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FEATURES OF THE PRECIPITATION OF PECTIN SUBSTANCES FROM THE VALVES OF *Gossypium hirsutum*

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An effective agent for precipitating pectin substances from the valves of cotton bolls has been selected.

Factors of the technological processes, including the precipitation of pectin substance, affect the physicochemical indices of the finished product [1, 2]. Organic solvents and salts of polyvalent metals are used to precipitate pectin from an extract [3]. The method of precipitating pectin with salts of polyvalent metals is used in its isolation from sunflower heads and beet pulp [4]. The capacity of a pectin for being precipitated by metal salts depends on the type of raw material, the method of its treatment, the degree of esterification of the pectin, and the conditions of the precipitation process [5].

In order to raise the yield of cotton pectin, a series of investigations has been conducted to select the optimum precipitant of pectin substances from an extract using organic solvents and salts of polyvalent metals. The influence of various precipitants on the physicochemical indices of the pectin has been studied. Comparative investigations have been made of the characteristics of the quality of the pectin obtained by the use of various precipitants.

The pectin-precipitating process has been studied with the use of extracts obtained by a single treatment of the raw material with a 0.5% solution of $(COOH)_2$ (method 1) and two treatments of the raw material successively with a 0.5% solution of $(COOH)_2$ and a 0.3% solution of HCl (method 2). Table 1 gives the yields and physicochemical indices of pectins obtained with various precipitants. The samples of pectin isolated by the various precipitants had different yields and physicochemical indices.

Among the disadvantages of polyvalent metals as precipitants of cotton pectin must be mentioned the increased color of the final product and the high consumption of acid and alcohol in the purification of the pectin from residual precipitants.

The results of an investigation of the process of precipitating cotton pectin with salts of polyvalent metals followed by purification of the pectin coagulate by washing with alcohol acidified with hydrochloric acid showed that this method does not enable the desired product to be obtained in high yield and with the optimum chemical indices [6].

In order to shorten the stages of the production cycle and increase the yield of pectin, we have made a search for the best organic precipitants of pectin (ethanol, isopropanol, acetone).

In the precipitation of pectin a clear separation of the mother liquor from the pectin precipitate is necessary. Therefore, in the precipitation of cotton pectin by organic solvents the ratio of the extract to be precipitated and precipitant is 1:2.

Table 2 gives the yields and physicochemical indices of the pectins obtained by precipitation with various organic solvents. A comparison of the results of the precipitation of cotton pectin by organic solvents shows the advantage of ethyl alcohol, which ensures the production of a pectin with a considerably increased yield and with the required physicochemical indices.

In the selection as precipitants of salts of polyvalent metals and organic solvents we took into account the yield of cotton pectin, the physicochemical indices of the samples obtained, and the economic efficacy and harmlessness of the use of these precipitants. Taking these requirements into account, 96% ethanol is recommended as an effective precipitant for the isolation of pectin substances from an extract of cotton valves.

EXPERIMENTAL

Purification of the Raw Material. Air-dry cottonplant valves from the 1995 harvest (1 kg) were steeped in 10 liters of a 3% solution of NaCl for 30 min. The insoluble residue was filtered off and was washed on the filter with 5 liters of water.

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TABLE 1. Yields and Physicochemical Indices of Pectins Precipitated by Salts of Polyvalent Metals

Precipitant	Yield, %	Moisture content, %	Content, %				Ash content, %	Degree of esterification, %	Viscosity	Molecular mass	pH of a 1%-solution	Equivalent mass	Gelling capacity, mm Hg	Pure pectin content, %
			COOH groups	OCH_3 groups	$COCH_3$ groups	ballast substances								
Method 1														
$AlCl_3$	7.8	13.26	12.27	3.37	0.50	15.85	1.24	28.74	2.6	29295.1	3.16	252.63	592.8	62.06
$Al_2SO_4 \cdot 18H_2O$	6.80	12.54	10.69	5.49	0.59	15.87	1.88	43.01	3.3	35705.8	3.20	319.40	532.0	65.70
$CaCl_2$	9.45	9.63	9.56	4.90	0.23	12.50	15.64	42.99	2.1	23547.1	3.44	319.28	212.8	60.22
$CuSO_4 \cdot 5H_2O$	15.6	8.56	9.03	4.03	0.32	9.89	21.25	39.63	1.75	18641.4	3.34	373.73	516.8	55.63
Method 2														
$AlCl_3$	7.95	13.30	12.28	3.95	0.59	16.71	1.59	32.11	2.4	27139.6	3.22	265.87	562.4	62.56
$Al_2SO_4 \cdot 18H_2O$	7.40	12.46	10.54	5.23	0.63	17.10	1.97	36.12	3.1	34058.28	3.26		516.8	60.18
$CaCl_2$	7.28	10.26	7.73	4.59	0.28	12.50	15.64	32.11	3.1	34058.28	4.1	341.88	440.8	52.44
$CuSO_4 \cdot 5H_2O$	11.28	11.86	10.76	3.43	0.42	12.10	6.73	31.94	1.84	19750.4	3.1	265.17	501.0	57.30

TABLE 2. Yields and Physicochemical Indices of Pectins Precipitated by Organic Solvents

Precipitant	Yield, %	Moisture content, %	Content, %				Ash content, %	Degree of esterification, %	Viscosity	Molecular mass	pH of a 1%-solution	Equivalent mass	Gelling capacity, mm Hg	Pure pectin content, %
			COOH groups	OCH_3 groups	$COCH_3$ groups	ballast substances								
Method 1														
C_2H_5OH	8.35	13.78	12.32	6.06	0.36	4.77	0.50	41.99	3.7	38484.30	3.18	313.53	706.8	83.23
C_2H_5OH*	6.90	13.02	12.56	6.12	0.24	4.98	0.21	48.05	3.4	36511.11	3.80	351.74	692.6	83.11
Isopropanol	6.70	13.54	11.47	6.78	0.60	7.22	0.74	46.52	4.5	44049.21	3.90	341.28	585.2	83.10
Acetone	7.45	12.75	12.14	6.58	0.42	7.40	0.47	44.36	4.1	41546.83	3.44	327.49	516.8	83.73
Method 2														
C_2H_5OH	7.40	13.13	11.42	6.71	0.50	6.71	0.13	46.38	5.6	49933.5	3.7	340.35	623.2	82.58
C_2H_5OH*	7.0	12.02	12.22	6.86	0.35	5.12	0.17	48.22	4.6	44643.8	3.7	352.74	586.2	84.42
Isopropanol	6.83	13.46	11.12	6.90	0.45	7.71	0.23	45.64	4.7	45219.9	3.92	344.74	562.4	82.85
Acetone	8.80	12.89	11.07	6.43	0.56	13.82	0.16	46.07	4.2	42191.0	4.2	339.25	577.6	73.29

*Precipitation of a concentrated extract with ethanol.

Extraction of the Pectin Substances. The pectin substances were extracted from the purified raw material in stages with a 0.3% solution of $(COOH)_2$ and a 0.25% solution of HCl by steeping for 80 min at 75-80°C. The mixtures were filtered and the filtrates were combined to give a total of 16 liters of extract, which was clarified by centrifugation at 3000-4000 rpm for 15-20 min.

For the precipitation of the pectin by organic solvents, the clarified extract was concentrated to 2 liters in a vacuum evaporator at 60-70°C. The pectin was then precipitated by the gradual addition of 4 liters of ethyl alcohol. The precipitated pectin was separated off by decantation. The pectin residue was washed in stages with 1 liter each of 70, 80, and 86% alcohols. The washed and pressed-out pectin was dried in a vacuum dryer at 70°C to a moisture content of 5-7%.

Precipitation of Pectin with Salt Solutions. The pectin extract was neutralized to pH 5-7 by the addition of 25% NH_4OH solution with vigorous stirring. To the neutral solution were added 20% solutions of polyvalent metal salts in an amount of 80-100 ml per 1600 ml of the initial extract, and the mixture was left for 1 h.

The pectin was obtained by the procedure of [6]. Its physicochemical indices were determined in accordance with OST [Sector Standard] 1862−72 [7], its gelling capacity by the procedure of [8], and its molecular mass viscometrically.

REFERENCES

1. G. V. Buzina, Khlebopek. Konditer. Prom-st', No. 4, 10 (1962).
2. V. G. Moiseev, G. M. Zaiko, and M. K. Anan'eva, Izv. Vyssh. Uchebn. Zaved., Pishch. Tekhnol., Krasnodar (1984).
3. V. G. Moiseev, G. M. Zaiko, and I. A. Medvedev, Izv. Vyssh. Uchebn. Zaved., Pishch. Tekhnol., Krasnodar, No. 2, 29 (1974).
4. V. V. Porfinenko, Dissertation ... Candidate of Technical Sciences [in Russian], Moscow (1969), p. 120.
5. L. B. Sosnovskii, in: Proceeding at an All-Union Conference on Questions of the Technology and Chemistry of Pectin [in Russian], Moscow (1982).
6. N. Sh. Muminov and Kh. T. Salomov, Investigation and Selection of the Optimum Precipitant for Cotton Pectin [in Russian], Moscow (1992), p. 2; Dep No. 2467, AgroNIITÉIpishcheprom., Jan. 18 (1992).
7. OST [Sector Standard] 18-62−72. Dry Food Beet Pectin [in Russian].
8. V. V. Arasimovich, S. V. Baltaga, and N. P. Ponomareva, Methods of Analyzing Pectin Substances, Hemicelluloses, and Pectolytic Enzymes in Fruits [in Russian], Shtiintsa, Kishinev (1970).