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IMPROVING THE TECHNOLOGY OF WAXES OBTAINING FROM OIL AND FAT WASTE

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The object of the research is the extraction of waxes from the waste of the oil and fat industry – spent perlite.

As a result of oil winterization, a significant amount of spent filter powders is formed, the disposal of which poses a danger to the environment. At the same time, winterization waste contains a significant amount of oil and wax, which are important components of many types of products. Oil and wax extraction involves the use of volatile, fire-hazardous solvents. An urgent task is to develop new safe and effective technologies for obtaining oils and waxes from winterization waste.

The technology of extracting waxes from spent perlite, obtained as a result of winterization of sunflower oil, which involves treatment of perlite with sodium chloride solution, was investigated.

Perlite was used according to SOU 15.4-37-210:2004 (CAS Number 93763-70-3) with indicators: mass fraction of fat – 20.1 %, peroxide value – 19.5 ½ O mmol/kg, acid value – 3.5 mg KOH/g. The temperature of mass settling after treatment with sodium chloride is 20 °C, the duration – 10 hours.

The following rational conditions for perlite processing using sodium chloride were determined: the concentration of dry sodium chloride (by weight of perlite) – 35.0 %, the duration of mass boiling – 30 minutes. Under these conditions, the experimental value of the saponification value of the obtained wax was 120.5 mg KOH/g. Wax quality indicators: melting point 71.5 °C, acid value 1.6 mg KOH/g, mass fraction of moisture 0.63 %.

The obtained results make it possible to obtain high-quality waxes from the waste of the oil and fat industry using a safe and affordable substance (sodium chloride). This will make it possible to avoid the need for waste disposal and increase the profitability of oil and fat enterprises due to obtaining valuable products from production waste

Keywords: spent perlite, oil winterization waste, wax saponification value, filter powder

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1. Introduction

Winterization is a stage of oil refining in which waxy substances are removed. This process takes place by cooling the oil and holding it at a temperature of up to 6 °C, followed by the separation of the resulting sediment. At the same time, filter powders are used that improve the drainage properties of sediment – kieselguhr, perlite, etc. Spent filter powders contain a significant amount of oil (up to 34 %) and waxes (up to 28 %). Disposal of winterization waste exacerbates the problem of environmental pollution. The waste has a significant oil concentration and contact surface. The specific surface area of pearlite powder can reach 24,000 cm^2/g. When such waste comes into contact with air oxygen, intense oil oxidation and heat release occur. This can cause self-ignition and release of toxic substances into the atmosphere [1].

Winterization waste processing usually involves the use of volatile and explosive and fire-hazardous solvents: hexane, methylene dichloride, isopropyl and ethyl alcohol. The ignition temperature of these substances is –22; 14; 11.7 and 12 °C, respectively [2]. Therefore, special conditions and equipment are needed to reduce the level of danger in production.

The current direction is the use of alternative methods for extracting the lipid part from spent filter powders. The application of electrolyte solutions, in particular sodium chloride, for waste treatment is promising. In addition to greater safety, the use of sodium chloride is more appropriate from an economic point of view: the cost of hexane is $11.5/kg, sodium chloride – $0.6/kg [3].

Processing of waste for extracting wax and oil reduces the cost of products containing wax and oil and improves the environment. The cost of beeswax is $4.3/kg, and sunflower wax obtained from winterization waste using organic solvents – $1.5/kg [4]. However, the use of dangerous organic solvents remains a problem.

Waxes are used in chemical, cosmetic, pharmaceutical, paint and other industries, as well as in construction [5].

Thus, the development of safe and effective technologies for obtaining waxes from the waste of the oil and fat industry with different physicochemical properties for many industrial purposes is relevant.

2. Literature review and problem statement

Waxes are a mixture of esters of high molecular weight fatty acids and monoatomic higher alcohols, and have a low saponification value due to the high content of non-saponifiable matter. The saponification value is inversely proportional to the length of the fatty acid chains that make up the wax. This indicator allows you to determine the average molecular weight of wax, estimate the content of the lipid part, calculate the required amount of reagents for an effective process involving the product, etc. Therefore, the saponification value of wax is an important technological characteristic.

There are different ways to extract waxes. Usually, in order to obtain waxes, the spent filter powder is treated with an organic solvent and the waxes are separated at low temperatures. Thus, the authors [6] studied various solvents for extracting waxes (n-hexane, dichloromethane, and acetone). Hexane turned out to be the most effective (the completeness of wax extraction is 77 %). The disadvantage is the lack of influence of the process conditions on the quality indicators of wax. The paper [7] compared the results of wax and oil separation using hexane and ethanol, followed by fractionation. Extraction with ethanol showed a higher wax yield (15 %) compared to hexane (11 %). However, there are no data on the rational process conditions and the saponification value of waxes.

The paper [8] investigated the extraction of sunflower wax from reservoir sediment containing 16.9 % wax. But no rational parameters for obtaining waxes and important physico-chemical parameters of the product are shown. The authors [9] investigated the extraction of waxes and oil from the filter sediment after the winterization of sunflower oil with hexane. Wax was obtained from two waste samples with a melting point of 76.70 °C and 80.53 °C. However, the disadvantage is the lack of rational extraction conditions.

An urgent issue is the search for alternative, safer technologies. According to the results of [10], a method of extracting waxes from sorghum using hexane and less toxic solvents: 2-methyltetrahydrofuran and diethoxymethane was considered. Each solvent was used for extraction at temperatures of (25–80) °C. Methyltetrahydrofuran showed maximum efficiency. The disadvantage of the study is the need for further separation of waxes from the obtained product. According to [11], the effectiveness of lipid extraction using cyclopentyl methyl ether, dimethyl carbonate, isopropanol, ethanol, ethyl acetate, d-limonene as an alternative to hexane was investigated. But the process of extracting the total lipid part from various types of plant raw materials, without separate extraction of waxes, is shown.

The paper [12] investigated the extraction of hydrocarbons (alkanes) from plant raw materials using mixtures of solvents: hexane:dichloromethane (9:1) and dichloromethane:methanol (9:1). The maximum yield of alkanes was demonstrated by a solvent mixture of hexane:dichloromethane (97 %). This testifies to the prospects of extracting hydrocarbons and other refractory substances of a lipid nature, including waxes, using these substances. However, the disadvantage is the lack of data on the quality indicators of the obtained product, as well as the use of dangerous solvents.

One of the methods for extracting waxes from various raw materials is supercritical carbon dioxide extraction. For example, the work [13] presents the results of separating the by-product of bioethanol production, consisting of triacylglycerols and waxes, into fractions. Supercritical carbon dioxide was used as a solvent (under conditions of 35 °C, 40 MPa and 4.1 h). The maximum yield of oil was 47.5 % by weight with a minimum wax content (1.0 % by weight). The wax-enriched fraction had a melting point of 60 °C. This fraction consisted of waxy compounds, wax esters, and aldehyde dimers. The disadvantage of the study is the lack of data on the quality indicators of the obtained fraction enriched with waxes. The authors [14] showed the production of bamboo green wax by extraction with supercritical carbon dioxide under the conditions of 26 MPa, 55 °C and 56 min. The wax yield was 2.327 %. But the influence of extraction parameters on the quality parameters of the wax is not shown. The paper [15] presents data on the supercritical carbon dioxide extraction of wax from date palm leaf waste. The wax obtained under optimal conditions (400 bar, 100 °C) had a melting point of 78 °C. The wax yield was 97 %. However, the influence of extraction parameters on the quality parameters of the wax is not shown. Another disadvantage of this method is the use of high pressure.

According to [3], it is effective to obtain waxes from spent perlite using sodium chloride. Rational conditions for processing perlite were determined: the concentration of sodium chloride solution was 7.5 %, the settling temperature – 20 °C, and the settling time – 10 hours. The yield of wax is 14.3 %. However, the influence of perlite processing parameters on the saponification value of wax has not been considered. Also, the duration of boiling perlite with sodium chloride solution is not taken into account as a factor of variation. This is important because long-term heating of the mass leads to oxidation of the lipid component, which deteriorates the quality of the products.

Thus, during the production of waxes, an unsolved problem is the use of dangerous solvents, as well as the lack of data on the influence of extraction parameters on the quality indicators of waxes. In particular, determining the influence of extraction parameters on the saponification value of waxes remains an unresolved issue. This indicator is an important technological parameter that allows a rational use of resources and efficient production process.

3. The aim and objectives of the study

The aim of the study is to determine rational conditions for processing spent perlite with sodium chloride: the concentration of dry sodium chloride and the duration of mass boiling. This will make it possible to obtain high-quality wax from oil winterization waste using advanced safe technology. The results of the research will make it possible to predict the saponification value of the obtained wax.

To achieve the aim, the following objectives were accomplished:

– to determine the dependence of the saponification value of wax on the conditions of processing spent perlite with sodium chloride: the concentration of dry sodium chloride and the duration of mass boiling;

– to investigate the physical and chemical parameters of wax obtained under rational conditions.

4. Materials and methods of research

4. 1. Object and hypothesis of the research

The object of the research is the process of separation of oil winterization waste (used perlite) into perlite, oil and wax. The main hypothesis of the research is that the concentration of dry sodium chloride and the duration of mass boiling affect the saponification value of extracted waxes. The study suggested that increasing the concentration of sodium chloride in the mass makes it possible to reduce the duration of boiling, which has a positive effect on the quality of products: oil and waxes. The simplification is adopted that the purity of the electrolyte used (sodium chloride) does not affect the indicators of the obtained wax, since washing of the finished product is used.

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

The following reagents and materials were used:

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

– spent perlite (CAS Number 93763-70-3).

4. 3. Methods for determining the quality indicators of spent perlite and waxes

The physico-chemical indicators of perlite are determined using generally accepted methods given in SOU 15.4-37-210:2004. The acid and peroxide values are determined according to ISO 660 and ISO 3960 standards, respectively.

The physico-chemical indicators of waxes are determined according to the generally accepted methods presented in the international documentation on the analysis of fatty substances. The melting point is determined according to ISO 6321, mass fraction of moisture – ISO 662, acid value – ISO 660, saponification value – ISO 3657.

4. 4. Methods for extraction using sodium chloride

Wax extraction was carried out according to the method given in [3]. The temperature of mass settling was 20 °C, the duration – 10 hours. The ratio of perlite: water was 1:2.

4. 5. Planning of experimental studies and processing of results

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. In the Statistica package, calculations were made using the "General Regression Models" module, which combines the procedures necessary for data processing. 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 rational conditions for processing spent perlite with sodium chloride

5. 1. Determination of the dependence of the wax saponification value on the conditions of spent perlite treatment with sodium chloride

Spent perlite according to SOU 15.4-37-210:2004 (CAS Number 93763-70-3), obtained after the winterization of sunflower oil, was used as the initial raw material. The following indicators of perlite were determined: mass fraction of fat – 20.1 %, peroxide value – 19.5 ½ O mmol/kg, acid value – 3.5 mg KOH/g. Boiling of perlite with sodium chloride solution, settling of the mass, washing and drying of the wax were used to extract waxes. The influence of the conditions of perlite treatment with sodium chloride on the important quality indicator of wax – the saponification value – has been determined. Variation factors and intervals:

– x_1 – concentration of dry sodium chloride (by weight of perlite): from 5 to 35 %;

– x_2 – duration of boiling: from 10 to 90 min.

The response function is the wax saponification value, mg KOH/g. The results were processed using the StatSoftStatistica v6.0 package (USA). The calculated regression dependence of the wax saponification value on the perlite processing conditions in real variables has the following form:

$$y = 176.88 + 1.13 \cdot x_1 - 0.07 \cdot x_1^2 - 0.46 \cdot x_2 + 0.002 \cdot x_2^2. \quad (1)$$

The significance level of the coefficients of the regression equation ($p>0.05$) and the coefficient of determination (0.999) were determined. Table 1 presents the planning matrix, experimental and calculated values of the response function.

Graphically, the dependence of the saponification value on the conditions of perlite treatment with sodium chloride is shown in Fig. 1.

Table 1

Planning matrix and response function values

Experiment number	Factors of variation		Wax saponification value, mg KOH/g (experimental value)	Wax saponification value, mg KOH/g (calculated value)
	Concentration of dry sodium chloride, %	Duration of boiling, min		
1	5	10	176	176
2	5	50	162	162
3	5	90	155	154
4	20	10	167	167
5	20	50	152	153
6	20	90	145	145
7	35	10	125	125
8	35	50	112	111
9	35	90	102	103

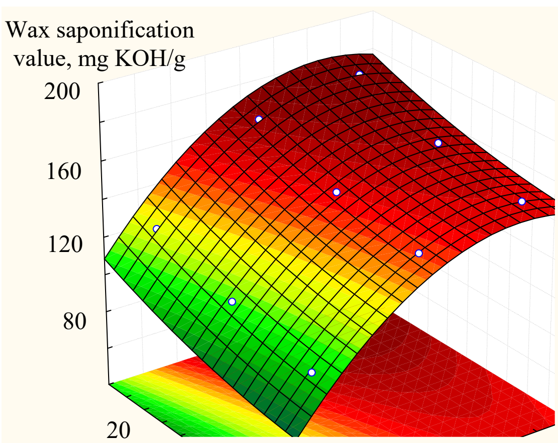

Fig. 1. Dependence of the wax saponification value on the concentration of dry sodium chloride and duration of boiling

With an increase in boiling duration from 10 to 90 min, the saponification value of waxes decreases by (21–22) mg KOH/g. When the concentration of dry sodium chloride increases (with a boiling time of 50 min.) from 5 to 20 %, the saponification value decreases by only 10 mg KOH/g, and from 20 to 35 % – by 40 mg KOH/g. It is rational to use the maximum concentration of dry sodium chloride (35 %) and the duration of boiling of 30 minutes. Under these conditions, the calculated value of the response function was 118.5 mg KOH/g, the experimental value – 120.5 mg KOH/g.

5. 2. Research of the physico-chemical indicators of wax obtained under rational conditions

A comparative analysis of the physicochemical indicators of the obtained wax and sunflower wax was performed. Table 2 shows the indicators of wax obtained in this study, as well as sunflower wax according to the literature data [16].

Table 2

Physico-chemical indicators of waxes

Indicator	Wax obtained in the study	Sunflower wax (according to [16])
Melting point, °C	71.5	70–75
Saponification value, mg KOH/g	120.5	110–124
Acid value, mg KOH/g	1.6	2–17
Mass fraction of moisture, %	0.63	–

Therefore, the quality indicators of the extracted wax correspond to the indicators for sunflower wax given in the scientific and technical literature [16].

6. Discussion of the results of the study of rational conditions for processing spent perlite with sodium chloride

The technology for extracting waxes from spent perlite using sodium chloride has been improved. According to equation (1), Table 1 and Fig. 1, rational conditions for perlite processing are determined: the concentration of dry sodium chloride is 35.0 %, the duration of boiling – 30 minutes. Under these conditions, the experimental value of the wax saponification value was 120.5 mg KOH/g, which corresponds to the literature data on the saponification value of sunflower wax. Sunflower oil has a saponification value of (188–194) mg KOH/g, and sunflower wax – (110–124) mg KOH/g [16]. The decrease in the wax saponification value with an increase in the concentration of dry sodium chloride and the duration of boiling is explained by the fact that the efficiency of wax extraction increases, and the content of impurities and oil in the product decreases.

Boiling the mixture of perlite, water and sodium chloride helps to effectively mix the components at high temperatures. As the concentration of sodium chloride increases, the density of the solution increases, and the separation of the light phase (mixture of oil and wax) is intensified. The presence of a dense solution of sodium chloride also facilitates the separation of oil and wax, since the density of oil is less than that of wax. If there is soap in the system, the presence of an electrolyte prevents emulsification. Consequently, the separation of components is intensified.

The concentration of dry sodium chloride has a greater effect on the response function than the duration of boiling. According to equation (1), Table 1 and Fig. 1, it is effective to use the maximum concentration of dry sodium chloride (35 %). Boiling time does less to decrease the saponification value, but using the minimum value is ineffective. Therefore, the rational value of this parameter is 30 min. Reducing the duration of boiling is advisable, because reducing the temperature treatment of the mass helps to reduce the intensity of oxidation processes and increase the quality of products. Compared to the data [3], where the duration of boiling perlite with a sodium chloride solution is 1 hour, wax obtained under the conditions of a shorter duration of boiling has a higher quality (Table 2): higher melting point, lower acid value and mass fraction of moisture.

The results of the work make it possible to rationally and safely use the waste of oil winterization to obtain a valuable product – wax.

The papers [6–15] provide data on the extraction of waxes using various solvents. The authors [10, 11] showed the possibility of using solvents alternative to hexane. According to [3], the use of sodium chloride solution for removing waxes is effective and appropriate. However, the influence of the processing conditions of the experimental raw materials on the saponification value of waxes is not shown. Therefore, the unsolved problem is the use of dangerous solvents and the lack of dependence between processing parameters and quality indicators of waxes, in particular, the saponification value. But there is not enough data regarding the influence of the processing conditions of oil and fat waste on important technological indicators of waxes, in particular, the saponification value. This task is solved in this work. The advantages of this study in comparison with similar known ones [6–15] are the use of a safe and affordable substance (sodium chloride) for the effective extraction of high-quality waxes, as well as the influence of the parameters of the extraction process on an important quality indicator of waxes – the saponification value.

A limitation of the use of research results is the intervals of factor variation. It is also necessary to wash the waxes from sodium chloride residues and dry the waxes under vacuum conditions to prevent oxidation processes in the product.

The disadvantage of the study is the significant duration of mass settling (10 hours). It is advisable to develop a method of accelerating phase separation.

A promising area of research is to determine the indicators and the possibility of using oil, which is also an important product in this process. This will contribute to a more complete and effective use of oil and fat production waste.

7. Conclusions

1. The dependence of the wax saponification value on the conditions of treatment of spent perlite with sodium chloride was determined in the form of the second-degree regression equation. The concentration of dry sodium chloride has a greater effect on the response function than the duration of boiling. With an increase in boiling duration from 10 to 90 min., the saponification value of waxes decreases by (21–22) mg KOH/g. When the concentration of dry sodium chloride increases (with a boiling time of 50 min) from 5 to 20 %, the saponification value decreases by only 10 mg KOH/g, and from 20 to 35 % – by 40 mg KOH/g. Rational conditions for perlite processing were determined: concentration of dry sodium chloride (to the perlite mass) – 35.0 %, duration of boiling – 30 min. Under these conditions, the experimental value of the wax saponification value was 120.5 mg KOH/g.

2. The physicochemical indicators of wax obtained under rational conditions were studied: melting point 71.5 °C, acid value 1.6 mg KOH/g, mass fraction of moisture 0.63 %. The obtained data show that the quality indicators of wax correspond to the standard indicators of sunflower wax.

Conflict of interest

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

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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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