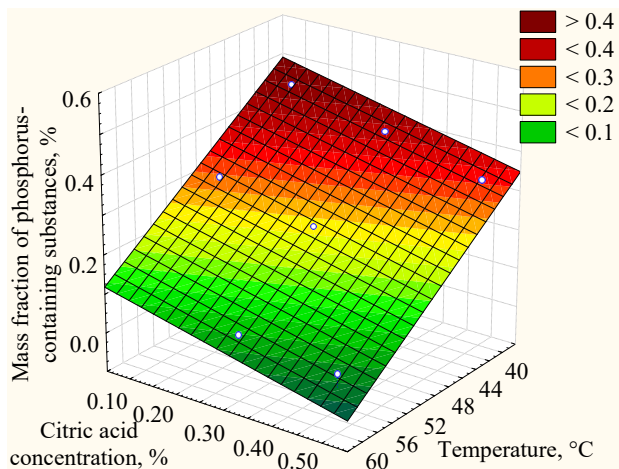


Fig. 1. Dependence of the mass fraction of phosphorus-containing substances in hydrated walnut oil on the hydration temperature and citric acid concentration

Therefore, with increasing citric acid concentration and process temperature, the mass fraction of phosphorus-containing substances in hydrated walnut oil decreases (Fig. 1). The maximum decrease in the mass fraction of phosphorus-containing substances in hydrated oil (without further significant changes in this indicator) occurs under conditions of citric acid concentration of 0.3% and temperature of 60°C. The mass fraction of phosphorus-containing substances in hydrated oil was 0.07%.

5. 3. Determination of quality indicators of walnut oil hydrated under established rational conditions

Walnut oil, hydrated under established rational conditions, was subjected to drying under vacuum conditions (temperature 95°C, duration 1 h). The qualitative indicators of the obtained hydrated dried oil were determined. The obtained data are given in the Table 4.

Table 4

Qualitative indicators of walnut oil (hydrated under established rational conditions and dried)

Name of indicator	Characteristics
Moisture and volatile matter mass fraction, %	0.1
Acid value, mg KOH/g	1.5
Peroxide value, 1/2 O mmol/kg	2.7
Mass fraction of phosphorus-containing substances (calculated as stearooleocithin), %	0.07

As shown in the Table 4, the drying also reduced the acid and peroxide values. The obtained hydrated dried walnut oil meets the requirements of DSTU 6047 for higher grade

oil (Table 1). Thus, the determined rational conditions for hydration of walnut oil allowed obtaining high-quality hydrated oil for various industrial needs.

6. Discussion of the results of establishing rational hydration conditions for walnut oil of substandard quality

The technology of hydration of walnut oil with an increased mass fraction of phosphorus-containing substances, as well as moisture and volatile substances, was investigated. The oil was previously subjected to water hydration under the following conditions: temperature 55°C, concentration of added water 1.0%, settling time 2 h. As a result of this process, the mass fraction of phosphorus-containing substances in the oil was 0.49%. The presence of small flakes of hydration sediment, which are difficult to settle and remove, as well as the estimated amount of phospholipids that are not hydrated (up to 87.5%), determined the need for further acid hydration (Table 1).

Based on equation (1), Table 2 and Fig. 1, rational conditions for acid hydration were established: temperature 60°C, concentration of citric acid to the mass of oil 0.3%. As a result of hydration, the mass fraction of phosphorus-containing substances in the hydrated oil was 0.07%.

The effectiveness of acid hydration is explained as follows. As a result of the process, non-hydrated phospholipids, consisting mainly of calcium and magnesium salts of phosphatidic acid and phosphatidylethanolamine, can be converted into a hydrated form. The acid also ensures the removal of phosphatidic acids and phosphatidylethanolamine in the free state by binding divalent ions of calcium and magnesium metals into a complex that can be removed with the aqueous phase.

The oil, hydrated under certain rational conditions, was subjected to drying under vacuum conditions (temperature 95°C, duration 1 h). The qualitative characteristics of the obtained hydrated dried oil (Table 4): mass fraction of moisture and volatile substances 0.1%, acid number 1.5 mg KOH/g, peroxide number 2.7 1/2 O mmol/kg. Due to the removal of volatile acids and oxidation products during vacuum drying, there was a decrease in the acid and peroxide values. Therefore, the obtained hydrated dried walnut oil meets the requirements of DSTU 6047 for higher grade oil (Table 1).

The obtained data will allow to increase the efficiency of hydration of walnut oil and other vegetable oils with increased initial values of the mass fraction of phosphorus-containing substances. After all, an important task of oil and fat enterprises is the processing of oils and fats with quality indicators that exceed standard values. In addition, the obtained mathematical dependence (equation (1), Table 2 and Fig. 1) allows to predict the influence of the applied hydration conditions on the final mass fraction of phosphorus-containing substances in the oil.

The studies [1, 8–10] provide data on the refining, in particular, hydration, of walnut oil. The effect of phosphoric acid on the phosphorus content in hydrated oil was studied. The minimum phosphorus content was observed under the conditions of phosphoric acid dosage of 1.25%, bleaching earth of 1.5% and temperature of 85°C [8]. The effect of hydration on the content of natural impurities in oil was established [1]. Aqueous hydration was described as a refining stage before alkaline neutralization [9]. In order to intensify the hydra-

tion processes of vegetable oils, new and improved methods using reagents of various nature were studied [11–17]. Existing studies have confirmed that acid hydration (which is used in this work) is more effective than aqueous. However, there is insufficient data on the use of classical hydration agents (water, citric, phosphoric acid) for the processing of walnut oil, including those with a high initial content of phosphorus-containing substances. The influence of the hydration conditions of walnut oil on the content of phosphorus-containing substances remains an unresolved issue. A distinctive feature of this work is the study of the effective extraction of phosphorus-containing substances from walnut oil by gradually conducting aqueous and acid hydration and establishing rational conditions for acid hydration.

The limitation of the application of the obtained data is the established rational conditions for acid hydration of walnut: temperature 60°C, concentration of citric acid to the mass of oil 0.3%. Carrying out hydration under conditions outside these parameters may affect the quality of the obtained hydrated oil, and also requires additional research.

The disadvantage of the work is the lack of research on the phosphatides obtained as a result of hydration. After all, phosphatides are a valuable product. But phosphatides obtained by acid hydration are not used as food.

A promising direction of research is to study the influence of other hydration parameters (duration, concentration of citric acid solution, number of revolutions of the mixer, etc.) on the efficiency of the process and the quality of the resulting hydrated oil.

7. Conclusions

1. The effect of preliminary aqueous hydration of walnut oil on the mass fraction of phosphorus-containing substances was studied. An oil sample was studied, for which the mass fraction of phosphorus-containing substances, mass fraction of moisture and volatile substances exceed the standards according to DSTU 6047 (0.56% and 0.4%, respectively). As a result of aqueous hydration of the oil (temperature 55°C, concentration of added water 1.0%, settling time 2 h), the mass fraction of phosphorus-containing substances decreased to 0.49%. In this case, small flakes of hydration sediment were formed, which are difficult to settle and remove, which indicates the predominant presence of non-hydrated phospholipids in the oil.

2. The effect of citric acid concentration and hydration temperature of walnut oil on the mass fraction of phosphorus-containing substances was determined. With increasing citric acid concentration and process temperature, the mass fraction of phosphorus-containing substances in hydrated walnut oil decreases. The maximum decrease in the mass fraction of phosphorus-containing substances in hydrated oil (without further significant changes in this indicator) occurs under conditions of citric acid concentration of 0.3% and

temperature of 60°C. The mass fraction of phosphorus-containing substances in hydrated oil was 0.07%.

3. The qualitative indicators of walnut oil hydrated under established rational conditions were determined. Walnut oil hydrated under rational conditions was subjected to drying under vacuum conditions (temperature 95°C, duration 1 h). The indicators of the dried hydrated oil are as follows. Mass fraction of moisture and volatile substances is 0.1%, acid value is 1.5 mg KOH/g, peroxide value is 2.7 ½ O mmol/kg, the mass fraction of phosphorus-containing substances (in terms of stearooleolecithin) is 0.07%. As a result of drying, the values of acid and peroxide values also decreased. The obtained hydrated dried walnut oil meets the requirements of DSTU 6047 for high-quality oil. Thus, the determined rational conditions for hydration of walnut oil allowed obtaining high-quality hydrated oil for various industrial needs.

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

Gabriella Birta: Resources, Conceptualization, Data Curation, Project administration. **Vladyslav Knysz:** Methodology, Data Curation, Validation. **Anatolii Shostia:** Resources, Investigation, Supervision. **Svitlana Usenko:** Conceptualization, Investigation, Writing – original draft. **Liubov Morozova:** Formal analysis, Writing – review & editing. **Yurii Burgu:** Writing – review & editing, Validation. **Nataliia Ofilenko:** Methodology, Formal analysis. **Yevhen Semenko:** Writing – original draft, Visualization. **Ivan Kostenko:** Visualization, Software. **Roman Mylostyvyi:** Software, Formal analysis.

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