

PRODUCTION OF PECTIN FROM SOME LEGUMES HULL

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Abstract

Pectin was isolated from soy and broad bean hulls using HCl 0.1 N, under reflux at 90^o C for 45 min. The precipitate was washed several times with isopropanol and the pectin was dried using the freeze drying method. The physico- chemical characteristics were determined and the results indicated that the pectin obtained from soy and broad bean hulls was high in either viscosity or shear stress. Also, methoxyl content was up to 8.0 % for pectin of the two sources. These results indicate that this pectin may have a good solubility and high gelatinization. Whereas, the degree of esterification and anhydro-galactouronic acid in soy and broad bean hulls pectin were (62.05 and 58.84%) and (78.07 and 79.6 %), respectively. These values indicate the high qualities of these isolated pectins.

Pectin prepared from soy and broad bean hulls was separately used at different levels (2.0, 4.0 and 6.0 g/kg sugar) for the laboratory preparation of strawberry jam. The sensory evaluation ascertained that the addition of 6.0 g soy hull pectin to each 1.0 kg sugar for the preparation of strawberry jam was the best. The net yield of the jam produced was about 1.45 kg, whereas, the same obtained by the addition of 4.0 and 6.0 g pectin from broad bean hull gave 1.45 and 1.50 kg respectively, of strawberry jam having the same scores of acceptability.

From these results, it could be concluded that the pectins prepared from soy and broad bean hulls had similar effects. However, the yield obtained by the utilization of commercial pectin (citrus pectin) was the best as it gave 1.6 kg yield of jam being as more economical.

INTRODUCTION

More than 50% of the world's pectin production is used for jellies, jams, marmalades and confectionery products. The ability of pectin to form gels is its most important property and consequently determines its commercial value. However, the

knowledge of setting time and temperature are important factors to obtain the optimal gel strength of the pectin in the product. (Barford and Pederson, 1990).

Pectin is a linear polysaccharide consisting of a few hundred to one thousand saccharide units. The average molecular weight of pectin varies from 50,000 to 150,000 Dalton (Whistler and Be Miller, 1997). Pectin consists of D. galacturonic acid units with very small quantities of natural sugars. The monomers are linked together by α 1-4 glycosides linkages (Thakur et al., 1997). The polygalacturonic acid is partially esterified with methoxyl groups and the free acid group may be partially or fully neutralized with sodium, potassium or ammonium ions. The textures of fruits and vegetables, and hence the processing characteristics are largely influenced by the pectin content. Pectin is an ingredient in the pharmaceutical preparation such as antidiarrheal detoxicant and demulcent formulations (Voragen et al., 1995). Moreover, medical applications of pectin include serum cholesterol- lowering agents (Fernandez et al., 1994), and affect glucose metabolism by lowering the glucose response curve (Voragen et al., 1995)

Soy hull pectin was prepared by hydrochloric acid extraction, followed by alcohol precipitation and the effect of acid concentration used in extraction and the pH value of the precipitating solvent on the yield and purity of pectin were investigated. Strength of acid used for extracting pectin from soy hull and the pH value of precipitation had significant effects on the pectin yield. Highest yields of 26 and 28% were obtained when the acid strength was 0.05 and 0.1 N., respectively, and the pH value of the precipitating solution was 3.5. Also, galacturonic acid content and degree of esterification of the products varied from 68- 72% and 56 – 60% , respectively (Kalapathy and Proctor, 2001). Whereas, Monsoor and Proctor (2001) found that galacturonic acid content and degree of esterification were 63.07 to 68.72 % and 17.59 to 20.79 % , respectively , in soy hull pectin extracted at different hull- solvent ratios. Ayyad (1997) found that natural sugar residues presented in all pectin are arabinose, galactose, rhamnose and further glucose and xylose. No mannose could be detected in most pectin. Total natural sugars ranged from 7 to less than 15%.

The aim of this investigation is to use soy and broad bean hulls as a waste of low cost to produce high quality pectin which could be used in jam processing and which

would partially reduce the hard foreign currencies parcel for the importations of commercial citrus pectin .

MATERIALS AND METHODS

Materials :

Soybean hulls (*Glycin max.*, L) and commercial pectin were obtained from the Soy Processing Unit, Food Technology Research Institute, and broad bean hulls (*Vicia faba*) were obtained from the Field Crops Res. Inst., Agric. Res. Center, Giza, Egypt. Soy and broad bean hulls were ground in Wiley mill into a fine powder.

Fresh strawberry fruits used for jam preparation were purchased from Local Market, Cairo, Egypt. Average total solids ranged between 7.5 – 8.5 %, pH value was 3.1 and moisture content was 92.4 % .

Methods :

Isolation of pectin

Pectin from soy and broad bean hulls were isolated by using 0.1 N HCl under refluxing at 90^o C for 45 min. with constant stirring . The extracts were cooled to room temperature and then centrifuged at 3000 rpm for 15 min. The supernatants were collected and dispersed in an equal volume of isopropanol . Pectin was precipitated by adjusting the pH value of the dispersion to 3.5 and allowed it to stand for 4.0 hr. The precipitate was collected , centrifuged , dispersed in 70 % isopropanol , stirred for 30 min. and centrifuged. The washing was repeated with 70 % isopropanol and then 100% isopropanol . The product was dispersed in 25- 30 ml deionized water, freeze dried and stored at room temperature according to the method reported by Kalapathy and Proctor (2001).

Physico – chemical characteristics of pectin.

Viscosity of pectin from both soy and broad bean hulls were determined according to Phatak et al., (1988) .Isolated pectin (2.0% w/v) was suspended in 0.1 M phosphate buffer (pH 7.0) and hydrated at 4.0^o C overnight before measuring viscosity (C Pa .s) at 25^o C by using Bookfield DV III Rheometer at 250 rpm test speed . Whereas , Shear

stress and molecular weight of both isolated pectin were calculated according to Phatak et al., (1988) and Christensen (1954).

Ash and pH value of both pectins were determined according to A.O.A.C (1990). Methoxyl content and equivalent weight were determined in soy and broad bean pectin according to Gree et al., (1958) and Rangana, (1977). Degree of esterification and anhydrogalacturonic acid of both pectins were determined by titration after acid washing of both pectins according to Ailam (1988). The natural sugars were determined calorimetrically using phenol sulphuric acid method as described by Smith et al., (1956).

Preparation of strawberry jam using isolated pectin

Strawberry jam was prepared in the laboratory at different levels (2.0, 4.0 and 6.0 g pectin / kg sugar) from soy and broad bean hulls pectin according to Guichard et al., (1991). Fresh strawberry and sugar (1.0 kg) both of them were mixed in a pan, then, heated and the mixture was stirred during heating, as the temperature of the mixture reached 60° C. the pectin was diluted in 30 ml water and added separately (2.0, 4.0 and 6.0 g levels concentration, respectively). Citric acid 2.0 g was added to the mixture. After addition of pectin to the mixture, pronounced thickness was reached. Heating was stopped at 105° C (220° F - 68° brix), where the jam was immediately transferred in jars. After cooling to room temperature, final pH value reached 3.1 ± 0.2 . The control jam was made from fresh strawberries and 4.0 g commercial citrus pectin per 1.0 kg sugar. Sensory evaluation of strawberry was conducted by ten panelists according to Guichard et al., (1991). The results were statistically analyzed by using L.S.D. test reported by Steel and Torria (1980).

RESULTS AND DISCUSSION

Physico – chemical characteristics of pectin.

The Physico- chemical characteristics of pectin prepared from soy and broad bean hulls were determined and the results are reported in Table (1).

Hydrochloric acid 0.1 N. was used for extracting pectin then with isopropanol solution and the reached pH value was adjusted to 3.5 which had significant effects on

pectin yield which reached 16 and 18% from soy and broad bean hull respectively. The pectin yield decreased with increasing the acid strength. These results are in agreement with Kalapathy and Proctor (2001).

Pectin extracted by 0.1 N HCl had lower ash contents being 1.80 and 1.28 % for soy and broad bean hulls compared to the control pectin. Ash content of pectin increased with increasing the strength of acid used for pectin extraction. This could be due to the effect of acidity to increase the solubility of indigenous minerals in the hull, with increasing the acid strength. The solubilized minerals would then be precipitated with pectin during alcohol precipitation (Kalapathy and Proctor, 2001). The pectin yield and ash content of the soy and broad bean hull products were compared to those of citrus pectin, indicating the potential of soy and broad bean hull as alternative sources for commercial production of pectin

Methoxyl content of pectin from soy and broad bean hulls were 8.07 and 8.13% respectively. Percentage of methoxyl groups refers to the quantity of methoxyl groups as an ester in the sample. The lowering of methoxyl content of pectin from soy and broad bean hull might have rendered the pectin more prone for demethylation during acid hydrolysis, resulting in the reduction of methoxyl groups. The methoxyl content of standard pectin used in the industry according to F.C.C (1981) is not less than 7%. Degrees of esterification (DE) referring to the ratio of carboxyl groups esterified with methanol to the free carboxyl groups. Degree of esterification for soy and broad bean pectin were 62.05 and 58.84 % respectively, which had indicated that the highest solubility of soy and broad bean pectin may be due to the presence of low levels of ester groups (Monsoor and Proctor, 2001). Moreover, anhydrogalactouronic acid content of soy and broad bean pectin were 78.07 and 79.6 % ,respectively. Galactouronic acid content is a very important character for pectin specification as described by F.C.C (1981), which had not less than 78%. Also, the molecular weight of soy and broad bean pectin hulls were 94.75×10^3 and 99.85×10^3 Dalton. The average molecular weight of pectin varies from 50×10^3 to 150×10^3 Dalton (Whistler and Be Miller, 1997).

Viscosity is considered as an important factor which plays an effective role in gel formation. The viscosity of soy and broad bean pectin was 134.2 and 145.13 Centipoise / sec, respectively. Moreover, shear stress was 79.28 and 86.95 Pascal / sec. for soy and broad bean pectin. From these results, it could be noticed that significant correlation existed between viscosity and shear stress. The higher viscosity is parallel with the higher shear stress. Monsoor and proctor(2001) found that the pectin at pH 3.5 is in a 50 % ionized state and which produces a strong gel network , due to a desirable balance between hydrophilic and hydrophobic character , which might have led to a good solubility in 50 % isopropanol.

Natural sugars of pectin isolated from soy and broad bean hulls were 7.35 and 6.72 %, respectively. Ayyed (1997) found that the total natural sugars in pectin were counted from 7 to less than 15%.

Application of pectin to prepare strawberry jam.

Soy and broad bean pectin were used at different levels being 2.0 , 4.0 and 6.0 g/kg sugar in laboratory manufacturing of strawberry jam. Jams were evaluated and the results are shown in Tables (2 and 3). The results show that highly acceptable strawberry jam was obtained when isolated pectin was added from soy and broad bean hull . Increasing the level of pectin induced some changes in color . The color of strawberry fruit is mainly due to anthocyanin pigments which are characterized with red and pink color during cooking. The available sugars and acid induce some changes in this pigment . Also , the sensory and ideal thickness of pectin consistency should not interfere with the flavor , taste and brightness of the final product into which it is added (Guichard et al., 1991). From these results, the sensory evaluations of soy and broad bean pectin used in manufacturing jam show that the best addition of pectin was 6.0 g/kg sugar from soy pectin followed by 4.0 and 6.0 g/kg sugar from broad bean pectin to give jam of good qualities.

The yield obtained from the preparation of strawberry jam using soy pectin at levels 2.0,4.0 and 6.0 g /kg sugar were 1.35 , 1.40 and 1.45 kg net weight respectively . Whereas , the net yield obtained from the addition of 2.0 , 4.0 and 6.0 g pectin from broad bean hull to prepare strawberry jam were 1.40 , 1.45 and 1.50 kg net weight respectively .

In conclusion , it is obvious that soy and broad bean hulls as low cost wastes could be used for pectin production. The characteristics of the isolated pectin were of good solubility, gelling power and high quality due to methoxyl content and anhydrogalactouronic acid which are not less than 7% and 78 %, respectively. The isolated pectin were used as gelling agent to produce strawberry jam of good qualities. Also , the best pectin addition was 6.0 g from soy pectin hull . Whereas, 4.0 and 6.0 g from broad bean pectin hull which gave the same effect.

It could be recommended that further researches would be necessary and of value ascertain this topic by using other fruits and vegetables.

REFERENCES

1. Allam, H.T. 1988. Studies on extraction of pectin from fruit waste. M.Sc. Thesis, Food Sci. and Techn. Faculty of Agric., Suez Canal Univ., Egypt.
2. A.O.A.C. 1990. Official Methods of Analysis of the Association of Official Analytical Chemists 15th Ed. Virginia 2001, USA .
3. Ayyed, K.M. 1997. Factors contributing to the gelling properties of pectin. The 3rd Alex. Conference of Food Sci. and Techn., Alex. March 1-3 , 1997
4. Barford, N.H. and Pederson, K. S . 1990. Determinating the setting temperature of high methoxyl pectin gel. Food Techn., 4 : 139 – 141.
5. Christensen, P.E. 1954. Methods of grading pectin in relation to the molecular weight (intrinsic viscosity) of pectin. Food Res . 19 : 163 – 167 .
6. F.C.C. 1981. Food Chemical Codex 3rd Ed., National Academy of Science, Washington, D.C., p.518.
7. Fernandez, M.L., Sun, D.M., Tosea, M.A. and Mc Namara, D.J. 1994. Citrus pectin and cholesterol interact to regulate hepatic cholesterol homeostasis and lipoprotein metabolism : A dose response study in Guinea pigs. Am.J. Clin. Nutr. 59 : 869- 872
8. Gree, M., Mc Comb, E.A. and Mc Cready, R.M. 1958. A method for characterization of pectin substances in some fruits and sugar beet marces. Food Res., 23 : 72.
9. Guichard, E., Issanchou, S., Descourvieres, A. and Etievant,p. 1991. Pectin concentration, molecular weight and degree of esterification influence of volatile composition and sensory characteristics of strawberry jam.J. Food Sci., 56 (6) 1621- 1627

10. Kalapathy, U. and Proctor, A. 2001. Effect of acid extraction and alcohol precipitation conditions on the yield and purity of soy hull pectin. *Food Chem.* 73, 393-396.
11. Monsoor, M.A. and Proctor, A. 2001. Preparation of functional properties of soy hull pectin. *J. J. Am. Oil Chem. Sec.*, 78 (7) 709 – 713.
12. Phatak, L., Chang, K.C. and Brown, G. 1988. Isolation and characterization of pectin in sugar beet pulp. *J. Food Sci.*, 53 (3) : 830- 833
13. Rangana, S. 1977. *Hand Book of Analysis and Quality Control for Fruit and Vegetable products*. 2nd Ed., Chapter 2, " Pectin", p. 31- 65. Mc Graw – Hill, pub . Co., Limited, Newdelhi.
14. Smith, F., Michel, O., Gilles, K.A., Hamilton, J. K. and Rebers, P.A. 1956. Colorimetric method for determination of sugar and related substances. *Analytical Chem.*, 28 (3) : 450 – 456.
15. Steel, G.D.R. and Torrie, J. H. 1980. *Principles and procedures of statistics. A biometrical approach*. 2nd Ed., Mc Grow- Hill, N.Y.
16. Thakur, B.R., Singh, R . K . and Handa , A . K . 1997. Chemistry and uses of pectin- a review. *Critical Reviews Food and Nutrition* , 37, 47 –73.
17. Voragen, A.G.J., Pilnik, W., Thibault, J.F., Axelos, M.A. V. and Renard, C.M.G.C. 1995. Pectins. In Stephan, A.M. Ed., *Food polysaccharides and their applications* (pp. 287- 339) .N.Y : Marcel Dekker.
18. Whistler, R.L. and Be Miller, J.N. 1997. Pectins. In Whistler, R.L. and Be Miller, J.N. *Carbohydrate chemistry for food scientists*, pp. 203- 210. Eagan press : St. Paul, M.N.

Table 1. Physico – chemical characteristics of pectin from soy and broad bean hulls.

Analysis	Soy bean pectin	Broad bean pectin	Commercial pectin**
Pectin yield g / 100 g*	16.0	18.0	-----
Ash content%	1.80	1.28	2.08
pH value of 1% suspension	3.0 ± 0.2	3.0 ± 0.2	3.0 ± 0.2
Methoxyl content %	8.07	8.13	9.86
Equivalent weight	561.78	583.67	615.21
Degree of esterification %	62.05	58.84	66.39
Anhydro galacturonic acid %	78.07	79.6	82.65
Molecular weight (Dalton)	94.75 × 10 ³	99.85 × 10 ³	102.55 × 10 ³
Viscosity (centipoise / sec)	134.2	145.13	160.02
Shear stress (pascal / sec)	79.28	86.95	96.60
Natural sugar %	7.35	6.72	7.67

* On dry weight basis

** Commercial pectin as control

Table 2. Sensory evaluation of strawberry jam prepared by using soy hull pectin at different levels

Addition gram	Texture 20	Color 20	Flavor 20	Taste 20	Brightness 20	Total score 100
Control *	17.1	18.1	17.6	18.7	19.3	90.8
Soy pectin 2.0	12.7	14.5	14.3	15.3	13.5	70.3
4.0	14.2	16.3	16.1	16.4	15.2	78.2
6.0	16.5	17.6	17.2	17.7	18.5	87.5
L.S.D. at 5%	1.412	0.588	0.2824	0.610	0.645	

*Control prepared by using 4.0 g commercial pectin to 1.0 kg sugar used.

Table 3. Sensory properties of strawberry jam prepared by using broad bean hull pectin at different levels

Addition gram	Texture 20	Color 20	Flavor 20	Taste 20	Brightness 20	Total score 100
Control *	17.1	18.1	17.6	18.7	19.3	90.8
Broad bean pectin						
2.0	15.9	16.8	14.3	15.5	16.7	79.2
4.0	16.1	17.4	16.0	17.7	18.1	85.7
6.0	16.9	17.9	16.6	18.2	18.7	88.3
L.S.D. at 5%	1.262	0.747	1.541	0.837	0.747	

*Control prepared by using 4.0 g commercial pectin to 1.0 kg sugar used.

إنتاج البكتين من قشور بعض البقوليات

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تم فصل البكتين من قشور فول الصويا والفول البلدى باستخدام حامض الأيدروكلوريك ٠,١ عيارى على درجة حرارة ٩٠م لمدة ٤٥ دقيقة. تم غسيل المترسب عدة مرات بواسطة كحول الأيزوبروبيل ٧٠% و ١٠٠% ثم جفف البكتين . قدرت الصفات الكيماوية والطبيعية للبكتين وأوضحت النتائج أن البكتين المتحصل عليه من قشور الصويا والفول البلدى كان مرتفع فى كل من اللزوجة shear stress وأيضا محتوى الميثوكسيل حيث ان قيمته أعلى من ٨% فى البكتين. توضح هذه النتائج أن البكتين المتحصل عليه مرتفع فى درجة اللزبان فى الماء والجلتة بينما درجة الاستره وحامض الالهيدروجلاكتيتورنك فى بكتين قشور الصويا والفول البلدى كانت (٦٢,٠٥ - ٥٨,٨٤%) و (٧٨,٠٧ - ٧٩,٦%) على التوالى . تدل النتائج على أن البكتين المنتج من قشور فول للصويا والبلدى ذات جودة عالية.

استخدم البكتين المتحصل عليه من قشور الصويا والفول البلدى منفصلاً فى تصنيع مربية الفرولة مع إضافته بمعدل ٢ و ٤ و ٦ جم/كجم سكر مضاف على التوالى . أوضح التقييم الحسى للمربى المصنعة أن نسبة الإضافة من بكتين قشور فول الصويا حتى ٦ جم /كجم سكر كان معدل التصافى للمربى ١,٤٥ كجم بينما كان معدل التصافى من نسبة إضافة بكتين ٤ و ٦ جم من بكتين قشور الفول البلدى لكل كجم سكر أعطت ١,٤٥ و ١,٥٠ كجم وزن صافى .

توضح هذه النتائج ان البكتين المحضر من قشور فول الصويا والفول البلدى يعطى نفس التأثير بينما الوزن الصافى من تحضير المربى المضاف اليها البكتين التجارى كان ١,٦ كجم وزن صافى وهذا يبين القيمة الاقتصادية للبكتين من مصادر مختلفة ومنها البكتين التجارى .