Chabanova, Oksana; Bondar, Sergii; Kotliar, Yevhenii et al.

Article

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**Oksana Chabanova,
Sergii Bondar,
Yevhenii Kotliar,
Tatiana Nedobiychuk,
Yakiv Verkhivker**

ANALYSIS OF THE PECTIN EXTRACTION PROCESS AT RECYCLING OF SECONDARY MATERIAL RESOURCES

The object of research is the secondary material resources of processing plant and animal raw materials, namely fruit pomace and milk whey. One of the most problematic areas is that the waste of these industries has high rates of biological and chemical oxygen consumption of wastewater, which significantly impairs the operation of local and city treatment facilities. In addition, the parameters of extraction and determination of the quality indicators of the obtained product are not well defined. The processing of whey and pomace of fruit crops can reduce the environmental burden on the environment and increase the efficiency of technological processes through resource conservation and obtaining a surplus product. The problem is solved, in particular, by using the process of extracting plant waste using milk whey and the process of energy-saving membrane concentration.

In the course of the study, let's use pectin-containing plant waste from juice production, namely watermelon, pumpkin, quince, beetroot, apple and a mixture of orange and tangerine pomace. The results obtained indicate that the process of extracting apple pomace with milk whey is promising, since the highest pectin content in the extract is established for apple pomace. The main amount of pectin substances passes into the extract starting from 75 minutes to 90 minutes. Extraction-hydrolysis for 2 hours at a temperature of 85 °C, pH=2–2.5 units determines the best results. Ultrafiltration of the obtained extract makes it possible to increase the pectin content up to 3.0 % with a protein content of more than 6.0 %. The membranes used in the experiment are characterized by high selectivity for protein and pectin. The fact is established that diafiltration makes it possible to effectively purify whey-pectin concentrates from ballast impurities with simultaneous enrichment of the concentrate with high-molecular components of whey. A technological scheme for the production of pectin-whey concentrate with high organoleptic and detoxification characteristics is proposed. The proposed technological process has a number of positive features, in particular, a high content of pectin substances, high value indicators, a significant reduction in the burden on the environment, economic benefits through energy conservation and obtaining a surplus product.

Keywords: *whey, juice production waste, pectin extraction, ultrafiltration, pectin substances, pectin-whey concentrate, waste disposal.*

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1. Introduction

Whey is one of the waste products of milk processing and a valuable reserve in the dairy industry. Due to its composition, properties and biological value, there is an urgent need for its full use in food production. In addition, the processing of whey without losses allows to reduce the environmental burden on the environment. Waste of juice production, namely pomace, is of the same importance. They can be considered as additional sources of valuable substances of natural origin, in particular pectin substances.

The most promising way to utilize pomace is to remove from them a valuable component – pectin, which has a high detoxifying effect and contributes to the normal course of physiological processes.

The relevance of the work lies in the development of a method for recycling waste from the dairy and canning industry solves the problem of resource conservation and greening of production and allows to obtain a valuable semi-finished product – pectin-whey concentrate, as the main component in the formulation of medical and prophylactic drinks.

2. The object of research and its technological audit

The object of research is the secondary material resources of processing plant and animal raw materials, namely fruit pomace and milk whey. Whey and fruit pomace obtained in the production of juices by pressing or rubbing quince, watermelon, apples, citrus fruits, containing a very valuable component – pectin. Indicators of the content of valuable components of these objects are given in Tables 1, 2.

Table 1

Whey chemical composition

Indicator	Value
Dry substances mass fraction of dry substances, %	6.6
Fat mass fraction of fat, %	0.2
Protein mass fraction, %	0.85
Lactose mass fraction of lactose, %	4.7

Table 2

Mass fraction of pectin substances in plant waste

Waste	Mass fraction of pectin substances, %
Watermelon pomace	0.03–0.06
Pumpkin pomace	0.99–1.2
Mixture of orange and tangerine pomace	2.7–3.1
Quince pomace	2.31–2.85
Beet pomace	1.85–2.1
Pomace	2.1–2.4

One of the most problematic areas in the use of these facilities is the extremely low degree of recycling under the existing conditions of production and the high content of substances easily assimilated by putrefactive microorganisms, which causes a high load on the treatment facilities and the environment.

3. The aim and objectives of research

The aim of research is to analyze the technological process of extracting secondary material resources of processing plant and animal raw materials.

This aim requires the solution of the following objectives:

1. To investigate the parameters of extraction of pectin substances from plant raw materials with milk whey.
2. To investigate the membrane process of concentration and purification of pectin-whey extract.
3. To determine the qualitative characteristics of the obtained pectin-whey extracts.

4. Research of existing solutions o to the problem

The catastrophic deterioration of the ecological situation is accompanied by the pollution of the environment and foodstuffs with toxic substances and radionuclides. This requires food safety and preventive measures. It causes the expansion of the production of pectin-containing products with detoxifying properties [1, 2].

Pectin deficiency is an important problem in the nutrition of the population of many countries of the world. Pectins are one of the most common polysaccharides contained in sufficient quantities in plant materials, in particular in fruit and vegetable pomace. Pectins are a detoxifier that normalizes the amount of cholesterol, increases the body's resistance to allergies and helps to restore the mucous membranes of the respiratory and digestive tract [3, 4].

The production of pectin, despite the general recognition of their usefulness and necessity, is still far from satisfying market requirements. The need for pectin significantly exceeds the volume of its production [4, 5].

The pectin production technology has a number of main stages: preparation of raw materials, hydrolysis-extraction of pectin substances, filtration of the suspension, increasing the concentration of dry substances, precipitation of pectin with alcohol, drying and grinding of the product [6, 7].

Depending on the used hydrolysis agent, hydrolysis-extraction can be divided into: acidic, saline and alkaline, of which the most common is acidic extraction [8, 9]. For hydrolysis-extraction of pectin substances, solutions of both mineral (nitric, hydrochloric, sulfuric) and organic (oxalic, lactic, citric) acids, as well as solutions of salts and alkali, are used. The hydrolysis of pectin substances is usually carried out in the acidic pH range (1.0–1.3) in different lengths of time (20–360 min) and temperature (60–100 °C) [9, 10]. The main problems are the degradation of pectin macromolecules and the loss of properties of the target products [11, 12].

Concentration of pectin extract is one of the most important stages of the technological process, on which the consumer quality of the product, liquid pectin concentrate, largely depends. The traditional methods of concentration and purification of pectin extracts include the processes of precipitation with ethyl alcohol, concentration on rotary film apparatus, cleaning with ethyl alcohol, acids, alkalis; by the method of treatment with ion-exchange resins, etc. [5, 8]. The traditional technology for the production of pectin (citrus, apple, beetroot) involves the use of caustic mineral acids, alkalis, ethyl alcohol and other chemical and explosive substances, does not provide environmental cleanliness and wasteless production, and also increases the requirements for equipment [11, 12]. Among the modern methods of concentrating pectin extracts, concentration using membrane technologies is recognized, which significantly affects the quality of the obtained pectin concentrates [6, 13].

In literary sources, little attention is paid to the problem of concentration, since it is believed that this problem is successfully solved by vacuum evaporation and, as an alternative, membrane treatment of liquid pectin extracts. However, these processes have many drawbacks that hinder their widespread adoption. Vacuum evaporation, for example, is a rather complex hardware-based process that justifies itself only with large-scale and long-term operation. Membrane methods win in the energy consumption of the concentration process, but are limited by the phenomenon of concentration polarization, the solubility of pectin in an aqueous medium, and the high cost of foreign equipment in the absence of domestic analogues [14].

Among the main directions for solving this problem in the resources of the world scientific periodicals, they can be distinguished [1, 2], but they do not consider the influence of the extraction parameters on the yield of pectin and subsequent membrane processing of concentrates. The work [6] is devoted to the properties and application of pectin, however, there is an unresolved issue of the complex use of plant and animal raw materials. Extraction of pectin is the main link in obtaining pectin, as indicated in [10]. However, this work does not fully disclose the effect of the type of extractant on the yield and properties of pectin. The authors of [6] have shown that nanotechnology occupies a promising position in the production of pectin. But there remains the question of widespread introduction of membrane diafiltration to improve the properties of pectin.

An alternative solution to the problem described in [9] does not provide for the use of membrane technologies for processing pectin concentrates. Acidic pectin extraction should use exclusively the aqueous medium according to the authors of [7]. This does not support a number of the results of our own laboratory tests, presented below.

Temperature control is key to the extraction process, as shown in [15, 16]. However, the described parameters have no actual confirmation in the practice of industrial production of pectin. The authors of studies [16, 17] emphasize the importance of precipitation of the pectin extract. Although this statement can be considered from the side of obtaining liquid pectin concentrates, for which this process is not necessary.

In [8], the issue of alcohol precipitation of pectin is considered, emphasizing inaccuracies in determining the properties of the target product. For example, for the therapeutic and prophylactic use of pectin, let's believe that a more convenient form is a liquid concentrate enriched with natural whey proteins.

Thus, the results of the literature review allow to conclude that the extraction of pectin from plant raw materials remains a promising problem. The use of whey as an extractant is a poorly studied aspect of pectin technologies. This can equally be attributed to the practice of using membrane technologies for the treatment of pectin solutions.

5. Methods of research

Under laboratory conditions, the pomace was extracted with whey. The acidity of the extraction medium was determined by adding citric and hydrochloric acids. Extractions were carried out in a flask with a capacity of 3 dm^3 with periodic stirring and variable temperature and duration of the process. The resulting extract was filtered through a cotton filter and immediately processed on a membrane unit for concentration and purification. Empty fiber membrane elements AR-2 manufactured by «Vladipor» (Russia) with PIF membranes (polyamide ultrafiltration fibers) was used. The pore diameter of the membranes – 350 °A, the molecular weight of the retention – 10000 kDa, the material of the membranes – polyamide. The membranes were included in the laboratory stand shown in Fig. 1. During processing, the concentration factor of the hydrolyzate was determined by the formula:

$$\Phi K = \frac{V_o}{V_k}, \quad (1)$$

where V_o, V_k – initial and final volumes of the hydrolyzate during ultrafiltration, respectively.

The selectivity of membranes for protein and pectin was determined by the formula:

$$R = \frac{C_c - C_f}{C_c} \cdot 100\ \%, \quad (2)$$

where C_c, C_f – concentration of components in the concentrate and filtrate, respectively, %.

The working pressure of the process (P, MPa) was determined by the formula:

$$P = \frac{P_{in} + P_{out}}{2}, \quad (3)$$

where P_{in}, P_{out} – pressure at the inlet and outlet of the module, respectively, MPa.

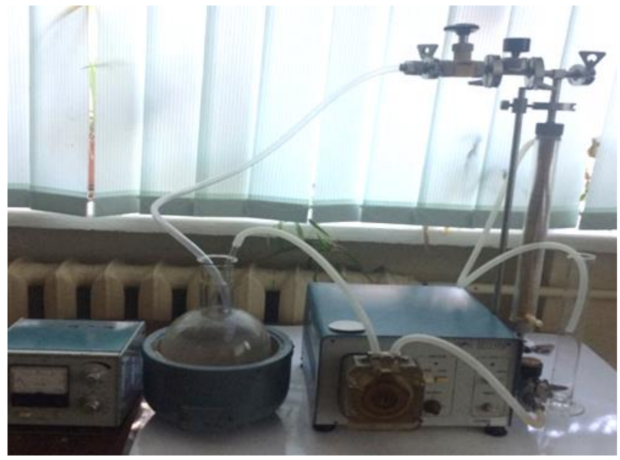

Fig. 1. Laboratory membrane unit for ultrafiltration UPL-0.6 (Russia)

Diafiltration was carried out in a continuous mode. Pasteurized whey served as a solvent. The concentration of pectin substances was determined according to GOST 29059-91. In pectin-whey concentrate, the ability of pectin to bind lead was determined by the method [4]. The complex of traditional and modern chemical, physicochemical, research methods, given in [18], was used to determine the chemical composition of the investigated products.

6. Research results

The chemical composition of the objects of research is given in Tables 1, 2.

The highest content of pectin substances was found in pomace of citrus fruits (2.7–3.1 %), quince (2.31–2.85 %) and apples (2.1–2.4 %).

Extraction-hydrolysis for 2 hours at a temperature of 85 °C, pH = 2–2.5 units with constant stirring gave the maximum yield of pectin substances when using apple waste. The main amount of pectin substances passed into the extract, starting from 75 min to 90 min, the subsequent increase in time did not affect the pectin yield. The best results were obtained when using hydrochloric acid in a concentration that provides pH = 2.2–2.5.

In the obtained plant extracts, the most mass fraction of pectin substances was observed for apple waste – 0.45 %, for citrus pomace – 0.27–0.31 %, for quince pomace – 0.2–0.25 %. In all samples, the mass fraction of proteins was at the level of the original whey and amounted to 0.85 % for further research, thus, let's choose the extraction of apple pomace.

The use of whey as an extractant of pectin substances made it possible to obtain a pectin extract enriched with whey proteins. The best results were obtained when using hydrochloric acid as a regulator of the acidity of the medium (Fig. 2).

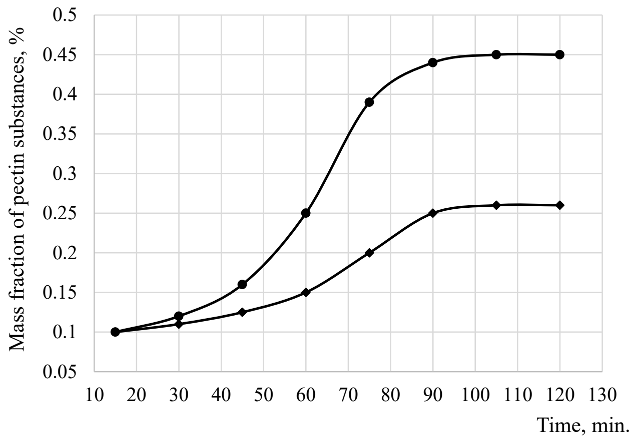

Fig. 2. Changes in the mass fraction of pectin substances in the extraction process: ♦ – citric acid; ● – hydrochloric acid

The obtained apple pectin-whey extract had the following chemical composition: mass fraction of dry substances – 7.12 %, including: mass fraction of pectin – 0.45 %, mass fraction of proteins – 0.85 %, mass fraction of lactose – 4.7 %. Active acidity=2.2–2.5 pH units.

To increase the concentration of high-molecular compounds in the obtained extract, in particular, proteins and pectin substances, it was subjected to ultrafiltration treatment. The membranes were highly selective for protein and pectin (99.0 % and 99.4 %, respectively). In the process of ultrafiltration, the specific productivity of membranes was investigated at various values of the concentration factor (Fig. 3).

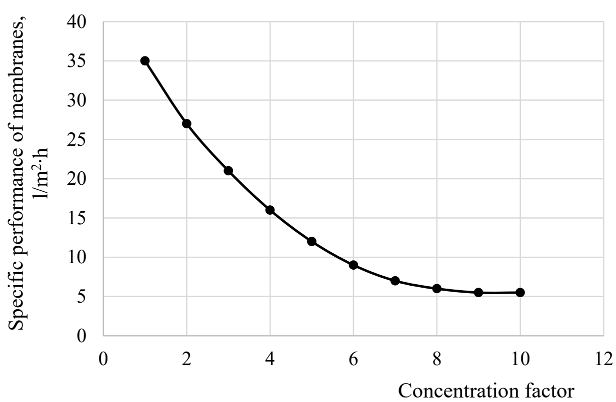

Fig. 3. Changes in the specific performance of membranes during ultrafiltration

The data obtained indicate that the specific productivity varies depending on the concentration factor within 35–8 l/m^2·h. This phenomenon can be explained by the effects of concentration polarization of membranes. The concentration of dry matter in the concentrate under such conditions was 16.6–17.3 % (Fig. 4).

Ultrafiltration of apple pectin-whey extract (concentration factor 7) yielded pectin-whey concentrate with a mass fraction of dry matter – 16.65 %, including: mass fraction of pectin – 3.15 %, mass fraction of proteins – 5.95 %, mass fraction of lactose – 4.73 %. The active acidity of the obtained ultrafiltration concentrate was 2–2.2 pH units.

To remove the remainder of hydrochloric acid, let's use diafiltration purification of pectin-whey concentrate using pasteurized curd whey with an acidity of 6.7 pH units. During diafiltration, the whey consumption was twice the amount of the extract.

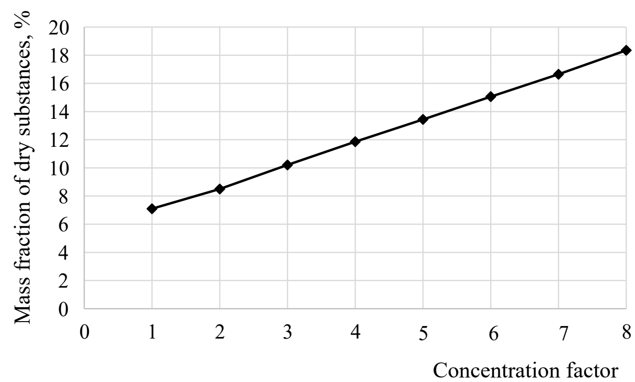

Fig. 4. Changes in the mass fraction of dry substances in ultraconcentrate during ultrafiltration

The mass fraction of pectin substances in the concentrate after purification was 3.13 % and had limitations caused by a sharp decrease in the productivity of the process, the mass fraction of proteins was 6.71 %, and the mass fraction of lactose was 4.72 %. The active acidity of the obtained diafiltration concentrate (purified pectin-whey concentrate) was 4.5 pH units. That is, diafiltration as a purification method makes it possible to effectively purify whey-pectin concentrates from ballast impurities with simultaneous enrichment of the concentrate with high-molecular whey components.

The resulting product had harmonious organoleptic properties and a high ability to sorb toxic compounds, in particular, lead (Table 3).

Table 3

Complexing ability of pectin

Complexing ability of pectin, mg Pb/mg pectin at pH:			
pH 2	pH 4.5	pH 7	pH 10
0.0020	0.01	0.012	0.014

For therapeutic and prophylactic purposes, in accordance with [2], it is necessary to eat 2 g of pectin. Taking into account these recommendations, such an amount can be provided when using 50 ml of the resulting concentrate. In this case, 16 mg of lead can be bound into an insoluble complex.

A technological scheme for the production of pectin-whey concentrate has been developed (Fig. 5).

Apple pomace before hydrolysis-extraction of pectin substances is washed three times with water at a temperature of 30–35 °C. Hydrolysis-extraction is carried out with whey with the addition of hydrochloric acid to pH 2.0–2.5 at a temperature of the hydrolysis mixture of 85 °C, the ratio of raw materials and extractant 1:20 for 2.0 h in a batch extractor. After the expiration of the process time, the A-extract is separated on the presses. The pressed apple pomace is again loaded into the extractor, filled with water at a temperature of 85 °C in a ratio of 1:20. Re-extraction of pectin substances is carried out for 1.5–2 hours. B-extract is separated by pressing, unloading the suspension into a press. The moisture content

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of the pressed pomace should be no more than 80 %. A- and B-extracts are mixed and defended for 2–4 hours to settle most of the mechanical impurities.

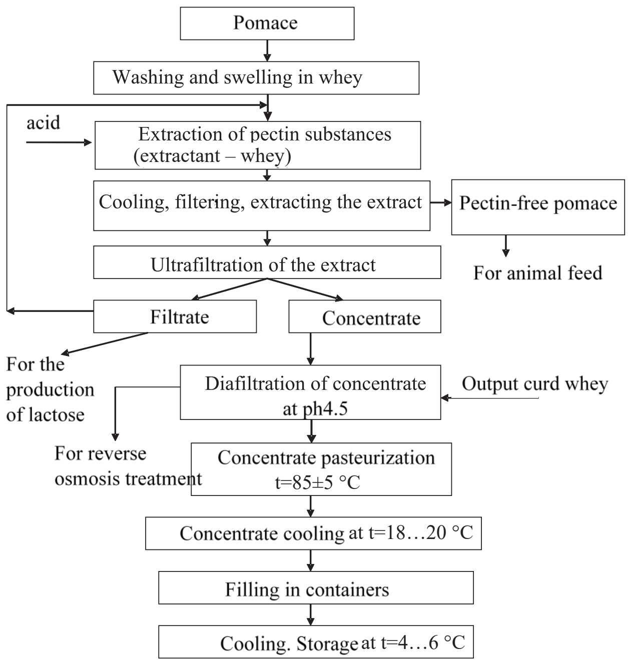

Fig. 5. Technological scheme for the production of pectin-whey concentrate

The average content of dry substances in the extract is 7.0–7.2 %, including pectin substances 0.2–0.45 %. The settled extract is separated and filtered on a kieselguhr filter. Concentration of the extract is carried out in an ultrafiltration unit. Process mode: pressure 0.15–1.0 MPa, temperature 40 °C, concentration factor 7. To accumulate the filtrate coming from the ultrafiltration unit, installed reservoirs. When curd whey passes through the ultrafiltration unit, its concentration increases due to the filtrate passing through semi-permeable membranes. The ultrafiltrate is discharged into a tank for intermediate storage, from where it is pumped for further use, or for treatment with reverse osmosis. When the target dry matter content is reached, the concentrate is discharged into a jacketed tank. In order to purify the concentrate from ballast substances, it is subjected to diafiltration. To do this, fresh curd whey is introduced into the tank with the concentrate in a ratio of 1:2 (whey concentrate) and the mixture is sent back to the ultrafiltration unit. The ultrafiltrate is reserved and sent for processing to the reverse osmosis unit (the resulting water is used for technological needs, and the reverse osmosis concentrate is used for the production of lactose, drinks, jellies, puddings, etc.). After diafiltration, the concentrate is reserved in an intermediate tank. From the intermediate tank, the ultraconcentrate is pumped into an automated tubular pasteurization-cooling unit for pasteurization and cooling. Pasteurization allows to increase the shelf life of the finished product due to the destruction of pathogenic microflora and inactivation of enzymes, hormones, toxins, etc. Pasteurization is carried out at a temperature of 85±5 °C without aging. The pasteurized concentrate is cooled to a temperature of 18–20 °C and sent for filling. Store the product at a temperature of 4–6 °C.

7. SWOT analysis of research results

Strengths. The strengths include: consumer interest in a new product, its therapeutic and prophylactic focus, high nutritional and biological value, natural ingredients and improved organoleptic characteristics. In addition, the product exhibits the ability to bind lead ions. The problem of recycling waste from both the dairy and canning industries is indirectly solved. The main care of milk processing – whey has the highest BOD (biochemical oxygen demand) and COD (chemical oxygen demand), which does not allow it to be discharged directly into the sewer network.

Among the strengths of this research are the technological ease of use of raw materials, good storage of raw materials, ease of transportation and processing of raw materials. Availability of raw materials on the market throughout the year. Affordable product price due to the use of raw materials that are production waste.

Weaknesses. Weaknesses include: poor consumer awareness of a new product, its benefits. In the experiments carried out, a limited range of raw materials was used, while similar properties have: different types of whey (from cottage cheese, casein), different types of plant processing waste. The use of these types of raw materials may affect the mechanism of therapeutic and prophylactic action through different degrees of esterification of pectin substances.

Additional research is needed on the mechanism of binding of heavy metals and radionuclides in the human body, which can also be caused by the properties of the structure of natural pectin. And also the specifics of the structure of natural pectin of various plants are not taken into account.

Opportunities. As for the capabilities of the new product, these are: the correct location of production, the development of information technologies, the presence of unsaturated market segments, there is almost no range of pectin-based food products for therapeutic and prophylactic nutrition, in particular, radioprotective ones.

Threats. Threats when a new product enters the consumer market: the possibility of new products and substitute products, growing competitive pressure as a result of the emergence of new competitors, price competition, an unstable economic and political situation in the country, a decrease in the purchasing power of the population.

Based on the SWOT analysis, the following solutions were proposed:

– entering new markets or market segments. This is an active marketing role and flexible pricing policy;
– expanding the range of products through the use of waste of various natural raw materials;
– let's propose to solve a more affordable price in comparison with analogue products on the market by reducing the price of our own product by using waste from the dairy and canning industries as raw materials and using modern energy-saving technologies;
– let's propose to solve the weak information content of consumers about the new product by informing consumers about the properties of the new product;
– to reduce the influence of such factors as «the emergence of new competitors» and «growing competitive pressure», let's offer: an active role of marketing, flexible pricing policy, expansion of the assortment. With regard

to the factor of «decreasing purchasing power», it is planned to carry out activities to position the new product among potential consumers;
– product promotion by means of merchandising.

8. Conclusions

1. A detailed analysis of the process of extracting pectin substances during the recycling of wastes of plant raw materials and milk whey has been carried out. It was found that the parameters of temperature, duration and type of extractant have the main influence on the process. The maximum yield of pectin substances was observed at a temperature of 85 °C, pH=2–2.5 units, when using hydrochloric acid for 2 hours with constant stirring.

2. The process of membrane processing of pectin-whey hydrolyzate has been investigated. With concentration factors from 1 to 8, the process productivity decreases from 35 to 8 $l/m^2 \cdot h$. The concentration of dry substances in the concentrate increases to 16.6–17.3 %, in the filtrate remains at 5.6 % during the entire filtration. The quality indicators of the obtained pectin-whey concentrate were: mass fraction of pectin substances 3.13 %, mass fraction of proteins – 6.71 %, mass fraction of lactose – 4.72 %. The purified concentrate exhibited the ability to bind lead ions depending on pH at the level of 0.002–0.014 mg Pb in terms of pectin.

3. The qualitative characteristics of the obtained pectin-whey extracts have been determined. The mass fraction of pectin substances for apple waste was 0.45 %, for citrus pomace – 0.27–0.31 %, for quince pomace – 0.2–0.25 %. In all samples, the mass fraction of proteins was at the level of the original whey.

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Oksana Chabanova, *PhD, Associate Professor, Department of Technology of Dairy, Olive-Fat Products and Beauty Industry, Odessa National Academy of Food Technologies, Odessa, Ukraine, e-mail: oksana_chabanova17@ukr.net, ORCID: http://orcid.org/0000-0002-1455-2987*

Sergii Bondar, *PhD, Associate Professor, Department of Ecology and Environmental Technologies, Odessa National Academy of Food Technologies, Odessa, Ukraine, e-mail: sergej.nik.bondar@gmail.com, ORCID: https://orcid.org/0000-0002-7908-2074*

Yevhenii Kotliar, *PhD, Associate Professor, Department of Technology of Dairy, Olive-Fat Products and Beauty Industry, Odessa National Academy of Food Technologies, Odessa, Ukraine, e-mail: yevhenii11@ukr.net, ORCID: https://orcid.org/0000-0003-0263-3939*

Tatiana Nedobiychuk, *PhD, Associate Professor, Department of Commodity Science and Customs, Odessa National Academy of Food Technologies, Odessa, Ukraine, e-mail: nedobeychuk@ukr.net, ORCID: https://orcid.org/0000-0002-8030-0198*

Yakiv Verkhivker, *Doctor of Technical Sciences, Professor, Department of Commodity Science and Customs, Odessa National Academy of Food Technologies, Odessa, Ukraine, e-mail: yaverkhivker@gmail.com, ORCID: https://orcid.org/0000-0002-2563-4419*