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Modification of the Process of Obtaining Pectin by the Methods of Membrane Technology

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ABSTRACT

The proposed approach to the processing of plant materials using membrane methods is new in the field of developing new methods for isolating pectin substances and obtaining not only pectin itself, but also new low-cost high-quality pectin-containing products. The studies were carried out on pressings obtained after squeezing juice from citrus fruits (Georgia): lemon ("Meer"), Washington-Navel orange variety, "Unshiu" mandarin and the largest citrus pomelo fruit (China). From the fruits harvested in April-December, the juice was squeezed out and from the remaining mass, which was crushed, by adding HCl (1:10) pectin isolates were obtained, which were concentrated by ultrafiltration. Soluble pectin was precipitated from the concentrated extract with ethyl alcohol, i.e. converted to an insoluble form. The resulting precipitate was thoroughly washed with alcohol and then dried at T = 55°C. The concentration of isolates was carried out by tangential filtration in dynamic mode on a UPL-06 unit, an AR-0.2 ultrafiltration separating apparatus was used as a membrane, which was located vertically in the unit and was a ready-made module with a filtration area of 2 m^2; obtained on the basis of polyamide and phenylone – C 2-B hollow fibers VPU-15PA with a pore size of 500 A. Ultrafiltration was carried out in circulation mode to the maximum possible concentration of the extract. The dependence of the productivity of the process of purification-concentration of pectin extracts from citrus fruit waste without their morphological division, pressure, duration and filtration mode, type and term of fruit harvesting was studied. The work performed has shown that the use of semi-permeable membranes for concentrating pectin extracts allows: to a large extent to remove carbohydrates from the extract, to achieve partial discoloration of the pectin extract; ensure 100% retention of pectin substances by membranes; to achieve a high degree of concentration of the extract and made it possible to obtain a pectin preparation with a purity of 95%. The proposed technology using membrane technology makes it possible to obtain pectin with a purity of 95% or more by purifying it from ballast impurities at the stage of extract concentration.

Keywords: pectin, ultrafiltration, hollow fiber.

INTRODUCTION

Despite the variety of existing methods for obtaining pectins, each of which has its own disadvantages and advantages, many researchers continue to search for new energy-saving and environmentally friendly technologies that allow for the complex waste-free processing of plant materials with a high content of soluble and insoluble dietary fibers, providing maximum extraction of pectins and a high degree of their cleaning from ballast substances without the use of aggressive media – organic solvents and mineral acids. From these positions, membrane technologies are effective. The use of membranes makes it possible to create unified lines for the production of soluble and insoluble dietary fibers, as well as concentrates formed from production waste with a high content of physiologically active ingredients [Kagramanov et al. 2013].

To precipitate pectin from the solution, ethyl alcohol with a strength of 95% is introduced into the condensed pectin extract. The resulting pectin precipitate is washed several times with alcohol to remove ballast impurities and acid ions used in the hydrolysis process. After each washing, the precipitated pectin is separated from the alcohol, and after each washing, it is pressed and dried.

The alcohol diluted and contaminated during the precipitation and washing process is neutralized and returned to production.

The resulting pectin precipitate is dried to obtain pectin flakes. To reduce the cost of reagents for coagulation of pectin, it is advisable to concentrate the pectin extract. According to the traditional technology, the concentration of pectin extracts is carried out by evaporation in evaporators, which is associated with a significant heat consumption. In addition, due to such a thermal effect, a partial splitting of pectin occurs, which leads to a decrease in its molecular weight [Sedysheva et al. 2012].

During evaporation, neutral side chains are split off from the main backbone of the pectin molecule, respectively, the total number of carboxyl groups increases, which is confirmed by an increase in the uronide component as the extract is concentrated during evaporation. With prolonged heat treatment of pectin extracts, arabinan is split, and galactan remains unchanged; the hydrogen bonds are broken and then the bonds between arabinan, galacturonan and galactan are cleaved.

The high content of neutral side chains in the pectin molecule and, accordingly, the shielding of carboxyl groups by neutral sugars contributes to the twisting and formation of globular structures of its molecules. The viscosity of such solutions decreases, and the average molecular weight decreases accordingly. With an increase in the duration of the concentration process, the steam consumption increases and the productivity of the evaporators decreases [Tovar et al. 2019].

It has been established that the concentration of pectin extracts by vacuum evaporation for more than 30 minutes is not advisable, since this leads to a significant destruction of pectin.

In the technical aspect, the evaporation process requires high capital costs and high qualification of the attendants; also, during thermal evaporation, the color of pectin changes and its physical and chemical parameters noticeably worsen. An alternative to evaporation is membrane filtration.

TRADITIONAL PECTIN PRODUCTION TECHNOLOGY

The traditional or so-called "acid-alcohol" technology for obtaining pectin from vegetable raw materials consists of the following main stages: preparation of raw materials, hydrolysis of plant mass, coagulation of pectin with ethyl alcohol or metal salts, subsequent washing of the coagulate with alcohol, drying of pectin, its grinding, sieving through sieves, standardization and packaging.

To extract pectin from plant tissues, hot water, solutions of hydrochloric, sulfuric, nitric, sulfurous and oxalic acids, ammonium oxalate and citrate, polyphosphates are used. The properties of the extracted pectin depend not only on the extraction methods, but also on the condition of the raw material, which can be fresh, well dried or partially dried [Davitadze and Bejanidze 2023]. The main factors affecting the rate of the hydrolysis process are: the rate of swelling of the plant tissue and the penetration of acid into the cell, the concentration of acid in the extractant, the temperature of the process and its duration.

The nature of the interaction of these factors is complex. The same factor at different stages of the process can have different effects, and as a result, the rate and direction of hydrolysis can noticeably deviate from the required ones. For example, an increase in temperature accelerates many stages of the process (swelling and penetration of acid into the plant cell to decompose protopectin), but promotes degradation and depolymerization of the pectin molecule and thus significantly degrades the quality of the resulting pectin [Güzel and Akpınar 2019].

In pectin production, for a more complete extraction of pectin from a hydrolyzed pectin-containing mass, pressing, extraction in a system of large ratios of "solid body-extractant", a direct-countercurrent method and a combination of these processes and methods are used.

After centrifugation and filtration to remove solid particles from the extract, the extract is concentrated in a vacuum evaporation plant.

To precipitate pectin from the solution, ethyl alcohol with a strength of 95% is introduced into the condensed pectin extract. The resulting pectin precipitate is washed several times with alcohol to remove ballast impurities and acid ions used in the hydrolysis process. After each washing, the precipitated pectin is separated from the alcohol, and after each washing, it is pressed and dried. The alcohol diluted and contaminated during the precipitation and washing process is neutralized and returned to production.

This production technology requires the presence of an energy-intensive distillation department of alcohol for its regeneration and return to the production cycle along with the pectin production workshop.

The resulting pectin precipitate is dried on a drum dryer to obtain pectin flakes. The coagulate drum dryer is inefficient, complex, energy-intensive, and difficult to operate. Next, you need to grind the resulting pectin, standardize it and pack it.

Evaporation of pectin extract

To reduce the cost of reagents for coagulation of pectin, it is advisable to concentrate the pectin extract. According to the traditional technology, the concentration of pectin extracts is carried out by evaporation in evaporators, which is associated with a significant heat consumption. In addition, due to such a thermal effect, a partial splitting of pectin occurs, which leads to a decrease in its molecular weight.

During evaporation, neutral side chains are split off from the main backbone of the pectin molecule, respectively, the total number of carboxyl groups increases, which is confirmed by an increase in the uronide component as the extract is concentrated during evaporation [Rodsamran and Sothornvit 2019].

With prolonged heat treatment of pectin extracts, arabinan is split, and galactan remains unchanged; the hydrogen bonds are broken and then the bonds between arabinan, galacturonan and galactan are cleaved.

The high content of neutral side chains in the pectin molecule and, accordingly, the shielding of carboxyl groups by neutral sugars contributes to the twisting and formation of globular structures of its molecules. The viscosity of such solutions decreases, and the average molecular weight decreases accordingly. With an increase in the duration of the concentration process, the steam consumption increases and the productivity of the evaporators decreases [Khattab 2022].

It has been established that the concentration of pectin extracts by vacuum evaporation for more than 30 minutes is not advisable, since this leads to a significant destruction of pectin.

In the technical aspect, the evaporation process requires high capital costs and high qualification of the attendants; also, during thermal evaporation, the color of pectin changes and its physical and chemical parameters noticeably worsen. An alternative to evaporation is membrane filtration [Sebaoui et al. 2017; Senit et al. 2019; Bejanidze et al. 2021].

Membrane pectin production technology

The concentration of pectin extract is the most important stage of the technology, on which the consumer qualities of the product largely depend.

The traditional pectin production technology includes acid hydrolysis of pectin-containing raw materials, separation of the liquid phase, precipitation of pectin with ethyl alcohol or acetone, its purification and drying. This scheme is certainly environmentally hazardous, energy-intensive and requires the use of equipment made of corrosion-resistant structural materials and expensive chemical reagents and treatment facilities [Davitadze et al. 2023; Fidalgo et al. 2016].

Pectin concentrates obtained by concentration on selective membranes have a number of advantages: concentration occurs at low temperatures up to 50°C, i.e. without phase transition of the medium, which does not change the organoleptic properties of pectin and greatly simplifies the process equipment; membrane concentration simultaneously removes a significant part of low molecular weight ballast substances (mineral elements, salts, organic acids, pigments), which impair the presentation of the product and its consumer properties; the gelling properties of such pectin are better than when evaporating under vacuum; ample opportunities are being created for automating the process of membrane preconcentration [Bejanidze et al. 2021a; Bejanidze et al. 2019]

Alcohol coagulation of pectin

According to traditional technology, after the stage of preparation of hydrolysates, the dissolved pectin is transferred to a solid phase that precipitates by changing the composition of the solution. A number of compounds can be used for this purpose: ethyl alcohol or salts of polyvalent metals.

The alcohol method of pectin extraction is based on the property of pectin not to dissolve in ethyl alcohol at a concentration of the latter above 45%. Precipitated pectin is subjected to 4-fold washing with alcohol, while being cleaned from low molecular weight ballast substances, alcohol-non-precipitating polysaccharides with low molecular weight. [Alekseev et al. 2020, Bejanidze et al. 2021b].

During alcohol precipitation of pectin substances, part of the impurities dissolved in the extract is co-precipitated together with pectin. Repeated washing of the precipitate with 96% alcohol leads to the production of a pectin preparation with a relatively high degree of purity [Davitadze and Bejanidze 2012].

Obtaining pectin by this method has a number of disadvantages: pectin hydrolyzate is obtained in the form of a dilute solution with a pectin content of up to 0.3%, which is concentrated according to traditional technology on vacuum evaporators with prolonged heating, which leads to additional destruction of polymer chains; a large amount of ethanol is used as a precipitant (for the production of one part of pectin, up to 50 parts of alcohol are needed); An important disadvantage of this method is the need for an energy-intensive process of regeneration of alcohol by distillation [Ilyina et al, 2022; Kopylova and Svittsov 2018].

Numerous studies have established that practically all industrial precipitants used in the production of biologically active substances are suitable for the precipitation of pectin from the hydrolyzate. The method of precipitation of pectin with metal salts is an alternative process that allows obtaining preparations of high purity. However, the pectin jelly precipitated with copper sulfate had a metallic taste. The best quality indicators of pectin were obtained by precipitation with aluminum chloride. But at the same time, a smaller yield of pectin is observed. It has been established that the precipitation of pectin with ethyl alcohol at pH 1.7–1.9 makes it possible to achieve the highest yield [Davitadze et al. 2022].

The gel-forming and binding abilities of pectins are determined by its purity, i.e. the degree of purification from ballast impurities, including hard-to-remove low-molecular ones: monooligosaccharides, pigments, phenolic substances, mineral and organic salts. These impurities in traditional alcohol technology are removed at the stages of multiple alcohol washings of pectin coagulates, and when using membrane technology at the stage of extract concentration [Kebaili et al. 2018; Bejanidze et al. 2018]

The purpose of the work is to substantiate the feasibility of using membrane separation methods in pectin technology, as well as to establish the patterns of the processes of baromembrane processing of pectin-containing solutions.

MATERIALS AND METHODS

Objects of study

The studies were carried out on pressings obtained after squeezing juice from citrus fruits (Georgia): lemon ("Meer"), Washington-Navel orange variety, "Unshiu" mandarin and the largest citrus pomelo fruit (China). From the fruits harvested in April-December, the juice was squeezed out and from the remaining mass, which was crushed, by adding HCl (1:10) pectin isolates were obtained, which were concentrated by ultrafiltration. Soluble pectin was precipitated from the concentrated extract with ethyl alcohol, i.e. converted to an insoluble form. The resulting precipitate was thoroughly washed with alcohol and then dried at T = 55°C.

Research methods

Preparation and concentration of pectin isolates

In general, in the process of ultrafiltration, substances are divided into inorganic (mineral), which pass through the pores of the filter, and organic, which are concentrated in the concentrate, if the size of their molecule is larger than the size of the membrane pore (for example, large proteins, high molecular weight pectin). When passing pectin isolates, two products are obtained: a concentrate of macromolecular substances, in this case, a concentrated pectin isolate, and permeate – a concentrate of mineral substances.

The concentration of isolates was carried out by tangential filtration in dynamic mode on a UPL-06 unit (Fig. 1), an AR-0.2 ultrafiltration separating apparatus was used as a membrane, which was located vertically in the unit and was a ready-made module with a filtration area of 2 m^2; obtained on the basis of polyamide and phenylone – C 2-B hollow fibers VPU-15PA with a pore size of 500 A. Ultrafiltration was carried out in circulation mode to the maximum possible concentration of the extract.

RESULTS AND DISCUSSION

The dependence of the productivity of the process of purification-concentration of pectin extracts from citrus fruit waste without their morphological division, pressure, duration and filtration mode, type and term of fruit harvesting was studied.

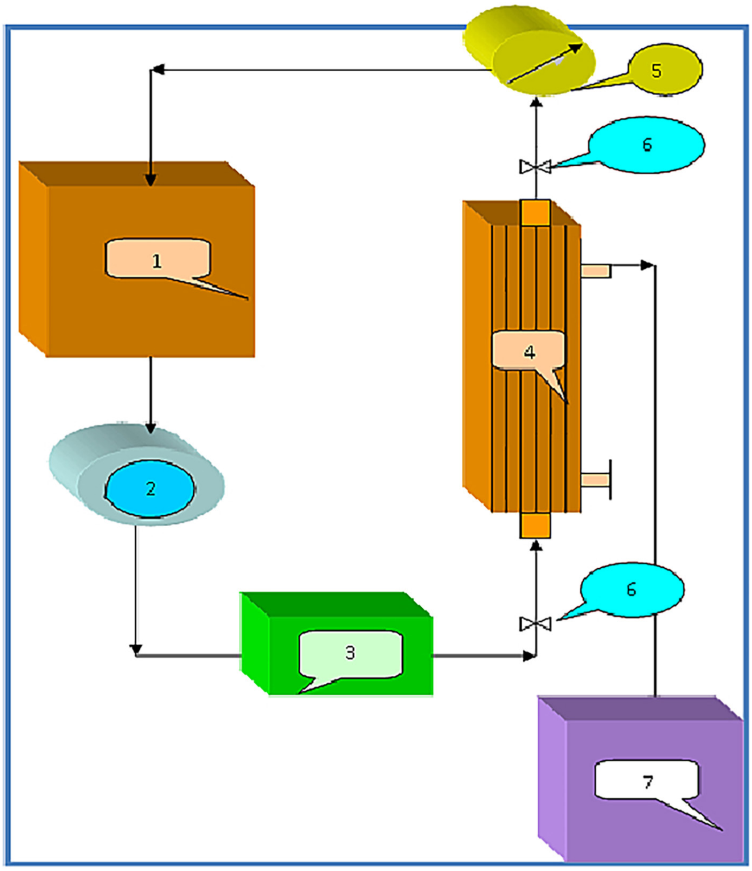

Fig. 1. Scheme of the ultrafiltration process: 1 – tank of the initial pectin isolate and concentrate collection; 2 – pump; 3 – mechanical filter; 4 – membrane module; 5 – manometer; 6 – valve for pressure regulation; 7 – container for collecting permeate/

Data on ultrafiltration of pectin extracts of citrus fruits are presented in Fig. 2–5. Extraction was carried out without fruits, i.e. from the peel of the whole fruit, from which the juice is squeezed.

Ultrafiltration is exactly the process that gives a sufficiently high product yield, the desired pectin fraction by molar mass and sorption properties.

It has been established that the ultrafiltration process is almost the same: the productivity of the process of concentrating pectin isolates in time, first, in the first minutes of filtration, drops sharply in time up to a certain point and then, monotonously decreases until equilibrium is established. The moment of establishment of equilibrium depends on the type of fruit and the timing of their collection. As the fruits ripen, the amount of pectin in the peel increases and, accordingly, in the isolate obtained from the peel, the viscosity of the isolate increases, as a result of which the concentration of the isolate proceeds at a slower rate and for a longer time – in practice, the concentration time is doubled, and the productivity process – is reduced by 30–50%. Pectin substances. are characterized by the lowest solubility in water, that is, the highest hydrophobicity, so they will have surface activity and will mainly be adsorbed in the pores and on the surface of the ultrafiltration membrane. Taking into account that hollow fibers, as an adsorbent, have rather thin pores, it can be concluded that in the places where pores are narrowed, the adsorption potential, due to the additivity of disperse forces, will be the highest, which will lead to the preferential sorption of these substances in the pores of hollow fibers. It is this fact that explains the sharp drop in the productivity of the process on hollow fibers in the first minutes of filtration. Over time, the mechanism of adsorption in the pores of the ultrafiltration membrane will predominantly be replaced by the mechanism of sediment formation on the membrane surface. Apparently, the initial thickness of such a deposit is limited by the radius of action of the adsorption centers of the membrane. Further thickening of the sediment layer occurs mainly due to the processes of aggregation, structure formation and polymerization of pectin molecules.

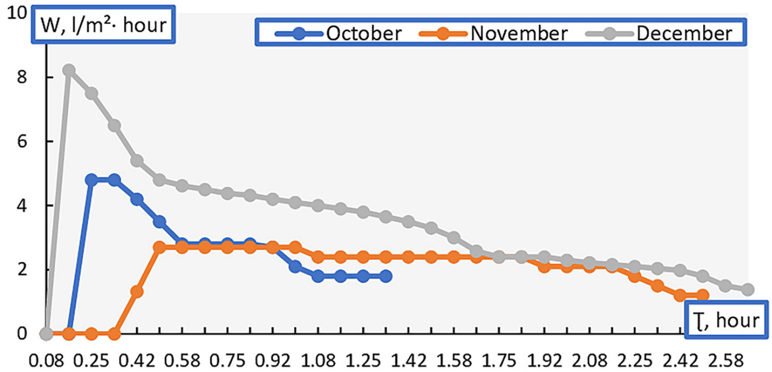

Fig. 2. Dependence of the productivity of the ultrafiltration process of concentrating the extract (extractant – HCl) of tangerine pectin on the time of ultrafiltration

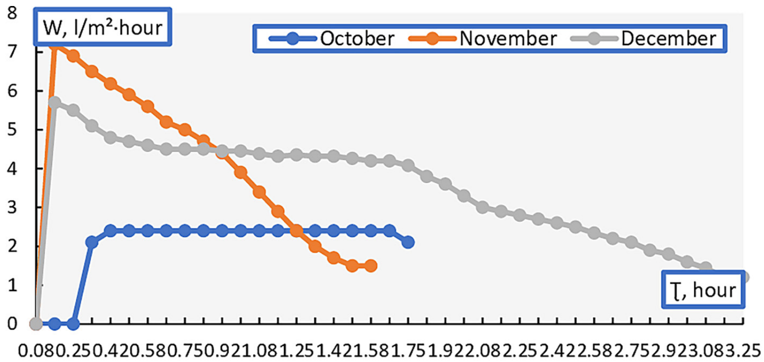

Fig. 3. Dependence of the productivity of the ultrafiltration process of concentrating the extract (extractant-HCl) of lemon pectin on the time of ultrafiltration

When comparing data on ultrafiltration of mandarin pectin extracts (Fig. 2), it was found that the productivity of the ultrafiltration process is higher for extracts obtained from fruits harvested in December and is almost the same for fruits harvested in November-October. This is probably due to the fact that in December almost all fruit pectin is in a soluble form and is better filtered.

As for pectin extracts from lemon (Fig. 3), here the productivity of the process clearly depends on the time of harvesting the fruits, and in order of its increase (the beginning of filtration), the following series was obtained: October > December > November, and (end of filtration) November > October > December. In this case, membrane equilibrium is practically not achieved for fruits harvested in November-December.

Figure 4 shows the data obtained by filtering orange pectin extracts. From which it follows that pectin extracts from fruits collected in December

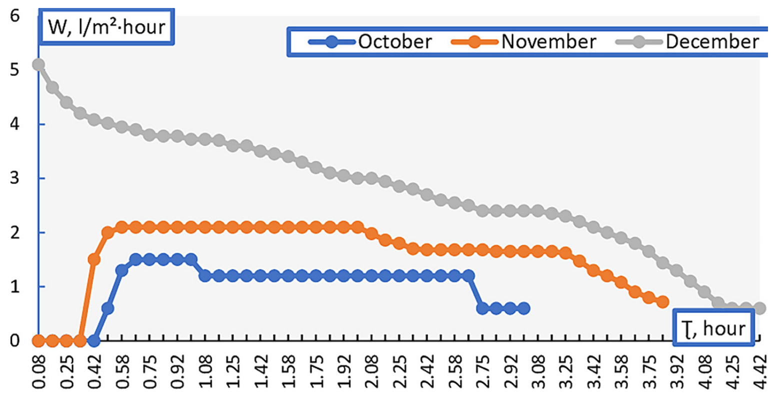

Fig. 4. Dependence of the productivity of the ultrafiltration process of concentrating the extract (extractant-HCl) of orange pectin on the time of ultrafiltration

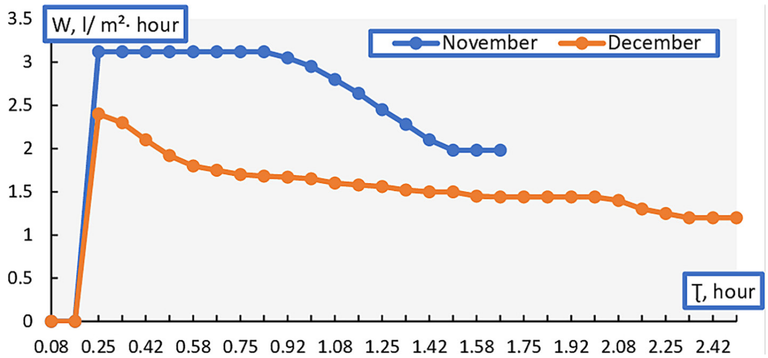

Fig. 5. Dependence of the productivity of the ultrafiltration process of concentrating the extract (extractant-HCl) of pomelo pectin on the time of ultrafiltration

are filtered faster, 2 times slower – in November, and 3 times – in October. Membrane equilibrium is achieved only in the case of filtration of fruits harvested in October-November.

As for the pomelo (Fig. 5), pectin extracts from this citrus are best filtered if the fruits are harvested in November. On Figures 6–9 comparative data on ultrafiltration of pectin extracts of citrus fruits are given

Established: in descending order of the performance of the process of ultrafiltration of pectin extracts, by months of fruit collection, the following data were obtained:

- in October (Fig. 6): tangerine – lemon – orange;
- in November (Fig. 7): lemon – pomelo – tangerine-orange;
- in December (Fig. 8): tangerine – lemon – orange – pomelo

Figure 9 shows comparative data on the degree (%) of concentration of citrus pectin extracts. It follows from the given data: the highest percentage of concentration was obtained for the pectin extract of the lemon collected in November (96.9%) and the lowest for the orange (85%):

- Lemon: November (96.88%) – October (93.19%) – December (87.5%);
- Orange: October (95.8%) – December (93.85%) – November (85%);
- Tangerine: December (95.46%) – November (95.32%) – October (92.78%);
- Pomelo: December (92.0%) – November (91.25%).

When comparing data (Fig. 9), one should:

- It is better to concentrate pectin extracts of orange either in October (95.8%) – in the process of their ripening, or in December (93.85%) – during the harvesting of fruits in the state of their technical maturity
- Pectin extracts of lemon should be concentrated in November (96.88%), and tangerine in December (95.32%) and November (95.46%) – concentration percentage is the same.
- Pomelo concentrates equally in November and December.

It follows from the data obtained that pectin can be obtained from a crushed fruit without dividing it into morphological parts, not necessarily

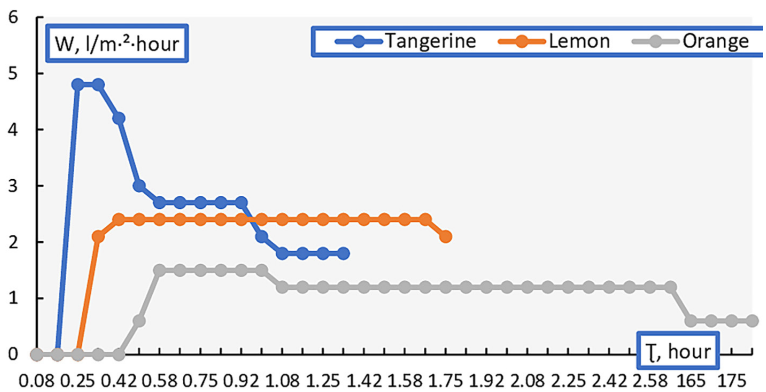

Fig. 6. Ultrafiltration of pectin extracts from citrus fruits harvested in October (2022)

during the ripening and harvesting of fruits, but also during storage, that is, pectin can be obtained throughout the year until the next harvest.

The effect of pressure on the process of concentrating pectin extracts was studied. It has been established that an increase in pressure at the initial stage of filtration leads to an increase in the performance of the ultrafilter. At P = 0.18 MPa, the filter permeability is maximum, however, with a further increase in pressure, the microfiltration rate decreases, since at a pressure exceeding the optimum, the pores of the polymer membrane are deformed, that is, the filter surface is deformed, which leads to an increase in the mass of sediment on the membrane, as a result, the membrane resistance, that is, the filtration resistance, increases significantly. The sediment mass will grow until the forces that press the sediment particles against the membrane are balanced by the forces due to the tangential flow of liquid over the filter surface.

In the case of concentrating the pectin extract by the method of membrane filtration, the question arises of restoring the functionality of the membranes after the completion of the working cycle. Layer of deposits of pectin, proteins, sugars and other components of the pectin extract is deposited on the working surface of the membrane, the performance of the ultrafiltration process decreases and balance is established

In addition, in the process of concentrating the extract, an increase in the number of microorganisms present in it occurs.

It should be noted that the stage of membrane regeneration is a crucial and necessary moment in the development of the process of membrane preconcentration, which requires careful study. Recovery of membrane devices is usually a multistage process that requires the sequential application of several detergents.

We conducted a reasonable search for chemicals and flushing modes to restore the membrane performance after the working cycle of extract concentration, taking into account membrane regeneration, and chose the NaClO option that fully meets the requirements of the main technological process.

The proposed technology using membrane technology makes it possible to obtain pectin with a purity of 95% or more due to its purification from ballast impurities at the stage of extract concentration.

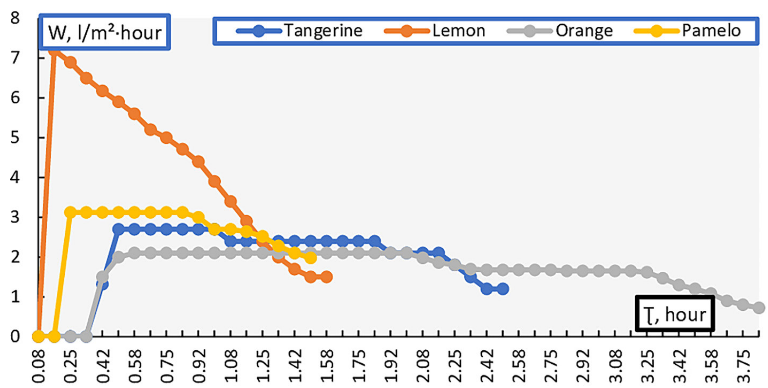

Fig. 7. Ultrafiltration of pectin extracts from citrus fruits harvested in November (2022)

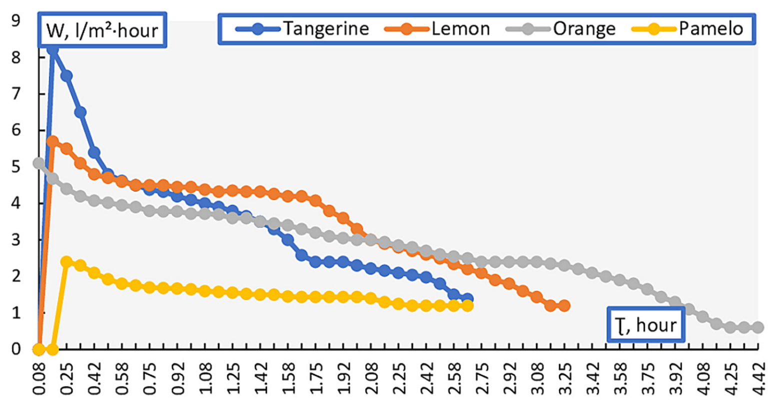

Fig. 8. Ultrafiltration of pectin extracts from citrus fruits harvested in December (2022)

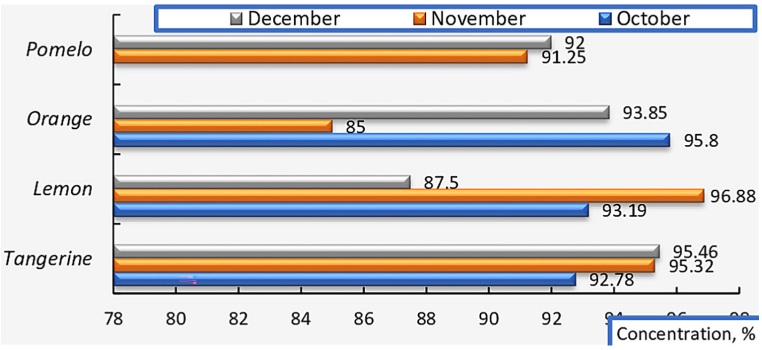

Fig. 9. Percentage of concentration of pectin isolates by ultrafiltration

CONCLUSIONS

The practical value of the work lies in the improvement of the technological scheme for processing citrus fruit waste (lemon (“Meer”), Washington-Navel orange variety, “Unshiu” mandarin and the largest citrus pomelo fruit (China)) into a liquid pectin concentrate: the optimal pore size of the membrane was chosen for ultrafiltration of pectin-containing solutions, a rational method for pre-treatment of the extract before its ultrafiltration was determined, and the optimal technological parameters of the process were established. It has been established that the productivity and quality of the process of concentrating pectin isolates depend on the type of fruit and the timing of harvesting. Concentration by ultrafiltration on hollow fibers with a pore size of 0.05 μm should be carried out at a pressure of P = 0.18 MPa, in circulation mode. You can concentrate the isolate 10–12 times.

The pore size of ultrafiltration membranes is determined by the degree of fractionation of pectin substances by molecular weight, which must be achieved during the processing of a pectin-containing product. The experimentally determined dependence of the membrane permeability on the value of the working pressure showed that the polymer structure of the sex fiber is easily deformable. The critical value, after exceeding which there is a deviation of the permeability of the real membrane from the ideal model, is the pressure P = 0.18 MPa.

It is shown that in the process of ultrafiltration of pectin extract, significant changes in the permeability and selectivity of the membrane are observed due to the formation of sediment on the surface and in the pores of the filter. The rate of sediment formation depends on the working pressure, tangential flow velocity, temperature of the extract and the concentration of suspended solids in it.

It is shown that at the stage of extract concentration, using membrane technology, ballast impurities are removed, including hard-to-remove low molecular weight mono-oligosaccharides, pigments, phenolic substances, mineral and organic salts. These impurities in the traditional alcohol technology are removed at the stages of multiple alcohol washes of pectin coagulates. The lower the content of these impurities, the purer the pectin and the higher its gelling and binding abilities.

The work performed has shown that the use of semi-permeable membranes for concentrating pectin extracts allows: to a large extent to remove carbohydrates from the extract, to achieve partial discoloration of the pectin extract; ensure 100% retention of pectin substances by membranes; to achieve a high degree of concentration of the extract and made it possible to obtain a pectin preparation with a purity of 95%.

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