Development and implementation of technological processes and modes of preparation of cotton fold for extraction of pectin substances

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Abstract. The technology for the production of pectin from apple pomace, beet pulp, sunflower baskets and many other types of vegetable raw materials is briefly described. The quality of the final finished product - pectin, depends not only on physical and chemical processes, but also to a large extent on the quality and method of preparation of pectin-containing raw materials. An important place in the production of pectin is occupied by the processes of preparation of pectin-containing raw materials, hydrolysis-extraction and precipitation of pectin substances, on the correct implementation of which the yield and physicochemical parameters of the target product largely depend. In this regard, the preparation of pectin-containing raw materials, the research and development of technological regimes and processes for preparing the cotton flap, as a new type of raw material, are relevant and of great scientific and practical importance. Based on the research results, a technological line for the production of pectin from cotton flaps was developed. Pilot-industrial production of powdered pectin was mastered at the created experimental production “Buttermilk” of the Chinaz district of the Tashkent province, Uzbekistan. Moreover, 2.6 tons of food pectin were produced from cotton pods and on its basis 72.0 tons of marmalade were made for sale.

Keywords. Pectin, pectin substances, organoleptic indicators, complexing properties, gelling properties, raw materials, cotton flap.

1. Introduction
Pectin substances as a group of acidic polysaccharides, a component of plant raw materials, were discovered in 1825, in the 19th century and the first half of the 20th century, studies were carried out to study the chemical structure of pectin substances, to determine their quantitative content in various fruits, berries and root crops, and to influence the structural components of a plant cell [1].

One of the most effective natural compounds for detoxifying the body from the harmful effects of radionuclides, heavy metals and other toxic substances is pectin in its various forms, medical preparations and food products based on it [2].

In the former Soviet Union, there was practically no industrial production of pectin, if you do not take into account some small, low-capacity workshops. The physiological and biological value of pectin determines their high demand in the food and pharmaceutical sectors of the economy, and it is currently satisfied much lower (within 25-30%) than its actual need [3]. So, in 1985-1995, an
insignificant amount of apple, beet and citrus pectin was produced, with its need for more than 220 thousand tons per year only for preventive purposes [4].

Another important aspect of the problem under consideration is that pectin, as a natural complexing agent, is of great interest because it has not been studied enough in world practice [5]. Pectin is essential for use in a balanced diet of certain categories of industrial workers, as well as for the population of regions of environmental degradation and environmental pollution, radioactive radiation.

The main reason that does not allow to increase the output of marmalade products is the insufficient supply of industries with gelling substances, including pectin [6]. Taking into account the special physical, chemical and consumer properties of pectin substances, the search for new types of raw materials for the production of pectin, research, improvement and development of new technologies for its production is an urgent task due to the significant excess of its need from the volume of production [7].

In this regard, it remains relevant to find ways to expand the raw material base for the production of pectin, the development of new technologies for the production of pectin substances from various types of raw materials, in particular, from the flaps of cotton bolls [8]. The study, conducted by the author of the article, of the complex influence of the main technological parameters on the processes of preparation of raw materials, hydrolysis-extraction and coagulation of pectin substances from an unconventional type of raw material - cotton flap remains relevant and is of great practical importance [9].

In this regard, the development and implementation of science-based technologies that provide for the production of pectin substances with desired properties from non-traditional types of plant materials and the production of food products based on them is an important industry task [10]. Cotton is a heat-loving tropical plant up to one and a half meters tall. It tolerates drought well, but the soil must be kept moist to produce rich crops. To do this, irrigation is mandatory on cotton fields. Cultivated cotton is an herbaceous plant from 70 cm to 2 m high. There are many branches on its stem and therefore it looks like a bush [11].

Cotton flowers are large, white, yellow or cream. The best, fine-fibered varieties have a reddish spot at the base of the petals. The fruit-box is divided into 3-5 nests. Each nest contains from 5 to 11 seeds [12]. From 5 to 15 thousand fibers 3-5 cm long develop on each seed. Cotton is grown for the sake of these fibers.

In Uzbekistan, mainly Mexican cotton is grown, which has a medium length fiber. The longer and finer the hairs, the higher the fiber grade. The best fiber is from Uzbek and Egyptian cotton. Peruvian cotton has the thinnest and longest fiber [13]. Varieties of fine-fiber flax have been bred from it. This type of cotton is especially valued. From one ton of fine-staple cotton, 16 thousand meters of fabric are obtained, and from 1 ton of medium-staple cotton, only 8.5 thousand meters.

Pectin is a water-soluble substance free from cellulose and consisting of partially or fully methoxylated polygalacturonic acid residues [14]. Depending on the number of methoxyl groups and the degree of polymerization, there are various pectins.

Pectin substances are a group of compounds of a polysaccharide nature that are extracted from a plant cell and contain a large number of chain-linked anhydrogalacturonic acid residues. Pectic substances are physical mixtures of pectins with related substances (for example, pentosans and hexosans) [15].

Protopectin is a water-insoluble natural pectin, consisting mainly of a network of pectin chains formed as a result of the combination of polyvalent metal ions with non-esterified carboxyl groups [15].

2. Materials and methods
It should be noted that the structural features of pectin depend not only on the source of raw materials, but also on the degree of maturity of plant materials. Pectin substances in various quantities are
contained in all parts of plants (in leaves, stems, roots, fruits and seeds). They are differently localized in different parts of the plant cell [2, 3] and perform different functions.

The total content of pectin substances, the ratio of protopectin and soluble pectin differ depending on the type, age, conditions of growth and development of plants. This causes differences in the technological parameters of the extraction and the physicochemical properties of pectin substances (Figure 1).

![Chemical formulation of pectin](image)

Figure 1. Chemical formulation of pectin.

According to the data [4], the largest amount of pectin substances is found in root crops, the most common and accessible raw material for industrial processing. Pectins of various plants have certain differences, the main differences are observed in the quantitative ratio of monosaccharides.

Despite this, in various plants, the main quality indicators of pectin are the same; pectin in fruits and leaves of an apple tree is characterized by a high degree of esterification. Sugar beet pectin is characterized by a low degree of esterification and a high content of free carboxyl groups [3].

The author believes that a deep knowledge of the basic chemical, physico-chemical and consumer properties of pectin and pectin substances is the logical basis for the study of various pectin-containing non-traditional raw materials for the development of optimal and modern technology. Naturally, these indicators are necessary for the rational use of pectin in the food industry and other sectors of the economy.

Based on this, the author took into account the need for a comprehensive and comprehensive study of the chemical and physico-chemical and consumer properties of finished pectin as a polysaccharide and solutions based on it as criteria for searching for a new pectin-containing non-traditional type of raw material.

**Organoleptic characteristics:** Pectin substances, depending on the raw materials produced, the presence of plant pigments, are powders from white to gray and yellowish to brown. Pectin powder is odorless, its taste is slightly acidic. Depending on the technology, pectin is a fine-grained or fine-fibered powder.

**Swelling of pectin:** Molecular weight, degree of esterification, pH, these are the main factors that determine the degree of swelling of pectin substances. With an increase in the molecular weight and degree of esterification, the ability of pectin substances to swell increases [5, 6].

**Solubility of pectin:** The best solvent for pectin is water. The solubility of pectin substances 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 [7, 8]. Mixed in dry form with 5-6 parts of sugar, finely ground pectin dissolves easily in water.

**Viscosity of pectin solutions:** Viscosity is one of the characteristic properties of solutions of pectin substances, like other lyophilic colloids. It increases with increasing pectin concentration. Solutions of pectin substances having a high viscosity are considered valuable in obtaining various food products (fruit juices, apple sauce, jelly, and others).
The molecular weight of pectin depends on its origin. For pectins from apples, pears and plums, this figure is 25000-35000, from beet pulp 16000-25000, from orange 40000-50000, from lemon 23000-71000, from cotton flap 30000-55000.

The author's research confirmed that the differences in the molecular weight of pectin depend not only on the feedstock, but also on the correct conduct of the technological process of its production. From the same raw material, under the influence of various technological parameters or extractants, pectin with different molecular weights can be obtained, which in the final result significantly affects the gelling ability of the drug.

Coagulability of pectin: Coagulability of pectin is proportional to the number of free carboxyl and hydroxyl groups, molecular weight, concentration of coagulated solution and coagulating agent [9, 10]. Pectin is insoluble in organic solvents and can be precipitated from aqueous solutions by adding solvents such as methanol, ethanol, isopropanol, and acetone [11]. Coagulation of pectin substances from aqueous solutions with acetone and ethanol is widely used in their production. Polyvalent metals are also used for coagulation of pectin substances.

Complexing properties: One of the most important properties of pectin substances is the complexing ability based on the interaction of the pectin molecule with ions of heavy and radioactive metals. This property gives reason to recommend pectin for inclusion in the diet of people who are in an environment contaminated with radionuclides and who have contact with heavy metals. Due to this chemical property, pectin can be classified as an indispensable substance for use in the production of food products for preventive and therapeutic nutrition.

The optimal prophylactic dose of pectin is no more than 2-4 g per day [3, 12] for those in contact with heavy metals, in conditions of radioactive contamination - not less than 15-16 g [13]. At the same time, beet pectin is one of the pectins with the highest complexing ability.

Gelling properties: Gelling ability is the most important property of pectin, which characterizes its consumer qualities for the confectionery and canning industries. Gel formation depends on a number of factors: molecular weight, degree of esterification, sugar concentration, the amount of ballast substances that exist in this pectin, temperature and pH, and the content of functional groups. The conditions for the gel formation of pectin are determined by the degree of esterification. To obtain jelly of sufficient strength, it is necessary: 60% sugar content, 0.5-1% pectin concentration, optimal pH (2.6-3.1).

In order to develop a universal technology of pectin, which provides for the processing of various raw materials on the same process-hardware scheme, and to identify other industrial raw materials for the production of pectin substances, a classification of pectin-containing raw materials was compiled according to the content of pectin substances and the features of preparing raw materials for hydrolysis-extraction and the technology for isolating pectin were studied.

3. Results and discussion

The study of existing technologies for the extraction of pectin substances from various types of raw materials allows us to conclude that in connection with the expansion of the field and the increase in the need for the use of pectin substances in the food, pharmaceutical industry, as well as in medicine, the search for new non-traditional raw materials resources to increase the volumes is timely and relevant.

The organization of highly profitable industrial production of pectin production requires the necessary improvement of technological schemes and their instrumentation based on deep scientific research, kinetics and the mechanism of processes.

Traditional types of raw materials for the production of pectin on an industrial scale are citrus peel, apple pomace and beet pulp. Due to the limited raw materials and the complexity of the technology, pectin enterprises do not operate at full capacity. Under these conditions, the problem arises of finding alternative readily available raw materials for the production of pectin.

The author of the article, as part of a scientific team, conducted research on the study and research of the cotton flap as a pectin-containing raw material. Every year in the cotton-growing republics of
Central Asia, more than 2.0 million tons of cotton flaps are formed that do not find proper practical application, from which about 150 thousand tons of valuable food pectin could be produced.

The best way to store raw materials is to keep the crushed leaf in an atmosphere of sulfur dioxide, which excludes the development of microorganisms that cause damage to the material. To do this, the cleaned leaf is crushed, immersed in hermetically sealed reinforced concrete tanks, where, after full loading, sulfur dioxide is supplied (100-125 mg/liter of tank volume). The unloading of the crushed sash is carried out by pneumatic transport devices.

In accordance with the requirements of the normative and technical documents on standardization “Cotton box flap”, the cotton flap must meet the requirements specified in Tables 1 and 2.

### Table 1. Organoleptic characteristics of dry cotton flap.

| Organoleptic indicators | Characteristics of raw materials |
|-------------------------|----------------------------------|
| Appearance              | In loose form without mold, dry  |
| Color                   | Greenish yellow to brown         |
| Odor                    | Specific without musty, moldy and other foreign odors |

### Table 2. Physical and chemical parameters of dry cotton flap.

| Physical and chemical indicators | Norms |
|---------------------------------|-------|
| Mass fraction of moisture, %, no more than… | 8.0   |
| Infection with microorganisms   | not allowed |
| Mass fraction of foreign impurities: stems, leaves, tops, raw cotton, sand and others, %, no more than… | 5.0   |
| Maturity                        | sash from immature boxes is not allowed |

*Note: The content of pectin substances precipitated by alcohol must be at least 5.0%.*

The quality of the final finished product - pectin - depends not only on physical and chemical processes, but to a greater extent on the quality and method of preparation of pectin-containing raw materials. In the production of pectin, special requirements are imposed on the preliminary preparation of raw materials, depending on its state (dried or wet) and the specified physicochemical properties of the pectin contained in it.

According to the theory of mass transfer in the solid-liquid system, in order to accelerate the process of extracting the target product, it is necessary to achieve the maximum surface of the extracted particles and ensure unimpeded access of the hydrolyzing agent to the internal parts of the plant cell. Therefore, regardless of the physical condition, it is necessary to ensure the maximum crushing of the cotton flap.

Dried cotton sash, unlike dried apple pomace and dried beet pulp, is ready for processing without the use of a heat agent. This is one of the distinguishing features of the cotton flap as a pectin-containing raw material for its industrial processing. It is not allowed to harvest raw materials in the rain and during snowfall or process rotten doors. Prepared flaps of cotton bolls should be promptly shipped to the pectin plant for industrial processing.

The sash is dried with a moisture content above 10%. In this case, modes of drying the cotton flap should be developed, which should ensure the safety of the molecules of pectin substances and the physicochemical parameters of pectin in the cotton flap. From the results [50], it can be seen that the temperature regime of drying has an effect mainly on the content of pectin substances in the cotton flap, the ability to form gel and its color. With an increase in temperature and duration of drying (above 100 °C for 2-2.5 hours), the gel-forming ability of pectin decreases, as a result of the oxidation of phenolic compounds of a plant cell under the influence of atmospheric oxygen, the color of the leaf changes from slightly brown to dark brown.

Based on numerous experiments of the author, it has been established that the optimal conditions for drying a cotton flap, which ensure its high quality indicators, are drying at a temperature of 70 °C for 1-1.5 hours with intense convection with hot air. Changes in pectin substances during storage of
the cotton flap: Studies have shown that when storing the cotton flap with low humidity (7-8%), microbiological spoilage of raw materials does not occur, conditions are created for processing a full-fledged material and obtaining a high-quality preparation.

In this regard, it became necessary to study the change in the state of pectin substances during the storage of the leaf. Samples of the Tashkent-1 variety valve from the Tashkent region were put into storage, the observation was carried out for two years. During storage, the parameters of the ambient air fluctuated within the following limits: relative humidity - 70-82%; temperature from -18 to +45 °C. The dynamics of changes in the content of pectin substances and the strength of a standard jelly are presented in Table 3.

**Table 3. Changes in the content of pectin substances.**

| Cotton sash storage by month | Yield of pectin substances on air-dry weight, % | Strength of the standard jelly on the Sosnovsky device, mm.rt.st. |
|-----------------------------|-----------------------------------------------|------------------------------------------------------------------|
| 2 month storage             | 7.95                                          | 668.0                                                           |
| 6 month storage             | 8.0                                           | 684.0                                                           |
| 10 month storage            | 8.10                                          | 653.0                                                           |
| 14 month storage            | 8.12                                          | 684.0                                                           |
| 18 month storage            | 7.78                                          | 638.0                                                           |
| 24 month storage            | 7.76                                          | 653.0                                                           |

The indicators presented in the table show that after the first 14 months of storage, there are no noticeable changes in the yield and quality of pectin substances. The subsequent storage period is a slight decrease in the content of pectin. All other indicators characterizing the quality of pectin remained at the same level.

Thus, the author's research has established that storage (24 months) of a dry cotton flap under conditions of temperature fluctuations $t = -18 + 45 ^\circ C$ and relative humidity of the ambient air $U = 70 - 82\%$ does not cause any special changes in the composition and physico-chemical parameters of pectin.

Development of the mode of swelling of the dried crushed cotton flap: The technological scheme for obtaining food pectin from the cotton flap is based on the processing of dried raw materials to a residual moisture content of 7-10%, the structure of which is a capillary-porous system. The rate of filling these systems with a hydrolyzing agent and the transfer of pectin substances from plant tissue depends on their diffusion capacity.

At various stages of the technological process (Figure 2), pectin-containing raw materials undergo significant changes in mass, structure, and physicochemical composition [7]. Hydrolysis of protopectin from a dried valve suggests swelling of the plant tissue, while the effect of an acidic environment on the upper particle of the material is longer than on the inner one. This leads to partial depolymerization of pectin molecules and negatively affects the yield and gelling ability of the preparation.

To identify physical and chemical changes in the raw material, the kinetics of pre-swelling of the dried leaf was studied. Leaflets weighing 100 g (moisture content 8%) were placed in a thermostated beaker and poured into 800 ml of water at a temperature of 70 °C.

After a certain period of time (30 min), the water was drained through the filter. The swollen pulp was weighed on a scale and the amount of water absorbed by the raw material was calculated. In the water after swelling, the content of solids and pectin substances, calcium, was determined with a refractometer using the pectate method.

It has been established that water is intensively absorbed in the first 20 minutes, with an increase in temperature, the water absorption of the leaf increases (Figure 2). Water, filling the capillary system of the pulp, diffuses from the composition of the plant cell into the liquid of polyphenols, ballast substances and soluble pectins, and thereby increases the concentration of solids in the liquid phase.
The regimes for washing raw materials from polyphenols with a 3% NaCl solution for 30 minutes, at a hydromodulus of 1:8, at a temperature of 70 °C, were established. The balance of products was compiled during the preliminary preparation of raw materials, and humidity - 8%, concentration of pectin substances - 10-15%. It has been experimentally established that the degree of extraction of pectin substances from the swollen pulp is 10-12% higher compared to the leaf without preliminary swelling.

Taking into account the results of research and the properties of pectin-containing raw materials, a schematic diagram of the preparation of the cotton flap for the process of hydrolysis - extraction, which is shown in Figure 3.

**Figure 2.** Material balance of the sash washing process.

**Figure 3.** Technological scheme for the preparation of the cotton flap.
4. Conclusions
An analysis of the current state of technology and pectin production technology shows that the demand for pectin far exceeds its production volumes. The use of pectin is extremely necessary in the conditions of environmental degradation and environmental pollution, in particular for the population of areas of ecological catastrophe. Research work is underway to improve the technological regimes for the production of pectin from apple pomace, beet pulp, sunflower baskets and many other types of vegetable raw materials.

The increase in pectin production depends on the search for cheap, easily accessible technological plant raw materials for these purposes, the development of fundamentally new technological schemes, taking into account the specificity of this raw material.

As a result of the search work, the team of authors identified a new source of raw materials for the production of food pectin - cotton bolls flaps. Every year in the cotton-growing republics of Central Asia, about 2 million tons of cotton flaps are formed, which do not find proper practical application, although the bulk of this amount is suitable as a cheap raw material for the production of food powdered pectin from cotton flaps.

The quality of the final finished product - pectin, depends not only on physical and chemical processes, but also to a large extent on the quality and method of preparation of pectin-containing raw materials. An important place in the production of pectin is occupied by the processes of preparation of pectin-containing raw materials, hydrolysis-extraction and precipitation of pectin substances, on the correct implementation of which the yield and physicochemical parameters of the target product largely depend.

In this regard, the preparation of pectin-containing raw materials, the research and development of technological regimes and processes for preparing the cotton flap, as a new type of raw material, are relevant and of great scientific and practical importance.

References
[1] Levic L and Petrov S 2001 Elektrokinetec properties of some compounds important in sugar guice refinig Indust Secern 35 30-35
[2] Reginald H and Ruth S 2003 The induct stablization of agueons pectin disprtions by ethanol Food Soi 48(4) 254-263
[3] Deuel N, Solms I and Denzler A 1994 Klarung won Fiuchtsabtttn mit Polymeren basen. -mitt Geb. Zebensmitte lunters und Hyg 45 73-84
[4] Stutze D 2005 Polymapholyte mit verschiedener La-dungsverteilung 61 1757-1763
[5] Degani O 2021 Synergism between Cutinase and Pectinase in the Hydrolysis of Cotton Fibers’ Cuticle Catalysts 11(1) 84
[6] Degani O, Gepstein S and Dosoretz C G 2002 Potential use of cutinase in enzymatic scouring of cotton fiber cuticle Applied biochemistry and biotechnology 102(1) 277-289
[7] Solbak A I, Richardson T H, McCann R, Kline K A, Bartnek F, Tomlinson G and Kerovuo J 2005 Discovery of pectin-degrading enzymes and directed evolution of a novel pectate lyase for processing cotton fabric Journal of Biological Chemistry 280(10) 9431-9438
[8] Lakshminarayanan K 2006 Physiology of host-parasite relationship in the Fusarium wilt of cotton In Proceedings of the Indian Academy of Sciences-Section B 44(6) 317-324
[9] Pedrolle D B, Monteiro A C, Gomes E and Carmona E C 2009 Pectin and pectinases: production, characterization and industrial application of microbial pectinolytic enzymes Open Biotechnology Journal 9-18
[10] Saharan R and Sharma K P 2019 Production, purification and characterization of pectin lyase from Bacillus subtilis isolated from moong beans leaves (Vigna radiata) Biocatalysis and Agricultural Biotechnology 21 101306
[11] Wang H, Guo Y, Lv F, Zhu H, Wu S, Jiang Y and Zhang T 2010 The essential role of GhPEL gene, encoding a pectate lyase, in cell wall loosening by depolymerization of the de-esterified pectin during fiber elongation in cotton Plant molecular biology 72(4) 397-406
[12] Ishii S and Yokotsuka T 2001 Maceration of plant tissues by pectin trans-eliminase *Agricultural and Biological Chemistry* 35(7) 1157-1159

[13] Sae-be P, Sangwataranaroj U and Punnapatak H 2007 Analysis of the products from enzymatic scouring of cotton *Biotechnology Journal: Healthcare Nutrition Technology* 2(3) 316-325

[14] Hoondal G, Tiwari R, Tewari R, Dahiya N and Beg Q 2002 Microbial alkaline pectinases and their industrial applications: a review *Applied microbiology and biotechnology* 59(4) 409-418

[15] Zhang Y, Chen S, Xu M, Cavoco-Paulo A, Wu J and Chen J 2010 Characterization of Thermobifida fusca cutinase-carbohydrate-binding module fusion proteins and their potential application in bioscouring *Applied and environmental microbiology* 76(20) 6870-6876