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EXTRACTION OF COTTON PECTIN

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Technological regimes for the hydrolysis-extraction of pectin substances from cotton valves have been developed.

The hydrolysis-extraction of pectin substances is one of the main stages in the technology of obtaining pectin from plant material.

It is known that the pectin substances in the bolls of cotton plants exist both in soluble form in the cell juice and in the form of insoluble pectin or protopectin in the plant cell walls. In addition to pectin, the cells contain cellulose, hemicellulose, proteins, lignins, and other biopolymers.

The cell plate is formed mainly from protopectin and Ca^{++} and Mg^{++} pectinates; it contains no cellulose. Cotton valves contain up to 8% of polyphenols of catechin nature [1], which must be subjected to purification. After cotton valves have been washed with a mineral salt solution the coloring substances, consisting of pigments of polyphenolic nature, pass from them into solution, the cell boundaries become sharper, and the walls are retained [2, 3]. After treatment with oxalic acid, the middle lamellae are exhausted and the cell walls break down as the result of an exchange reaction between the calcium (magnesium) ions of the pectinates and the oxalate ion. As a result, the pectin, freed from metal ions, becomes water-soluble and passes into the extract.

With the aim of determining the optimum conditions of hydrolysis ensuring the required physicochemical indices and a high yield of pectin, we have carried out a series of investigations, and, from the results of these, have developed parameters, regimes, and methods of extracting the pectin substances of cotton valves by varying the temperature, the length of the process, the pH of the medium, the liquor ratio, and the type of hydrolyzing agents and their concentration.

In the hydrolysis-extraction of pectin substances different hydrolyzing agents are used according to the type of raw material [4, 5]. In order to choose the extractant we used solutions of the following acids: H_2SO_4, H_3PO_4, HNO_3, HCl, $(COOH)_2$, and $C_6H_9O_7$. As can be seen from Fig. 1, the best results were obtained with 0.3-0.5% solutions of $(COOH)_2$.

In view of the relatively low yield of pectin in a single extraction, the insoluble residue was subjected to a second extraction. The best yield was ensured by the use of a 0.3-0.5% solution of $(COOH)_2$ in the first stage and a 0.2-0.3% solution of HCl in the second stage. In the first stage of this process, the calcium and magnesium pectinates are decomposed with the formation of pectin and the oxalates of these metals, and in the second stage the intracellular protopectin is cleaved to form low-molecular-mass water-soluble pectin [6].

In order to determine the best liquor ratio for extraction, the comminuted raw material was treated with a 0.5% solution of $(COOH)_2$ and the ratio of raw material and extractant was varied within the range of 1:6-1:15. At a liquor ratio of 1:6 the viscosity of the hydrolysis mixture was high, a gruel-like mass being formed. Increasing the liquor ratio led to a fall in the concentration of pectin substances in the extract. In this case the quality and indices of the samples did not change appreciably but the consumption of organic and mineral acids and alcohol, and also of other auxiliary materials and electrical energy, increased. On the basis of the results obtained, we recommend that the hydrolysis-extraction of pectin substances from cotton valves should be carried out at liquor ratio of 1:8-1:10. The amount of cotton pectin in the extract is then considerably increased but no appreciable influence of the changes in the dilution coefficient on the yield of pectin substances and their main indices and also on the gel-forming capacity of the pectin has been detected.

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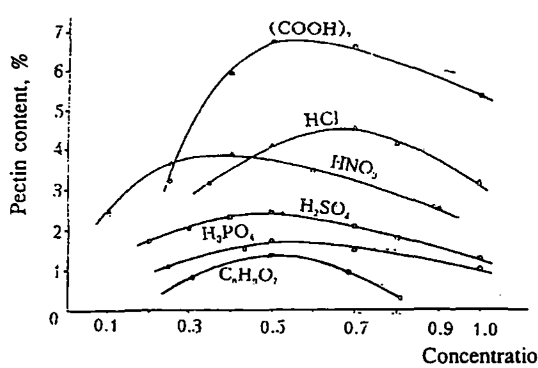

Fig. 1. Dependence of the yield of pectin on the concentration of various acids.

The hydrolysis-extraction of pectin substances depends on the diffusional properties of the plant tissue [7] and the permeability of the cells. With a rise in the temperature, the bond between the high polymers in the plant tissue is broken, the speed of the molecules and the permeability of the cells are increased, the conversion of the protopectin into pectin is accelerated, and the solubility of the latter is enhanced. In the experiments the temperature of hydrolysis-extraction was varied from 60 to 90°C. The best result for yield and quality of the pectin was obtained at 75-80°C.

In order to establish the optimum duration of the hydrolysis-extraction of pectin substances, the length of the experiments was varied from 50 to 130 min. With an increase in the time of treatment from 50 to 90 min the passage of pectin substances into the extract increased, but above 90 min the yield of pectin substances fell and there was a breakdown in the structure of the cotton valve particles as a result of which the conditions for separating the liquid phase in the process of filtration deteriorated sharply, the amount of ballast substances increased, and the intensity of the color of the final pulverulent product rose. The gelling capacity of all the samples was independent of the time of the process, and the jelly obtained by the use of a solution of HCl was transparent, glassy, and straw-colored.

The highest yield of pure pectin powder (containing 80-85% of polyuronides and the smallest amount of ash) was obtained at a time of hydrolysis-extraction of the pectin substances from cotton valves of 80-90 min.

EXPERIMENTAL

Purification of the Raw Material. Cottonplant valves from the 1995 harvest (1 kg) were comminuted to 4-5 mm and were loaded into a bottom-discharge diffuser. Water-soluble impurities were extracted with 8 liters of 3% NaCl solution at 20-25°C by steeping for 30 min. The raw material was separated from the liquid phase by filtration on a suction filter and was washed with 5 liters of water to displace the residual extractant.

Extraction of the Pectin. The purified raw material was loaded into a diffusor and covered with 10 liters of a 0.3-0.5% solution of $(COOH)_2$ at ~85°C. The temperature was kept at 75-80°C by the passage of live steam into the steam jacket. The process took 80 min with periodic stirring. Then the extract was separated by centrifugation. A second extraction was conducted analogously, using a 0.25% solution of HCl.

Concentration of the Extract. The combined extract (16 liters) was concentrated in vacuum to 1.6 liters at a temperature not exceeding +60°C.

Precipitation of the Pectin. In a glass reactor with a stirrer, the 1.6 liters of concentrated extract was diluted with 3.2 liters of 96% C_2H_5OH with constant stirring, and the mixture was left for 2 h for complete precipitation. Then the pectin precipitate was centrifuged off.

Purification of the Pectin Precipitate. The pectin precipitate was treated with 2 liters of a 1% solution of HCl in 70% C_2H_5OH to eliminate traces of ash-forming substances, and the residue was treated successively with 70, 80, and 96% alcohols.

Drying of the Pectin. The dewatered pectin precipitate was dried in a vacuum dryer at 70°C to a final moisture content of 5%.

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