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RATIONAL PARAMETERS OF WAXES OBTAINING FROM OIL WINTERIZATION WASTE

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Production of waxes from spent perlite, which is a waste of sunflower oil winterization, is studied.

Winterization is characterized by significant losses of oil with filter powders, and waste utilization is an environmental and economic problem. At the same time, winterization waste contains valuable components – wax and oil, which can be used in different ways.

The content of waxes in spent perlite using hexane (18 %), as well as the quality indicators of the obtained wax: melting point 70 °C, saponification number 115 mg KOH/g, acid number 2.6 mg KOH/g, mass fraction of moisture 0,82 % are determined.

Spent perlite was treated with a solution of sodium chloride during boiling, settling of the obtained mass, washing and drying of wax. The dependence of the yield and melting point of the extracted waxes on the processing parameters: the concentration of sodium chloride solution, temperature and duration of settling is found.

Rational conditions for spent perlite processing are determined: the concentration of sodium chloride solution – 7.5 %, settling temperature – 20 °C, settling duration – 10 hours. The experimentally determined wax yield at this point is 14.3 %.

Quality indicators of the wax sample obtained under rational conditions are studied: melting point 68 °C, saponification number 110 mg KOH/g, acid number 2.8 mg KOH/g, mass fraction of moisture 0.85 %. These values correlate with the data for wax extracted using hexane, as well as with reference data on the quality of beeswax and sunflower wax.

The data obtained allow recycling spent perlite without organic solvents, which makes the process more environmentally friendly and cost-effective, as well as solves environmental problems associated with the utilization of winterization waste

Keywords: waste of oil and fat industry, winterization, spent filter powder, perlite, sunflower wax

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1. Introduction

All stages of oil and fat refining generate waste is that needs to be disposed of or further processed. Currently, one of the most relevant research areas is the development and improvement of oil and fat waste processing technologies to obtain valuable products for many food and non-food purposes [1].

The main oil and fat waste in the production of vegetable oils are oilseed cleaning waste, soapstock, waste bleaching clays and filter powders. These wastes contain significant amounts of fat, waxes and soap [2]. However, waste recycling requires additional costs, equipment, reagents and rational technologies to be developed.

One of the main causes of environmental pollution is the open resource cycle, which leads to the accumulation of waste. Due to its volume, the production of vegetable oils is accompanied by the generation of significant amounts of waste, processing which is relevant both for the environmental safety of production and for economic reasons.

At the stage of oil winterization, when waxy substances are removed, filter powders are used, which improve the drainage properties of the sludge – diatomites (kieselguhrs), perlites, etc. During winterization, a large amount of spent filter powders is formed, containing significant amounts of oil and waxes (final oil content of filter powders can reach up to 50 %, wax content – 16 %) [3].

Utilization of waste of the winterization stage as household waste causes a serious problem of environmental pollution. Spent filter powders have a significant contact surface, high oil concentration, and, consequently, in the contact of such a surface with oxygen, oil is oxidized with significant heat release [4]. This can cause spontaneous combustion of spent filter powders. At the same time, a large amount of toxic substances enters the atmosphere. Currently, one of the priority research areas in the world is to reduce the negative environmental impact of oil and fat production waste. To this end, enterprises introduce innovative resource-saving processing technologies and special equipment [5].

Winterization waste can also be used as fertilizers, feed additives, in the production of detergents and building materials. However, valuable components (wax and oil) are not used [6].

The use of waste as raw materials for new products can significantly reduce production costs and at the same time solves a number of environmental problems. Thus, the cost of beeswax is $ 4.3/kg, carnauba – $ 46.4/kg, and sunflower wax obtained using organic solvents from winterization waste – $ 1.5/kg. However, the use of solvents increases the danger in enterprises, requires special equipment, premises that reduce the harmful effects of solvent vapors on workers and the danger of using these substances [6].

Currently, there is a worldwide trend to switch to low-waste and non-waste technologies for the production and processing of oils. Rational use of all components of plant raw materials is a priority, which will reduce industrial waste, increase production profitability and reduce its negative impact on the environment [5].

Therefore, the search for rational, cost-effective technologies for processing spent filter powders is an important task in general. And, given the dangers of organic solvents, it is necessary to find more environmentally friendly ways to recycle waste without the use of hazardous substances.

2. Literature review and problem statement

Spent filter powder contains a significant amount of oil and waxes – up to 34 % and 28 %, respectively [3]. The rational use of this waste is the separation into filter powder, wax and oil. Under the conditions of appropriate processing, filter powder can be returned to the oil winterization stage, the oil can be used for technical purposes. A particularly valuable component is wax, which is used in many industries. Waxes are used in the food, cosmetic, pharmaceutical, paint, chemical and many other industries. The addition of waxes allows creating cosmetics of the desired consistency and with high heat resistance. Waxes are widely used in the manufacture of various types of coatings, in particular, for wooden products [7].

Wax is used in the production of modern thermoplastics for road marking, as well as asphalt-concrete mixes, antifriction materials [8, 9].

In the food industry, wax is used in confectionery, to create margarine recipes [10].

An innovative way to use sunflower wax is creating a hydrophobic paraffin-wax film former on the surface of winter wheat seeds. This film-forming agent protects seeds from moisture loss, regulates the germination of seeds, helps preserve accumulated nutrients, enhances growth, plant development and increases wheat grain yield. Effective use of sunflower wax is due to the fact that it contains, in addition to wax esters, saturated higher fatty acids from C16 to C24 and alcohols of plant origin. The presence of surfactant fatty acids gives waxes not only a solid consistency at room temperature, but also adhesive properties in relation to samples of plant origin. The use of sunflower wax simultaneously solves an important environmental problem of oil production waste disposal. Pre-sowing heat treatment of winter wheat seeds in a paraffin-wax melt at the temperature of (80–85) °C contributes to the death of pathogens and pests. This makes it possible to do without expensive plant protection products, reducing the cost of pesticides [11].

Due to the wide application possibilities of waxes, technologies for their production are promising. At the same time, waxes can be obtained from the waste of oil and fat production – spent filter powders. There are various ways to extract waxes from the given type of waste. For this, the spent powder is usually treated with a solvent, and wax substances are precipitated from the extract at reduced temperatures.

In [12], the results of research on the regeneration of spent filter powder using methylene dichloride as a solvent are presented. However, methylene dichloride is capable of hydrolysis, resulting in the formation of chlorine derivatives that interact with the oil.

The authors [13] showed the possibility of using a propane-butyl mixture as a solvent for wax extraction. The disadvantage of this method is the significant loss of solvent during the process, which increases the cost of waxes.

In [14], the use of trichlorethylene for wax separation is investigated. At the same time, an ultrasound that intensifies the wax separation process is used. However, this technology is multi-stage and requires special equipment, which increases the cost of the resulting wax substances.

In [15], hexane is used to separate the filter powder, oil and waxes, resulting in sunflower wax from two different samples of spent filter powder with a melting point of 76.70 °C and 80.53 °C. However, an explosive, flammable solvent is used, which requires special equipment and premises for the process. There are also significant losses of solvent during filter powder processing.

The authors [16] showed the extraction of waxes from spent filter powder using a catalytic process of alcoholysis carried out directly with the filter powder, without prior extraction of the oil and wax mixture. Ethyl alcohol was used as the reagent. The disadvantage of this method is the use of chemical compounds, special hardware, which complicates the process and increases the product cost.

Thus, the processing of winterization waste is a rather complex process, which involves the use of explosive, flammable organic solvents (in particular, hexane, ethyl alcohol) and special chemicals. The use of these substances requires strict control of equipment and assessment of industrial hazards in the workplace [17].

An urgent task now is the development of technologies for obtaining waxes from spent filter powders without organic solvents. This is possible due to the sequential separation of the lipid part (waxes and oils) from the spent filter powder, settling and separation of waxes from the oil under certain conditions (temperature and duration of settling). This method will increase the safety of processing winterization waste and reduce the cost of the resulting wax. An unresolved issue in this direction is setting rational parameters and conditions for separating perlite, oil and waxes.

3. The aim and objectives of the study

The aim of the study was to determine the dependence of the yield and melting point of wax substances on processing parameters of spent filter powder (perlite): concentration of sodium chloride solution, temperature and duration of settling. This will reduce the negative impact of fat and oil production waste on the environment and provide a valuable component for many industries (sunflower wax). The research results will help reduce the cost of waxes extracted from oil winterization waste and increase the profitability of oil and fat enterprises through the sale of waxes.

To achieve the aim, the following objectives were set:

– to investigate the wax content in spent perlite (using the organic solvent – hexane) and quality of the obtained wax;

– to determine experimentally the influence of waste perlite processing parameters (concentration of sodium chloride solution, temperature and duration of settling) on the yield and melting point of waxes and determine rational parameters of spent perlite treatment;

– to compare the quality of wax obtained using the organic solvent (hexane) and sodium chloride solution.

4. Materials and methods to study the dependence of wax extraction efficiency on perlite processing parameters

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

The following reagents and materials were used in this study:

– rectified ethyl alcohol, according to DSTU 4221:2003;

– distilled water, according to the current normative documentation;

– potassium iodide, “pure chemical” grade, according to the current normative documentation;

– soluble starch, according to the current normative documentation;

– sodium thiosulfate, according to the current normative documentation;

– acetic acid, according to the current normative documentation;

– chloroform, according to the current normative documentation;

– potassium hydroxide, “clean for analysis” grade, according to the current normative documentation;

– phenolphthalein, according to the current normative documentation;

– sodium chloride, according to DSTU 3583;

– hexane, “pure chemical” grade, according to TU 6-09-06-657.

4. 2. Procedure for determination of wax parameters

The acid number of waxes is determined by standard methods according to DSTU ISO 660: 2009.

The saponification number of waxes is determined by the standard method according to DSTU ISO 3657:2004.

The mass fraction of moisture and volatile substances is determined by the standard method according to DSTU 4603:2006.

The wax melting point by the open capillary method is determined by the standard method according to DSTU 6321:2003.

4. 3. Procedure for wax extraction using organic solvent

A portion of the spent filter powder is placed in a conical flask, the organic solvent – hexane is added. An air refrigerator is attached to the flask. The contents of the flask are heated with an electric stove. Under boiling hexane, extraction is carried out for 1 hour. At the end of the process, the perlite is separated from the oil and wax solution in hexane by settling. Waxes are crystallized from the solution (process duration 10–12 hours), which are separated by filtering.

4. 4. Procedure for wax extraction using sodium chloride solution

A portion of the spent filter powder is placed in a conical flask, an aqueous sodium chloride solution of a given concentration is added. The contents of the flask are heated with an electric stove and boiled for 1 hour. At the end of the process, the resulting mixture is slowly cooled and separated by settling at a given temperature and duration. The settling temperature is set and maintained using an oven. The mixture is divided into the sediment (perlite), aqueous layer, wax layer and top layer – oil. After settling, the wax layer is separated, washed with hot water, and the wax is dried in a vacuum.

4. 5. Planning experimental research and processing results

In order to plan our study and process the results obtained, a fractional second-order factorial experiment was applied; the calculation was performed in Microsoft Office Excel 2003 (USA) and Stat Soft Statistica v6.0 (USA). The experiments were repeated twice.

5. Results of studying the dependence of the yield and melting point of waxes on processing parameters of spent perlite

5. 1. Studying the content and quality of waxes obtained using hexane

Since the regeneration of spent filter powders is commonly done with organic solvents, the content of waxes in spent perlite and their quality indicators with the use of hexane have been previously determined. The content of waxes in spent perlite by this method was 18 %. Quality indicators of the obtained wax are given in Table 1.

Table 1

Quality indicators of wax obtained using hexane

Indicator	Value
Melting point, °C	70
Saponification number, mg KOH/g	115
Acid number, mg KOH/g	2.6
Mass fraction of moisture, %	0.82

Thus, the wax has a fairly high melting point, low values of acid number and mass fraction of moisture, which indicates the high quality of the product.

5. 2. Studying the influence of spent perlite processing parameters on the yield and melting point of waxes

The relationship between the main processing parameters of spent perlite and the yield and melting point of waxes is determined. The following parameters of spent perlite treatment were investigated: concentration of sodium chloride solution, temperature and duration of settling. To conduct the experiment and obtain a mathematical model, a fractional factorial experiment was used: number of factors – 3, number of experiments – 9, number of levels – 3. Variation of factors: x_1 – sodium chloride concentration: from 5 to 10 %; x_2 – settling temperature: from 10 to 20 °C; x_3 – settling duration: from 4 to 16 hours. The fractional factorial experiment used in the work is a half-replica of the full factorial experiment 3^3 (the number of factors and variation levels of factors is 3). In this case, a semi-replica 3^{3-1} is used, i.e. the number of experiments is reduced to 9.

Table 2 shows the matrix of experiment planning with actual values of factors, as well as experimentally determined values of response functions.

Table 2

Experiment planning matrix and experimental values of response functions

Experiment No.	Concentration of sodium chloride solution, %	Settling temperature, °C	Settling duration, hours	Wax yield, %	Wax melting point, °C
1	5	10	4	8.1	45
2	5	15	16	9.5	50
3	5	20	10	10.7	57
4	7.5	10	16	12.2	65
5	7.5	15	10	13	68
6	7.5	20	4	13.9	75
7	10	10	10	12.7	67
8	10	15	4	13	71
9	10	20	16	15	80

Fig. 1–4 show the graphical dependence of experimental data on the yield and melting point of waxes on the concentration of sodium chloride and settling temperature.

As a result of processing the experimental data in Stat Soft Statistica v6.0 (USA), mathematical models reflecting the dependence of the response functions on processing parameters of spent perlite were obtained. The response functions are marked as follows: y_1 – wax yield, %; y_2 – melting point of the extracted waxes.

In normalized form, the regression dependence of the wax yield on the processing parameters of perlite has the form:

$$y_1 = 12.98 + 2.07 \cdot x_1 - 1.53 \cdot x_1^2 + \\ +1.1 \cdot x_2 + 0.27 \cdot x_2^2 + 0.28 \cdot x_3. \qquad (1)$$

In real variables:

$$y_1 = -8.34 + 4.5 \cdot x_1 - 0.24 \cdot x_1^2 - \\ -0.1 \cdot x_2 + 0.01 \cdot x_2^2 - 0.05 \cdot x_3. \qquad (2)$$

In normalized form, the regression dependence of wax melting point on the processing parameters of perlite has the form:

$$y_2 = 67.89 + 11.00 \cdot x_1 - 7.67 \cdot x_1^2 + \\ +5.83 \cdot x_2 + 1.83 \cdot x_2^2 + 0.67 \cdot x_3. \qquad (3)$$

In real variables:

$$y_2 = -62.68 + 22.85 \cdot x_1 - 1.23 \cdot x_1^2 + \\ +0.72 \cdot x_2 + 0.07 x_2^2 + 0.11 \cdot x_3. \qquad (4)$$

In the dependences (1)–(4): x_1 – concentration of sodium chloride, %; x_2 – settling temperature, °C; x_3 – settling duration, hours. In equations in normalized form, the values x_1, x_2, x_3 are substituted in coded form (for example, the minimum value of the parameter is denoted by –1 and the maximum +1). In equations with real variables, the values x_1, x_2, x_3 are substituted for calculations in actual dimensions (for example, temperature in °C, duration in hours). Both types of equations are used for calculations, but in general the equations in real variables are convenient.

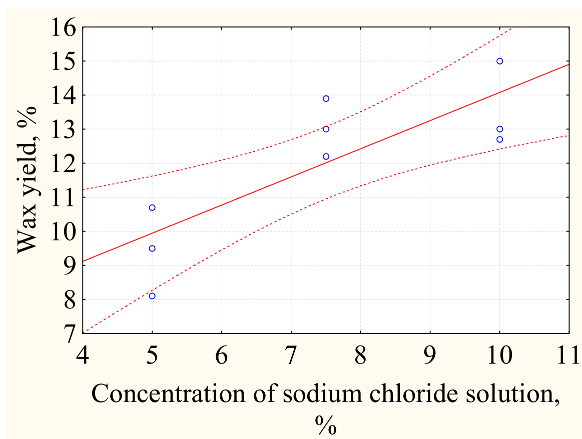

Fig. 1. Dependence of experimental data on the wax yield on the concentration of sodium chloride (experimental data and approximating curves)

The adequacy of the obtained models was checked using Fisher's criterion, which found no loss of consistency (the level of significance of the coefficients $p>0.05$). The values of determination coefficients for the yield and melting point of waxes were 0.99977 and 0.99979, respectively (close to unity). Thus, the obtained models adequately describe the response functions. Table 3 shows the estimated values of the response functions – yield and melting point of the extracted wax substances, determined by equations (2) and (4), respectively. Designations of experiments 1–9 correspond to the planning matrix (Table 1).

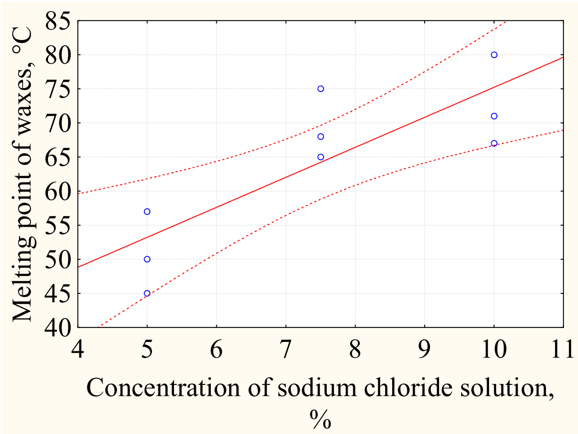

Fig. 2. Dependence of experimental data on the melting point of waxes on the concentration of sodium chloride (experimental data and approximating curves)

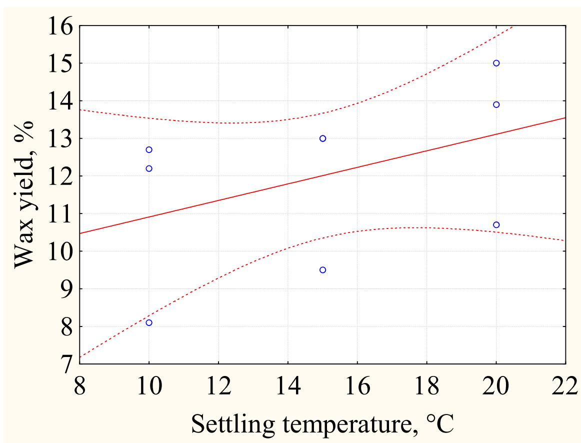

Fig. 3. Dependence of experimental data on the wax yield on settling temperature (experimental data and approximating curves)

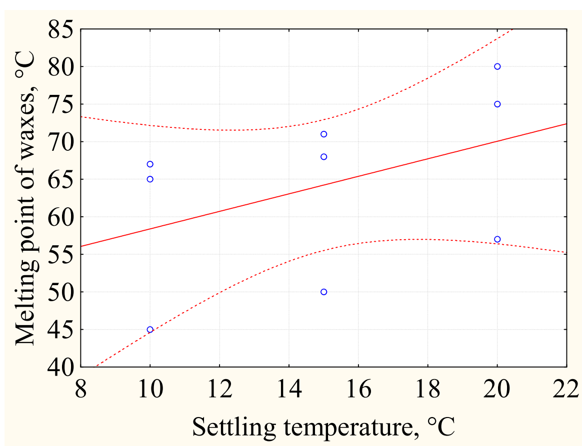

Fig. 4. Dependence of experimental data on the melting point of waxes on settling temperature (experimental data and approximating curves)

Fig. 5 shows the projection of the response surface, which is the dependence of the wax yield on the concentration of sodium chloride solution and settling temperature.

Table 3

Estimated values of yield and melting point of waxes, %

No.	1	2	3	4	5	6	7	8	9
Wax yield, %	8.08	9.48	10.74	12.24	12.98	13.88	12.68	13.04	14.98
Wax melting point, °C	44.89	50.22	56.89	64.89	67.89	75.22	67.22	70.89	79.89

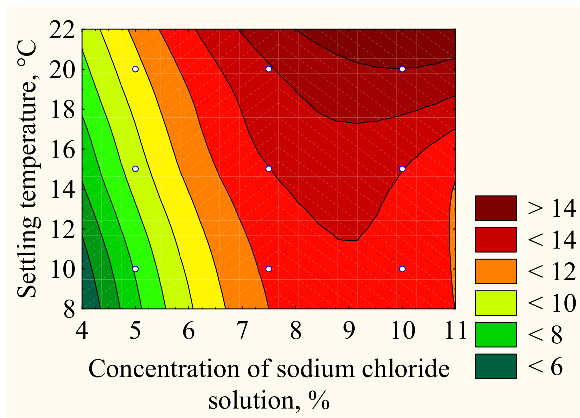

Fig. 5. Dependence of wax yield on the concentration of sodium chloride solution and settling temperature

Based on the analysis of the dependence (2) and Fig. 5, it is found that an increase in the concentration of sodium chloride solution increases the yield of waxes. However, there is no significant difference in the yield of waxes with increasing sodium chloride concentration from 7.5 to 10 %. An increase in the settling temperature also significantly affects the yield of waxes. The duration of settling is not essential for increasing the yield of waxes. Therefore, the rational conditions for perlite processing in terms of wax yield are as follows: concentration of sodium chloride solution – 7.5 %, settling temperature – 20 °C, settling time – 10 hours. The experimentally determined wax yield at this point is 14.3 %.

Fig. 6 shows the projection of the response surface, which is the dependence of wax melting point on the concentration of sodium chloride solution and settling temperature.

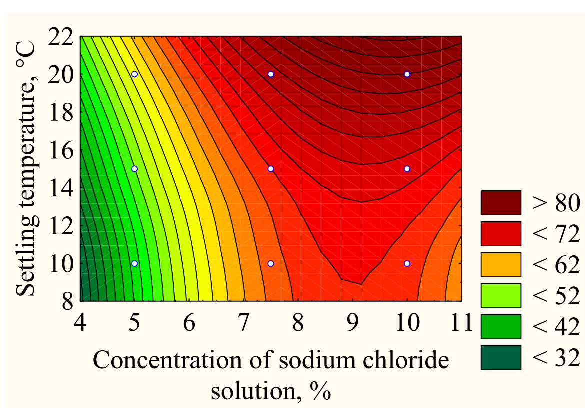

Fig. 6. Dependence of wax melting point on the concentration of sodium chloride solution and settling temperature

By analyzing the regression dependence (4) and Fig. 6, it is found that the dependence of wax melting point on perlite processing parameters is similar to that for the wax yield. An increase in the yield and melting point of waxes indicates an increase in wax extraction efficiency. An increase in sodium chloride concentration and settling temperature is most significant for increasing the melting point of the resulting wax. An increase in the melting point corresponds to higher purity of the waxes, i.e. lower concentration of oil in the resulting wax sample. An increase in settling duration has a less significant effect on the response function. But with increasing duration, the melting point of waxes still increases. Therefore, the rational conditions for perlite processing in terms of yield and melting point of waxes are as follows: concentration of sodium chloride solution – 7.5 %, settling temperature – 20 °C, settling time – 10 hours.

5. 3. Determination of quality indicators of wax obtained using sodium chloride solution

Quality indicators of wax obtained under the determined rational parameters of spent perlite processing are investigated. The values of wax indicators are compared to the literature data on beeswax and sunflower wax indicators [18]. The corresponding data are given in Table 4.

Table 4

Quality indicators of beeswax, sunflower wax and wax obtained using sodium chloride solution

Indicator	Wax obtained using sodium chloride solution	Beeswax	Sunflower wax
Melting point, °C	68	63–69	70–75
Saponification number, mg KOH/g	110	85–101	110–124
Acid number, mg KOH/g	2.8	16–21	2–17
Mass fraction of moisture, %	0.85	–	–

Thus, the quality indicators of wax obtained by boiling with sodium chloride solution are close to those of wax obtained using hexane, from the same spent perlite sample. The quality indicators of the obtained wax correlate with the corresponding values given in the scientific and technical literature for beeswax and sunflower wax. This indicates the possibility of obtaining high-quality sunflower wax from spent perlite by this method.

6. Discussion of the results of studying the dependence of wax extraction efficiency on processing parameters of spent perlite

Rational conditions for processing spent perlite are determined: concentration of sodium chloride solution – 7.5 %, settling temperature – 20 °C, settling duration – 10 hours. The experimentally found wax yield at this point is 14.3 %, melting point 68 °C. Calculations of wax yield and melting point for real variables are performed according to formulas (2) and (4). Quality indicators of the obtained wax are compared to the corresponding indicators of wax obtained with the organic solvent, as well as literature data on beeswax and sunflower wax indicators. The results indicate the high quality of the product, which can be used along with other types of waxes for various industrial needs.

This feature of the work compared to the existing scientific research [12–17] on the regeneration of spent filter powders is the absence of organic solvents and special complex equipment during wax extraction, which favorably distinguishes this technology from others. Therefore, the results of the work are important for the oil and fat and wax-using industries.

As the concentration of sodium chloride increases, the density of the solution increases. This intensifies the separation of the lighter phase (oil and wax). The density of oil is less than that of wax. Therefore, the presence of a dense sodium chloride solution also contributes to oil and wax separation. However, according to research, an increase in the concentration from 7.5 to 10 % has little effect on the yield and melting point of waxes. That is, with the achievement of the appropriate solution density, the rate of mass transfer in the system ceases to increase.

During cooling, the oil and wax mixture solidifies, and the separation of oil and wax stops. The mass becomes homogeneous. During heating and mixing the wax and oil, the oil is separated, after slow cooling and settling, the wax crystallizes. The settling temperature affects the distribution rate of wax and oil fractions. The results showed that under the experimental conditions, the temperature of 20 °C showed the highest yield and melting point of waxes.

The duration of settling also affects wax separation efficiency, but less significantly than other factors. Under the difference between the densities of the solution components and the settling temperature, separation occurs over time. The rational duration of separation is 10 hours.

Therefore, the influence of the parameters of spent perlite processing on the yield and melting point of waxes was determined in the work, the quality of the obtained waxes was compared with that of sunflower wax and beeswax. The characteristics of wax obtained using sodium chloride solution and organic solvent are compared.

When using the results of the study, the recommended rational conditions for spent perlite processing should be taken into account (sodium chloride solution concentration – 7.5 %, settling temperature – 20 °C, settling time – 10 h), lower settling temperature and sodium chloride solution concentration adversely affect wax extraction efficiency. After settling, the waxes contain a sodium chloride solution, therefore, need to be thoroughly washed with water, as well as dried under a vacuum. Drying at elevated temperatures without vacuum will promote the oxidation of residual oil, which will adversely affect wax quality.

A promising area of research on the extraction of waxes from spent perlite by this method is to determine the purity, composition and parameters of phase transitions of the extracted waxes. These data will contribute to a more detailed study of this method of wax extraction and more efficient use of waxes in the industry. Different industries require different characteristics of raw materials, in particular, wax substances. Varying the processing parameters of spent perlite provides waxes with different indicators. Combined with studies of the composition and temperature behavior of waxes, this will make it possible to produce waxes from winterization waste for well-targeted purposes.

7. Conclusions

1. As a result of experimental research, the content of waxes in experimental spent perlite (using the organic solvent – hexane) and quality indicators of the resulting

wax are defined. The wax content was 18 %, the quality indicators of the obtained wax are as follows: melting point 70 °C, saponification number 115 mg KOH/g, acid number 2.6 mg KOH/g, mass fraction of moisture 0.82 %.

2. The influence of the parameters of spent perlite processing on the yield and melting point of waxes is determined. An increase in the concentration of sodium chloride solution and settling temperature has a significant effect on the yield and melting point of waxes. Rational conditions for spent perlite processing are as follows: concentration of sodium chloride solution – 7.5 %, settling temperature – 20 °C, settling duration – 10 hours. The experimentally determined wax yield at this point is 14.3 %, melting point 68 °C.

3. A comparative analysis of the quality indicators of wax obtained using hexane and sodium chloride solution was performed, which indicates the similar quality of waxes obtained by these two methods. The quality indicators of wax obtained using hexane are as follows: melting point 70 °C, saponification number 115 mg KOH/g, acid number 2.6 mg KOH/g, mass fraction of moisture 0.82 %. The quality indicators of wax obtained using the sodium chloride solution: saponification number 110 mg KOH/g, acid number 2.8 mg KOH/g, mass fraction of moisture 0.85 %. It is shown that the obtained results in terms of quality correlate with the corresponding literature data for beeswax and sunflower wax. Therefore, this technology provides high-quality waxes without using hazardous chemical reagents. This will contribute to the rational use of oil and fat waste, improve the environment and increase the profitability of oil and fat enterprises through the production of waxes for the needs of various industries.

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