

Development of Technology for the Extraction of Natural Pectin from Juice Production Waste

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ABSTRACT

In the article, the questions of pectin extraction from citrus fruits are discussed. The research was carried out on the extracts obtained after squeezing the juice from citrus fruits: lemon (Georgian and Meer), Washington-Navel orange (Georgia and Turkey), Unshiu mandarin and the largest citrus fruit pomelo (China). Fruits collected in April-December were morphologically divided into flavedo, albedo, and tissue of fruit lobes, from which pectin isolates were obtained. The dependence of the production of isolates on the ratio of components of the hydromodule (acid: water), the type of acid (HCl, HNO₃, H₂SO₄, H₂C₂O₄ and C₆H₈O₇), the duration of the process (1, 2, 3, 4, 5 and 24 hours) and the extraction temperature (20°, 60°, 80°C), the type and time of fruit ripening, as well as the type of precipitation reagent pectin (AlCl₃, CaCl₂, 95% C₂H₅OH, isopropanol) and its concentration, duration of extraction (2h, 8h, 12h, 24h) and temperature (20, 40, 60, 70, 80°C). A technological scheme for obtaining pectin extracts was developed. Established: extraction of pectin depends on the type and time of fruit collection, temperature and duration of extraction, type of extractant; the ratio of water and acid in the hydromodule (H₂O : acid) should be 1:10; isolate should be extracted with HCl, H₂SO₄ or lemon acid; pH of the hydromodule of the isolate should be 1.8–2.0; Extraction of pectin should be carried out with 95% C₂H₅OH, during 24 hours, with a module of 1:3 at room temperature. Identification of pectin isolates and obtained samples was carried out by the method of high-efficiency liquid chromatography. Obtained: practically all samples contain pectin and galacturonic acid and do not contain polygalacturonic acid, which indicates the complete extraction of pectin.

Keywords: pectin, sorption, sorbent, waste.

INTRODUCTION

One of the most important areas for improving the efficiency of modern production is the creation of low-waste and waste-free technologies, the wider involvement of secondary raw materials in the economic turnover.

Nowadays, residues are wrongfully disposed of and underutilized, becoming an increasingly alarming problem for the environment and the population's well-being. One of the primary sources of waste is the food industry. It is

estimated that about 1600 M.ton of food residues are produced annually, and about 500 M.ton are entirely derived from fruits [Durán-Aranguren et al., 2022]. The consumption of natural fruit juices has been increasing recently, mainly due to health concerns in the population. A shift toward a healthier and more natural lifestyle implies a reduction in the intake of soft drinks that could contain a high concentration of sugars, artificial colorants, and artificial sweeteners with possible adverse effects on the human body [Senit et al., 2019].

To the greatest extent, these requirements are met by the production of pectin and pectin products from secondary raw materials: beet pulp, peel, seeds and pulp residues of apple, grape and citrus pomace, which account for almost 50% of the total mass of the fruit [Zupanič, Mis NF, Pravst, 2019]. The level of use of secondary raw materials on average in the food industry is 10–15% of their total number.

Over the years, research has been made to develop ways to use organic waste as a source of chemical substances and energy. There are many studies regarding the obtention of multiple products from citrus peels [Gómez-Mejía et al., 2019; Ortiz-Sanchez et al., 2021; Calabrò, Panzera, 2018].

Nonetheless, pectin has been one of the main chemical substances retrieved from citrus residues with organoleptic characteristics that depend highly on the processing steps and conditions used for its production [Martín et al., 201; Conteratto et al., 2021; Conteratto et al., 2021].

The need for pectin significantly exceeds the volume of its purchases. So, taking into account the minimum preventive rate of pectin consumption – 2 g per day, in environmentally friendly areas, its amount with year-round consumption of pectin-containing products per 100 million people is over 70 thousand tons [Güzel M, Akpınar Ö. 2019; Ortiz-Sanchez et al., 2021; Köse, Bayraktar 2018].

In Georgia, in particular in Adjara, 80,000 tons of citrus fruits are processed annually, the amount of waste in this case is 6,000 – 8,000 tons, they are not processed and are dumped into rivers, the sea, remain on the ground, rot, and the environment is polluted, in particular soil and natural waters, which complicates the ecological situation in the region [Bejanidze et al, 2018,2019; Davitadze, Bejanidze. 2012].

Dietary fiber, including pectin, is a necessary nutritional component that ensures the functioning of the entire gastrointestinal tract. The consumption rate is up to 30–40 g per day (in dry form), while the actual content in the usual food is reduced several times. Therefore, it is advisable to additionally introduce them into our diet [Tsouko et al, 2020; Hilali et al, 2019; Cameron 2016].

An insufficient amount of pectin substances in food products leads to a decrease in the resistance of the human body to environmental influences. This has become especially noticeable in

recent years, when food safety is intertwined with the environment [Fidalgo et al., 2016; Patsalou et al., 2020;].

Pectin is one of the most common polysaccharides contained in sufficient quantities in vegetable raw materials – fruits, vegetables, root and tuber crops, apple and citrus pomace and other secondary resources. Despite this, a paradoxical situation has now arisen: pectin has not become cheap and affordable [Patsalou et al., 2020]. The cost of food pectin reached 25–35 US dollars per 1 kg, medical pectin – 60–120 US dollars (depending on purity) [Tovar et al., 2019; Olufunke et al., 2021; Jahidul et al., 2022].

The need to increase the range and production volumes of functional food has led to a significant expansion of the food hydrocolloids market. The world market for hydrocolloids is currently estimated by experts at about 3 billion US dollars. The production of pectin at the same time is only 10.91% of the total volume of hydrocolloids in the amount of 319 million dollars. However, in recent years, the demand for pectins has been increasing, the scope of which is steadily expanding with the advent of a new generation of functional foods. To date, there has been a steady increase in pectin consumption on average 3.0–3.5% per year. In addition, pectin is a soluble dietary fiber and is one of the nutraceuticals used in a healthy diet. In the past few years, terms such as “pharmaceutical pectin” and “biopectin” have been increasingly used. The multifaceted spectrum of the therapeutic action of pectin determines its use for the manufacture of medicinal preparations [Olufunke et al., 2021; Jahidul Hassan et al., 2022, Sebaoui O et al., 2018]

In 1790, the French chemist Louis Nicolas Vauquelin, who actively studied objects of plant origin, isolated a substance from fruit juice that was highly soluble in water and capable of gelation. After 40 years, the modern name of the isolated substance was born – pectin, translated from the Greek “pektos” – curdled, frozen, but the chemical structure of these compounds was clarified only in the second half of the 20th century. The reason is the difficulty of obtaining pure preparations of pectin substances in an unchanged state [Rodsamran, Sothornvit, 2019].

Pectins are present in the stems and leaves of plants, as well as in root vegetables and fruits [Kebaili et al.2018; Schiermeier Q. 2011; Guo et al., 2017; Ortiz-Sanchez et al., 2020].

For example, a rich source of pectin is lemon and orange peel (pectin content can reach 20–40% by weight of dry matter). Pectin is also found in apples (10–20% of dry matter weight), turnip, beet mass and other carbohydrate-storing plant organs. Blooming cotton contains ~5% pectin. As cotton matures, the amount of pectin decreases to ~0.8–1%. The content of pectin in fruits changes (decreases or, most often, increases) during fruit ripening [Hoz Vega et al., 2018; Casas-Orozco et al., 2015; Asra Hamidi, 2022].

Classification of pectin substances

Currently, in the chemistry of pectin substances, the names established in 1944 by the Committee for the Revision and Nomenclature of Pectin Substances of the American Chemical Society [Abboud et al., 2020; Mohamed and Fernanda, 2022] are accepted, according to which pectin substances are divided into the following types:

- protopectin – natural, water-insoluble, cross-linked pectin associated with many metals and other compounds;
- pectin is a water-soluble substance consisting of partially or completely methoxylated polygalacturonic acid;
- pectic acids – completely demethoxylated pectins with an intact chain;
- pectates salts of pectic acids;
- pectinates – salts of incompletely esterified pectin;
- pectin derivatives – pectins associated with various groups according to their main valences, for example, acetyl pectin.

The classification of pectin substances is shown in Fig. 1.

Thus, pectin substances exist in several forms. These forms perform various physiological functions in the plant tissue and, depending on the direction of the biochemical processes in the plant, can pass from one state to another.

Structure of pectin

Pectin substances are part of the cell wall of the middle lamellae, cytoplasm and in the intercellular space of plant cells. In plant tissues, pectin substances are present mainly in the form of protopectin, which is found mainly in the walls of the plant cell (sometimes in combination with hemicelluloses and cellulose), in the intercellular cementing material, playing the role of supporting elements of tissues. If straight (linear) cellulose fibers, like the steel frame of a building, constitute the main structural lattice of a plant cell, then fibrillar protopectins serve as the material of structural details. Cell sap contains pectins and pectinates. They play an important role in cell division and growth, in maintaining the water and salt balance of non-lignified tissues, and are involved in the processes of cell wall stretching. Along with the structural function, they play an important role in the metabolism of reserve substances.

Pectin substances are found in almost all parts of the plant: roots, stems, inflorescences. However, mainly pectin substances are found in fruits and vegetables. To date, there is no clear classification of pectin-containing raw materials. Some authors [Fidalgo et al., 2016; Patsalou et al., 2020] propose to divide all pectin-containing raw materials into three groups:

- The first group includes vegetables (root crops, leafy, stem, fruit, pumpkin, legumes);
- To the second – pome fruits, stone fruits, berries, tropical and subtropical fruits;
- The third group includes other types of raw materials: tea leaves, sunflower baskets, bark of coniferous trees.

This classification is convenient when sorting pectin-containing raw materials and choosing the parameters for pectin extraction, since the methods of preparing raw materials for hydrolysis and extraction processes inevitably differ due to the different tissue structures of these plant products.

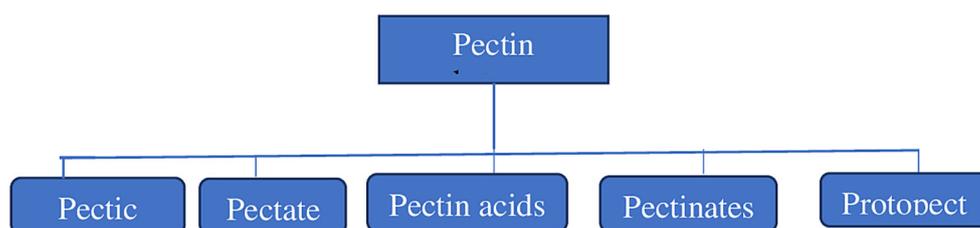


Fig. 1. Classification of pectin substances.

Currently, four types of classic pectins are produced in the world (from one type of raw material): apple, citrus, beet, sunflower baskets. Along with the classical ones, combined pectins are produced, which are isolated from mixed raw materials.

By chemical nature, pectins are high-molecular compounds of the cell wall matrix, localized in the primary cell wall of all higher plants and belonging to the group of heteropolysaccharides, which are based on derivatives of polygalacturonic (pectic) acid. Through the side chains, pectin is connected to cellulose fibers and a number of other heteropolysaccharides, which belong to hemicelluloses – colloidal polysaccharides or plant glucopolysaccharides. The content of this substance in the cell wall is maximum in the central layer, which connects the cells to each other. The morphological and physiological role of pectin in plants, as a structural element of the cell, is to regulate the water metabolism of plants and, as the main component of intermediate tissues, it provides adhesion and stability in tissues and cells [Akin-Ajani and Okunlola, 2021; Asra Hamidi (Ataran), 2022.].

According to modern concepts, pectin has a linear structure with a molecule length of about 10–4 μm . Pectin substances belong to the class of polyuronic acids, since pectin macromolecules – polygalacturonic (pectic) acid are built from units – residues of predominantly unbranched α -D-galacturonic acid (300–1000 units) form a filamentous molecule, have a pyranose configuration and are connected 1,4 – L – glycosidic bond and 2-O-substituted α -L-rhamnopyranose linked by α -1,4- and α -1,2-glycosidic bonds [Mohamed Bassim Atta and Fernanda Ruiz-Larrea. 2022] Rhamnose-knot of the bend of the pectin molecule (Fig. 2).

Higher plants (Fig. 3) in pectin, along with polygalacturonic acids, contain small amounts of residues of neutral monosaccharides L-arabinose, D-galactose, D-xylose and fructose, which are attached to pectin molecules in the form of side chains, giving pectin the properties of a heteropolysaccharide. The presence of rhamnose in the pectin molecule justifies its other more correct name – rhamnogalacturonan. Other neutral sugars – araban, galactan – form side chains that combine with cellulose molecules. Part of the carboxyl groups of polygalacturonic acid is esterified with methanol (Fig. 4). Hydroxyl groups at C_2 and C_3 can

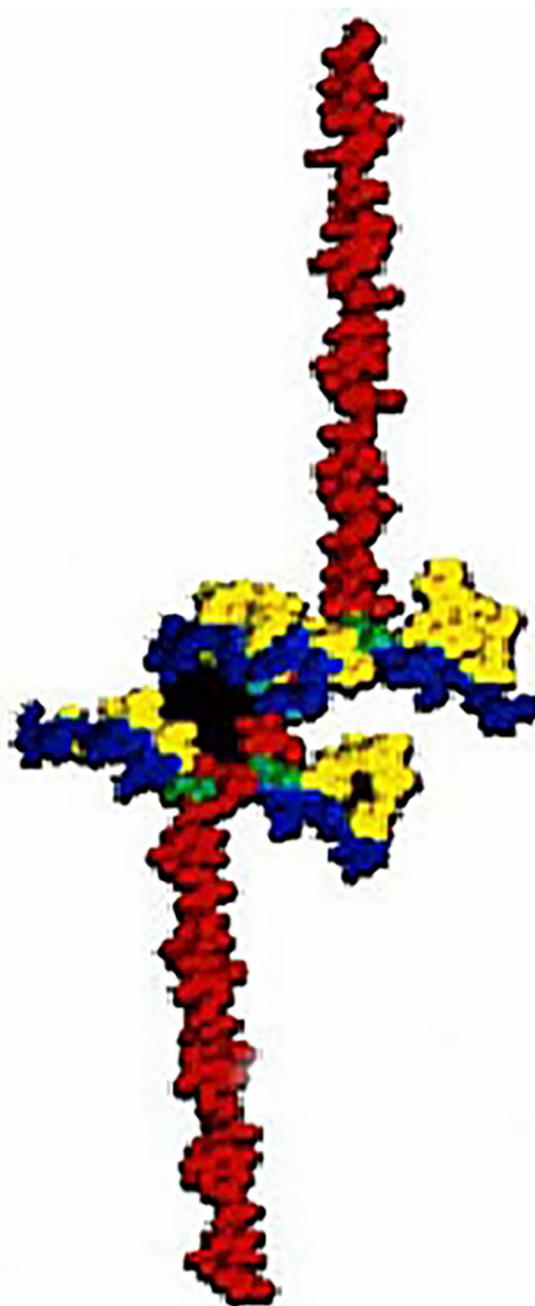


Fig. 2. Model of a pectin molecule with side chains of arabinogalactan-II

be acetylated. As a rule, the degree of acetylation of pectins is low (does not exceed a few percent) and depends on the source of pectin.

The composition of pectin powder depends on the feedstock, since various fruits, vegetables, root crops, medicinal plants contain only their inherent components. In some seaweeds, polyuronic acid has a different structure and is called alginic acid.

Pectin is considered to be pectin if it contains at least 65% galacturonic acid, which determines the “behavior” of pectin and its properties.

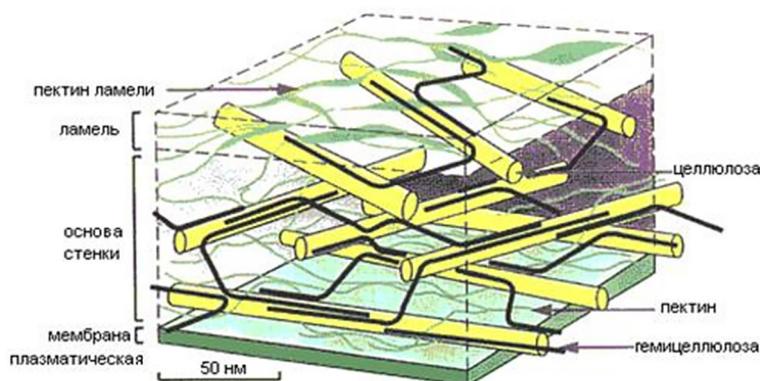


Fig. 3. The structure of the plant cell wall.

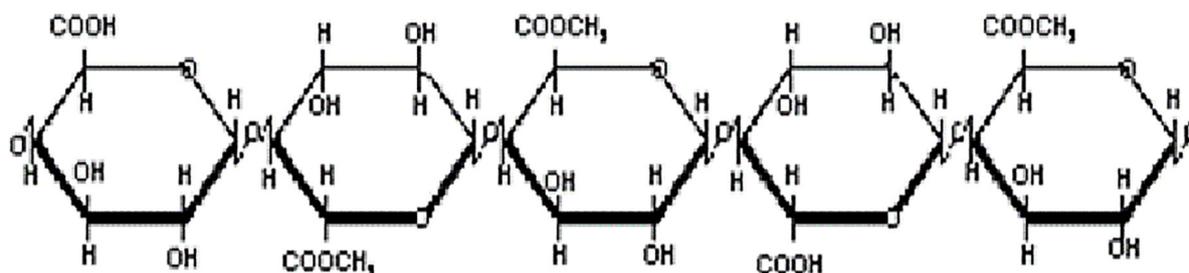


Fig. 4. Pectin molecule

Thanks to this structure, pectin behaves like a gluing material with an important supporting and hardening function, and thanks to its colloidal character and a pronounced swelling property, it binds and manages the water balance in plants.

The study of domestic and foreign literature showed that the composition and structure of pectin substances cannot be considered definitively established

Currently, scientists have come to the conclusion that pectin substances can be considered as a mixture of three polysaccharides: galacturonan, galactan and arabinan, which are extracted in different amounts during acid hydrolysis along with pectin.

The structure of pectin molecules isolated from plant objects has its own distinctive features, which include: the size of the molecule (molecular weight), its chemical composition (in particular, substituted acetylated hydrophilic groups), the degree of esterification of the polygalacturonic acid molecule, the nature of the distribution of carboxyl groups along the length polymer molecule.

The degree of esterification of pectin

The degree of esterification of pectin is determined by the ratio of esterified units of

galacturonic acid to their total number in the molecule. Depending on the degree of esterification, i.e. the number of methoxyl groups. A distinction is made between high-esterified pectins (the degree of esterification is more than 50%) and low-esterified pectins (the degree of esterification is less than 50%). The degree of esterification of high-esterified pectins determines the rate and temperature of gelation, which is reflected in the designation of pectins as fast and slow gelling [Abdelrahman Mosaad Khattab. 2022; Abboud, et al., 2020].

Pectins isolated from apple pomace and sunflower heads are high molecular weight, from beet pulp and citrus peels – low molecular weight. In molecules of pectins from beet pulp and sunflower heads, part of the hydroxyl groups at the second and third carbon atoms is replaced by acetyl groups. According to the degree of esterification (E), beet and sunflower pectins are classified as low esterified (E less than 50%), apple and citrus pectins are high esterified (E more than 50%).

In the properties of the latter, differences were found in the nature of the distribution of carboxyl groups in the molecule. In pectins from apple pomace, a uniform distribution of carboxyl groups is observed along the entire length of the pectin molecule, while in citrus fruits, carboxyl groups are distributed unevenly. This character

of the distribution of carboxyl groups in the citrus pectin molecule is explained by the action of the pectinesterase raw material enzyme, which partially deesterifies the pectin molecule by the block mechanism.

The molecular weight of pectin substances depends, first of all, on the nature and quality of the raw material source, the method of isolation and the method of its preparation for production. The average molecular weight of pectin substances is from 10,000 to 400,000, which corresponds to a degree of polymerization from 50 to 2000. Commercial pectin preparations have an average molecular weight from 30,000 to 120,000, depending on the type of preparation.

The nature of the connection of pectin substances in a plant cell is an objective criterion for their biochemical classification. Concerning the nature of connections of pectin substances in plants the hypotheses based on reactivity of functional groups of pectins are stated. These are, first of all, hydrogen bonds formed by hydroxyl and carboxyl groups. Stronger carboxyl-carboxyl interactions with the formation of bridges from polyvalent cations (usually Ca^{2+} and Mg^{2+}), ester bonds with fiber involving hydroxyl and carboxyl groups. There are also data on the relationship of pectin substances in the cell wall with proteins. To isolate pectin substances from the plant mass, it is necessary to break these bonds. But if the isolation conditions are harsh, then the destruction of the pectin molecule itself will occur.

From plants, three groups of pectin substances can be distinguished, differing from each other in the nature of the bond in the plant cell:

- pectin substances connected mechanically and through hydrogen bonds;
- carboxyl-bound pectin substances retained in the cell due to carboxyl-carboxyl interactions through the formation of dimeric groups and calcium bridges;
- protopectin. The nature of the bonds of this part of pectin substances has not been reliably established. The experimental data do not contradict the hypothesis of an ester bond between and other cell polymers.

Pectin substances isolated from vegetable raw materials, depending on the source of production and the degree of purification, are a powder from white to light brown.

Pectin properties

Pectin does not dissolve in alcohol and other organic solvents, but dissolves in 84% phosphoric acid and liquid ammonia; swells in glycerol and formamide.

The solubility of pectin depends on the degree of polymerization and esterification. Solubility in water increases with an increase in the degree of esterification and a decrease in the size of the molecule, i.e. the solubility of pectins in water decreases with increasing chain length and the number of carboxyl groups ($-\text{COOH}$). An increase in COOH – groups leads to an increase in molar energy (cohesion) between COOH groups. The value of cohesive energy for COOH – groups is 90000 cal/mol, while for COOCH_3 groups – 5600 cal/mol (Fig. 5). To obtain a homogeneous solution, it is better to pre-moisten pectin with ethanol or mix with sugar.

Aqueous, 1% pectin solution has $\text{pH} = 2.9\text{--}3.2$. As a typical polyelectrolyte, pectin has a relative dissociation constant of $0.1 - 10.0 \cdot 10^{-4}$, and the monomer galacturonic acid – $3.25 \cdot 10^{-4}$. Being a macromolecular compound, pectin gives viscous solutions, and the viscosity depends on the degree of esterification, pH and electrolyte concentration. Pectins, in addition to water, are soluble in formamide, dimethyl sulfoxide and hot glycerin, insoluble in most organic solvents. Therefore, pectin substances can be precipitated from aqueous solutions by adding water-miscible solvents: methanol, ethanol, acetone, as well as quaternary detergents, water-soluble bases, proteins, polyvalent cations.

Pectins are able to form colloidal solutions, characterized by high viscosity, which is explained, firstly, by the fact that the molecules of pectin substances, interacting with each other, form aggregates.

The second reason is the excessive hydration of pectin molecules, which determines their shape, and the high degree of dissociation of carboxyl groups. In aqueous solutions of pectin substances, the molecule has the form of a helix, the carboxyl groups of which are located in adjacent turns. At a high degree of dissociation of carboxyl groups, as a result of the interaction of like-charged particles, the helical conformation of molecules is broken, their linear dimensions increase, and the viscosity of the solution increases. Since the viscosity of pectin is determined by its high molecular nature, it decreases with a

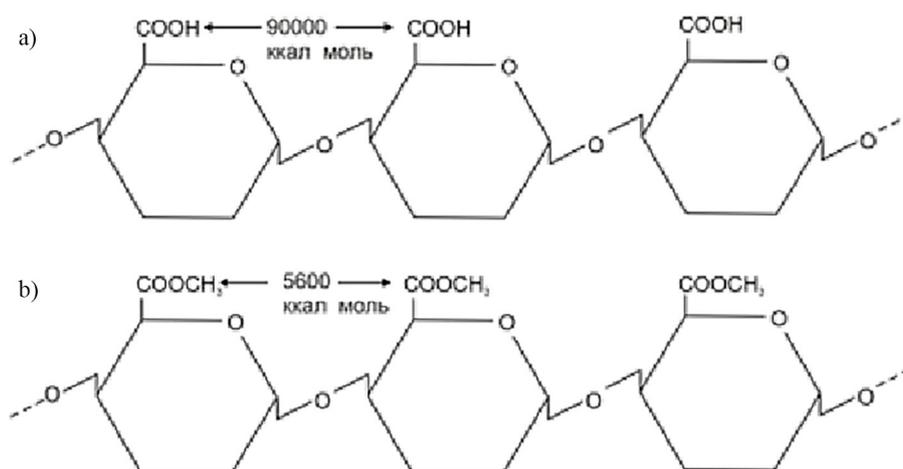


Fig. 5. Molar energy of functional groups of pectin: (a) carboxyl groups, (b) methoxy groups

decrease in the size of the molecules as a result of heating, when treated with acids, etc.

In colloidal aqueous solutions, pectin molecules are surrounded by a hydrated shell and carry a negative charge. A decrease in hydration or a decrease in charge, or both, causes precipitation (coagulation) of the pectin. The need for coagulation occurs most often in the pectin industry when pectin is isolated from the extract, and also in cases where pectin needs to be removed from the system (for example, in the wine industry). Pectins can be precipitated from aqueous solutions with electrolytes and organic solvents: ethanol, acetone [Zupanič et al., 2019].

Under the action of acids and alkalis, as well as enzymes, the process of saponification of methoxyl and acetyl groups occurs, which leads to a decrease in the degree of esterification.

The main method for obtaining pectin is hydrolysis, extraction of raw materials with an aqueous solution of strong acids (nitric, hydrochloric, sulfuric, phosphoric), followed by precipitation with ethyl alcohol. Also known is a method of obtaining pectin from dry apple pomace. Enzyme preparations (pectinesterase, pectinmethylesterase) are also used to obtain highly purified pectins. However, due to the high cost of enzymes, this method has not yet found wide industrial application and is used mainly for laboratory and medical purposes.

MATERIALS AND METHODS

Objects of study

The studies were carried out on pressings obtained after squeezing juice from citrus fruits: lemon (“Georgian” and “Meer”), Washington-Navel

orange variety (Georgia and Turkey), “Unshiu” mandarin and the largest citrus pomelo fruit (China). The fruits collected in April–December were morphologically divided into flavedo – the upper layer of the peel, albedo – the lower layer of the peel, partitions and tissues of the fruit lobules, from which pectin isolates were obtained, pectin was isolated by extraction, washed and dried. The dependence of obtaining isolates on the ratio of the components of the hydromodulus (acid : water), type of acid (HCl, HNO₃, H₂SO₄, H₂C₂O₄ and C₆H₈O₇), duration of the extraction process (1, 2, 3, 4, 5 and 24 hours) and temperature (20°, 60° and 80°C), the type and time of fruit ripening. The photo (Fig. 6a, b, c, d) shows the morphological composition of citrus fruits.

Research methods

Preparation of pectin isolates

The technological scheme for obtaining pectin developed by us is shown in Figure 7. To isolate pectin from citrus fruits, juice was squeezed, the squeezes were washed well with running water, crushed, and then pectin was extracted with a mixture of water and acid (fruit: mixture ratio was 1:5, mixture pH 1.6–1.8). Acid for the isolation of pectin isolate was obtained by electro dialysis.

To isolate pectin from citrus fruits, juice was squeezed, the squeezes were washed well with running water, crushed, and then pectin was extracted with a mixture of water and acid (fruit: mixture ratio was 1:5, mixture pH 1.6–1.8). The extraction was carried out for 2, 8 and 24 hours, then the resulting pectin isolate was concentrated before separating the pectin.



Fig. 6. The morphological composition of citrus fruits; (a) “Unshiu” mandarin; (b) lemon “Georgian” and “Meer”, (c) Pomelo; (d) orange “Washington-Navel”

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 $T = 55^{\circ}\text{C}$.

RESULTS AND DISCUSSION

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.

An important step in the complete isolation of pectin is the correct choice of the extractant and the establishment of the required extraction

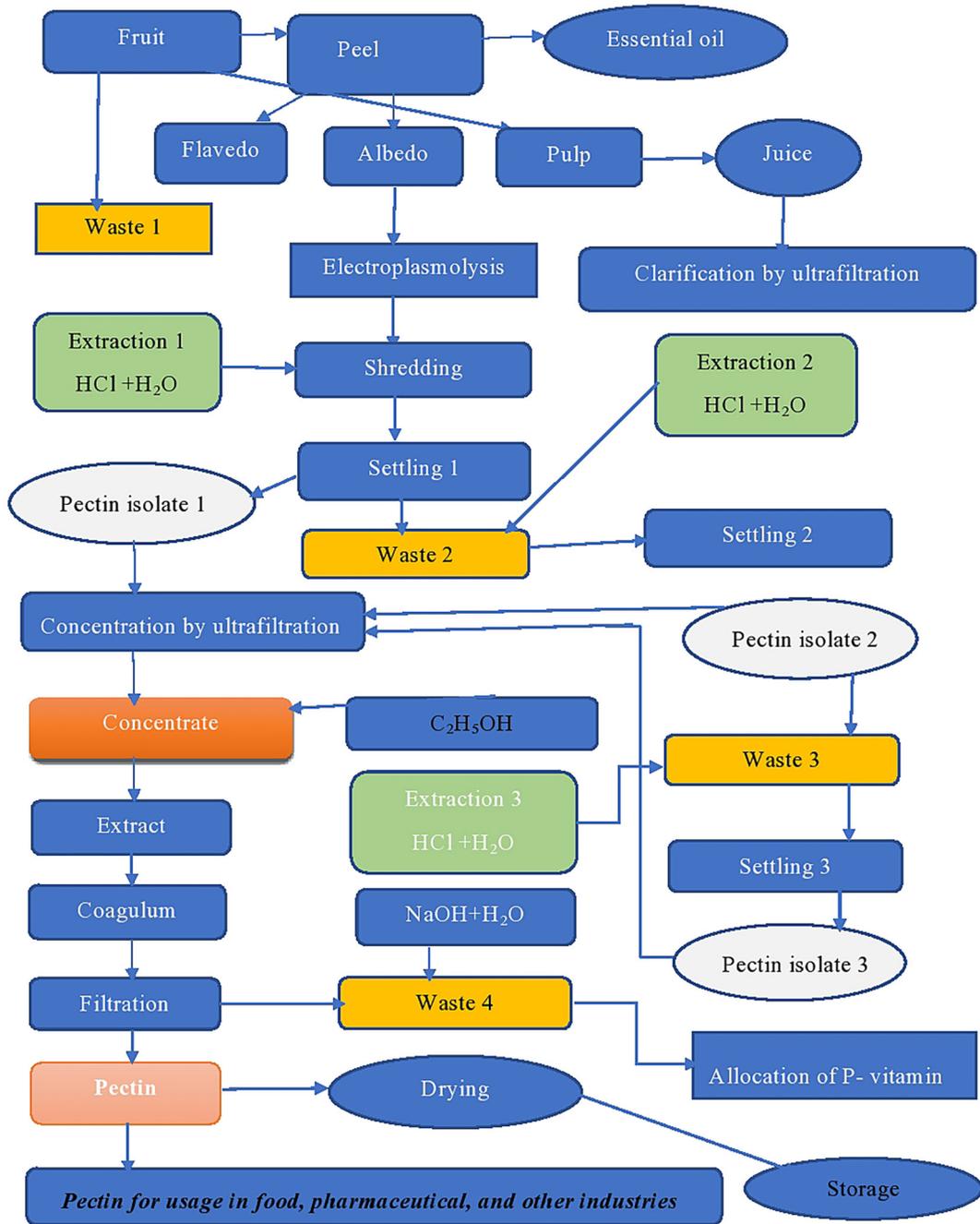


Fig. 7. The technological scheme for obtaining pectin

time. As follows from the data (Fig. 8) for lemon, with an increase in the extraction time, the amount of isolated pectin increases, and the most complete extraction of pectin occurs within 24 hours when using citric acid, 7.3 g of pectin is extracted, approximately the same amount of pectin is extracted nitric and hydrochloric acid (4.7 g) and worst of all – sulfuric (3.85 g) and oxalic (3.45 g) acids. When extracting for 1 hour, pectin is best extracted with sulfuric acid (5.46 g), 2 hours with nitric acid (4.73 g) or hydrochloric acid (4.63 g).

It has been established that the temperature of the process (Fig. 9) and the type of extractant (Fig. 10) affect the completeness of pectin extraction: with an increase in temperature and extraction time, hydrochloric acid is a more effective extractant for extracting pectin than citric acid.

The amount of pectin released depends on the fruit ripening time (Fig. 11). It was found that, in fruits harvested in May, there is less pectin than in April. This is explained by the fact that with the maturation of the fetus, the amount of insoluble

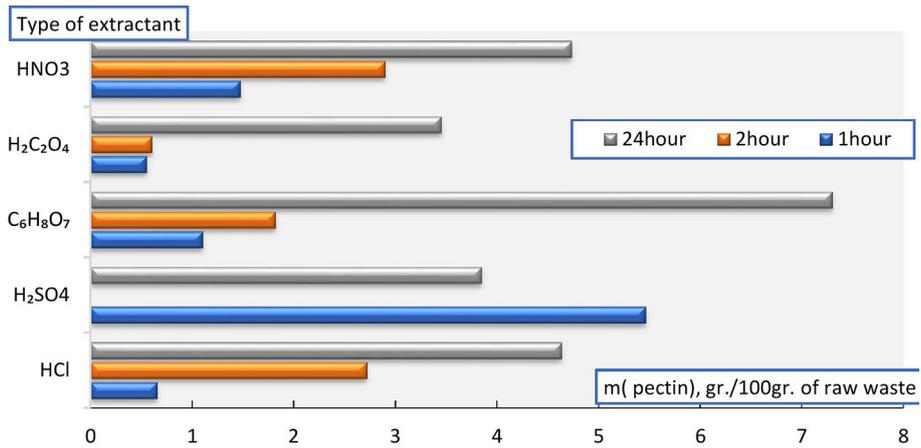


Fig. 8. The dependence of the extraction of pectin from the fruit of the Georgian lemon on time extraction and type of extractant (T(extraction) = 20°C), harvest date – May 2022, water ratio 1:20

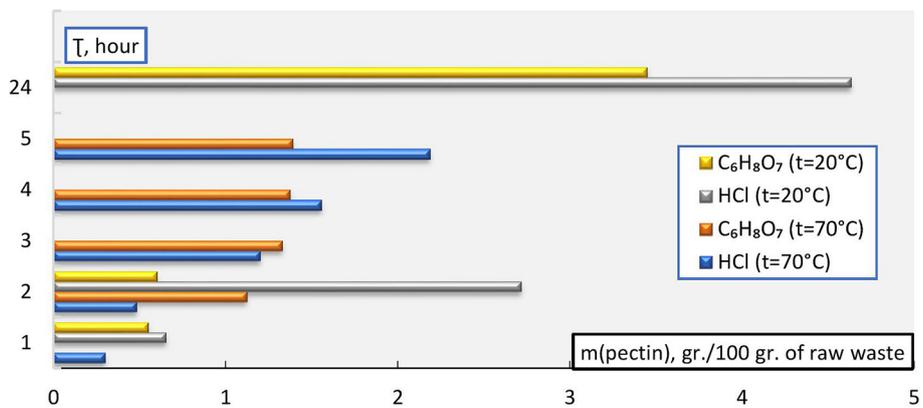


Fig. 9. The dependence of the extraction of pectin from the fruit of the lemon “Meer” on time and temperature extraction (collection date – June 2022, water ratio 1:20)

protopectin decreases, while the amount of soluble protopectin increases.

By extraction, we dissolve protopectin in the extractant, and then we isolate it with alcohol – turning it back into an insoluble state. Such manipulations ensure the completeness of the release of pectin.

It should be noted that at 80°C, regardless of the time of harvesting the fruits, pectin stands out better, but later we worked at a temperature of 60°C, considering it more acceptable – not causing possible partial destruction of the pectin structure

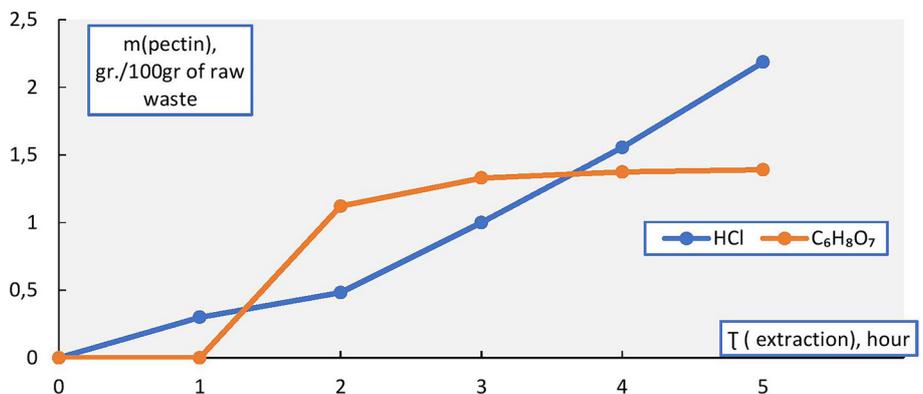


Fig. 10. Dependence of isothermal extraction of pectin from fruits of Meer lemon on the time of extraction and the type of extractant (collection date – June 2022, water ratio 1:20, T= 70°C)

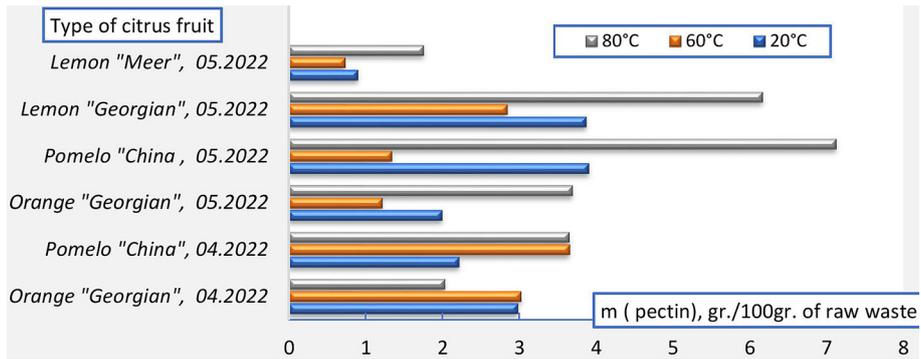


Fig. 11. The dependence of the extraction of pectin on the time of their collection

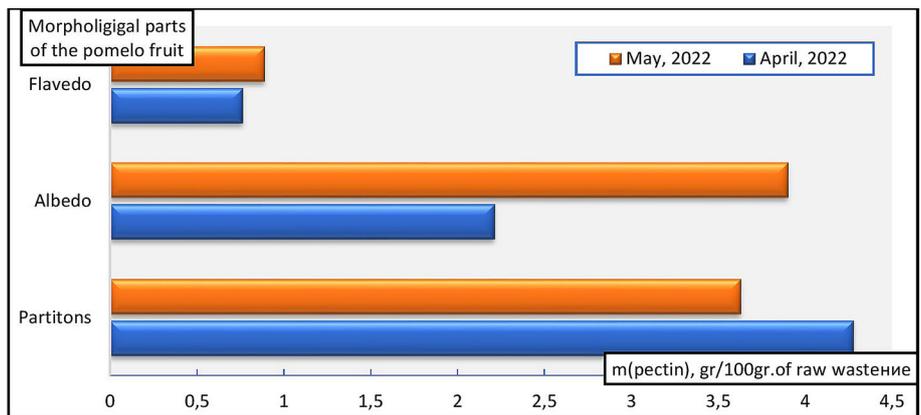


Fig. 12. The dependence of the isothermal morphological extraction of pectin (T=20°C) from the pomelo fruit "China" on the time of their collection

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.

The effect of fruit morphology on the completeness of pectin extraction at different extraction temperatures was studied. From the data obtained it follows (Fig. 12–14) that pectin should

be isolated from the albedo or tissues of the lobules at temperatures of 20 °C and 60 °C from fruits harvested in April, and at a temperature of – 80 °C, harvested in May.

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.

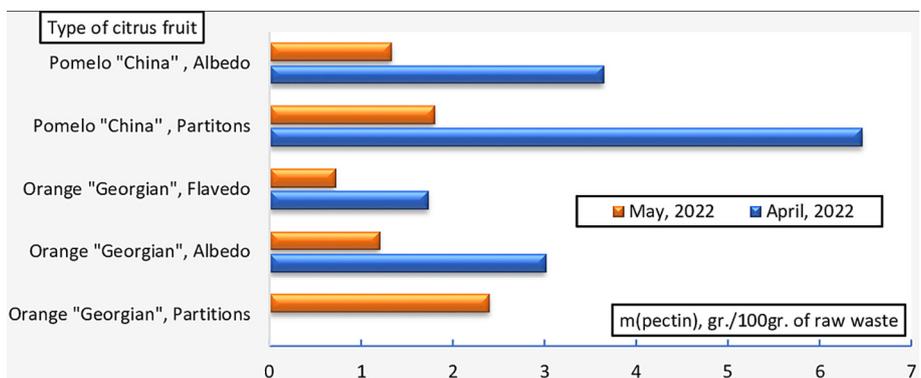


Fig. 13. Dependence of isothermal morphological extraction of pectin (T=60°C) from citrus fruits Pomelo "China" and orange "Georgian" on the time of their collection

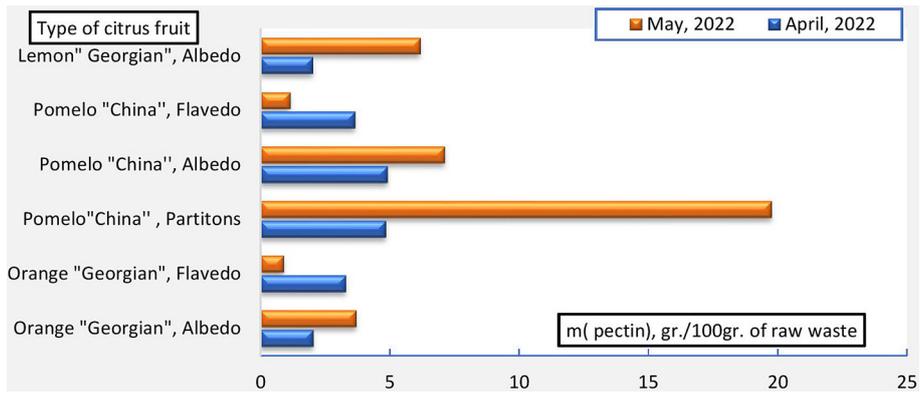


Fig. 14. Dependence of isothermal morphological extraction of pectin (T=80°C) from citrus fruits on the time of their collection

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. Figure 15 shows freshly precipitated and dried samples of citrus pectin.

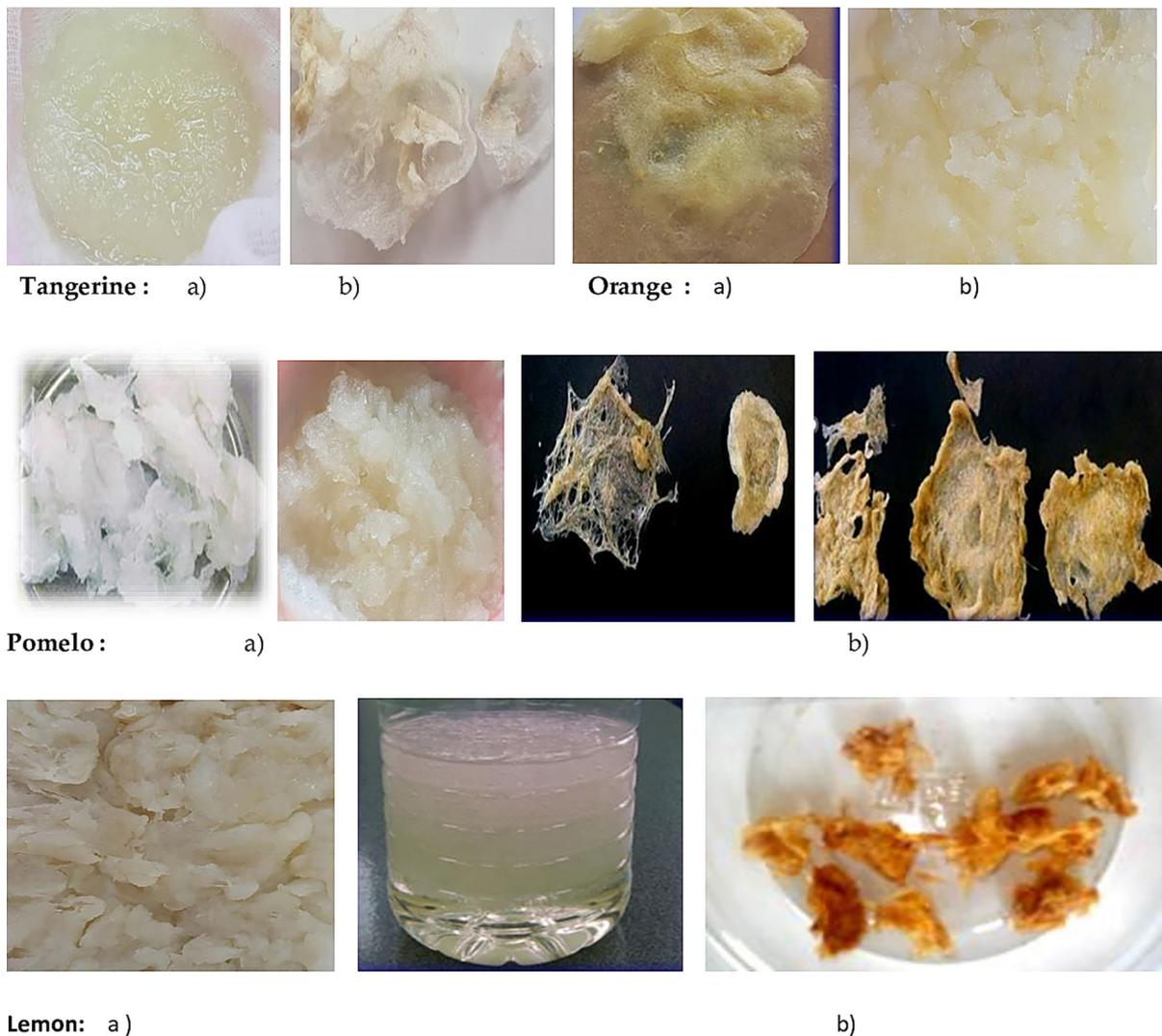


Fig. 15. Samples of citrus pectin (a-freshly precipitated, b-dried): 1.- tangerine, 2.-, 3.-pomelo, 4.- lemon

The resulting pectin samples were analyzed by high performance liquid chromatography. The analysis carried out confirmed the completeness of pectin extraction and proved the purity of its samples.

CONCLUSIONS

The dependence of the production of isolates on the ratio of components of the hydromodule (acid: water), the type of acid (HCl, HNO₃, H₂SO₄, H₂C₂O₄ and C₆H₈O₇), the duration of the process (1, 2, 3, 4, 5 and 24 hours) and the extraction temperature (20°, 60°, 80°C), the type and time of fruit ripening, as well as the type of precipitation reagent pectin (AlCl₃, CaCl₂, 95% C₂H₅OH, isopropanol) and its concentration, duration of extraction (2h, 8h, 12h, 24h) and temperature (20, 40, 60, 70, 80°C). A technological scheme for obtaining pectin extracts was developed. Established extraction of pectin depends on the type and time of fruit collection, temperature and duration of extraction, type of extractant; the ratio of water and acid in the hydromodule (H₂O : acid) should be 1:10; isolate should be extracted with HCl, H₂SO₄ or lemon acid; pH of the hydromodule of the isolate should be 1.8–2.0. Extraction of pectin should be carried out with 95% C₂H₅OH, during 24 hours, with a module of 1:3 at room temperature. Identification of pectin isolates and obtained samples was carried out by the method of high-efficiency liquid chromatography. Obtained practically all samples contain pectin and galacturonic acid and do not contain polygalacturonic acid, which indicates the complete extraction of pectin.

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