

Obtainment and characterization of pectin through the peel of passion fruit (*Passiflora edulis f. flavicarpa* Degener)

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Abstract

In this study the influence of the skin color (greenish-white, greenish-yellow and yellow) and of the extracting agent (HCl, H_3PO_4 , $H_3PO_4-(NaPO_3)_6$) on the pectin of the dry peel of passion fruit was analyzed. The content of pectin was determined through the method of acid hydrolysis under the following extracting conditions pH: 3.0, temperature: 90-95°C heating period: 90 minutes. The quality of the pectin was evaluated by means of the analysis of the humidity, ashes, equivalent weight, methoxyl, anhydrouronic acid, esterification, gelling period, relative viscosity and infrared spectrum. The maximum yield of pectin obtained was 18,45% when $H_3PO_4-(NaPO_3)_6$ was used as extracting agent; whereas in terms of quality the pectin was better when HCl was used as extracting agent, with a content of anhydrouronic acid and methoxyl of 78% and 9,9%, respectively. The peel of the passion fruit in the yellow state of ripeness showed the highest content of pectin, and the greenish-white state of ripeness showed the best gelling properties. The IR spectrometry confirmed that pectin had a high methoxyl. The analysis of the minerals showed the following results: calcium 0.10 to 0.15%, magnesium 0.05 to 0.08% and sodium 0.02 to 0.04%. The pectin of the peel of passion fruit does not have unusual characteristics that would indicate some potential commercial disadvantage.

Key words: passion fruit, pectin, extracting agent, skin color.

Introduction

Pectin is an excellent colloid that has the property of absorbing a huge quantity of water. It belongs to the group of polysaccharides and is found in most vegetables, mainly in fruits as for example oranges, grapefruit, lemon and limonzón (*citrus grandis*). Pectin is mainly concentrated in the primary wall and in the medium blade, being the mesenchyma and parenchyma particularly rich in this substance, hence having the role of intercellular cement (17, 23).

Pectin has an important role in the processing of food as an additive and source of dietary fiber. Gels of pectin are important to create or modify the texture of blends, jellies, sweets and dairy products low in fat. It is also used as an ingredient in pharmaceutical preparations as antidiarrheals, treatments for drug addiction, among others. Besides, this reduces the intolerance of glucose in diabetics and it even lowers the cholesterol level in the blood and of the lipo-protean fraction of low density (8).

For industrial purpose, the obtaining of pectin is mainly restricted to the peel of citric fruits, with approximately 25% of peptic substances and of apple husks, producing from 15 to 18% of pectin. Other sources of pectin include peels of mango, residues of sunflower, guava among others (9).

From the nutritional and toxicology point of view, pectin in general and specially the one found in

passion fruit as a food additive based in the content determinations of saccharose, total sugar, reductive sugar, brix grade, sodium, phosphorus, calcium, magnesium, among others; besides other important chemical characteristics, it has a legitimate and non limited use in the processing systems, therefore its administration is completely trustable (1).

On the other hand, the physical (gelification time) and chemical (methoxyl content, content of galacturonic acid, esterification grade and viscosity) properties in the molecule of pectin are in function of the nature of the plant, the ripening stage, and of the extraction methodology (16), establishing variations in the content and quality of pectin.

The cortex of the yellow passion fruit is a sub-product of the processing industries of juice, obtaining from 70 to 75% of residue material (11). The production statistics of yellow passion fruit in Zulia state shows a rate of 24.500 Ton/year, with an average yield in the national market of 35.000 kg/Ha/year (15). Nowadays, there is an accelerate growth in the crop of passion fruit, which tends to continue increasing due to the demand of fresh fruit for the internal consumption and the one existent by the industries of juices. The increased in the production of yellow passion fruit in the country and mainly in Zulia state, indicates an increased in the processing of the fruit for the

production of huge volumes of juice, and as a consequence, the accumulation of a higher quantity of residue material which is normally devoted as food for livestock.

With the previous mentioned, and considering that all the pectin that is being consumed in the country is imported, it is suggested as an

objective to evaluate the quantity and quality of pectin in the cortex of passion fruit according to the coloring stage (green-white, green-yellow and yellow) with the extraction agents HCl, H_3PO_4 and $H_3PO_4 \cdot (NaPO_3)_6$ and to quantify the content of minerals: calcium, magnesium and sodium.

Materials and methods

Raw matter. The raw matter used were fruits of passion fruit *Passiflora edulis* var. flavicarpa Degener (yellow), coming from the uniform plants in the agronomical handle of San Luis farm, located at the Km 22, via Santa Bárbara of Zulia, El Vigía, in the South of Maracaibo's Lake, Zulia state. Samples of 12 kg of fruits were taken by each coloring stage: green-white (V.B), green-yellow (V.A) and yellow (A.)

Preparation of the dry cortex of passion fruit. Pulp and seeds of passion fruits which were previously weighted, were taken out manually. The cortex of fruits was weighted and washed, later it was mashed in a blender with little quantity of water, and immediately recovering the mashed peel through a thick cotton fabric, extracting the higher quantity of water. The mashed cortex was put on a container with 4 L of distilled water at a temperature of 95-98°C for 15 min.; with the aim of inactivating the pectinase enzymes that hydrolyze the methyl esters groups, forming methanol and therefore, pectin of lower methoxyl;

also inactivating the polygalacturonase, which breaks the glycosides links between galacturonic molecules, despolimerizing the chain to shorter fractions, and finally getting to the monomer of the galacturonic acid.

The solid phase was washed several times until not detecting soluble solids through the determination of Brix grades. It was pressed manually and was submitted to a dry process at 60°C until reaching a constant weight; was weighted, pulverized and kept on a container closed hermetically (3).

Extraction of pectin through the dry cortex of passion fruit. Three extraction agents were used, the chlorhydric acid, phosphoric acid and a mix of phosphoric acid and hexametaphosphate of sodium (HCl, H_3PO_4 , $H_3PO_4 \cdot (NaPO_3)_6$). For the industrial obtaining of pectin, it is extracted with acids in heat, to dissociate the protopectin to the soluble pectin (18, 5).

The acidulous water of each extraction procedure was prepared with chlorhydric acid (HCl) (3), phosphoric acid (H_3PO_4) and phosphoric acid with 0.75% of

hexametaphosphate of sodium ($\text{H}_3\text{PO}_4\text{-(NaPO}_3)_6$) (2) until adjusting each one to a pH of 3.0 (in triplicate), once added the dry matter in a dry cortex-acidulous water relation, 1:16. The acidulous water was heated with the cortex, constantly moving it until reaching a temperature of approximately 90-95°C for 90 min.; then it was filtered, was squeezed manually and frozen rapidly to minimize the degradation of the pectin by the heat. Subsequently, the filtrate was centrifuged for 10 min. at 3000 r.p.m. 1.5 volumes of ethanol at 95% was added to the peptic solution for precipitating, later it was filtered on a cotton fabric, was washed and then, was submitted to a dry procedure at 60°C until obtaining a constant weight (12).

Characterization of raw pectin. The quality of pectin was determined by the humidity content

(10), ashes (10), equivalent weight (14), methoxyl (14), anhydrouronic acid (14), esterification grade (14), time of gelification (7), relative viscosity (7), spectrometry of infrared and analysis of calcium, magnesium and sodium minerals (17). At the same time, the commercial pectin was analyzed in order to do comparisons with the pectin under study.

Statistical analysis. The obtained results were analyzed using the statistical software SAS (20) using a completely randomized design, which consisted on a factorial arrangement of 3x3 for the variables coloring stage and extraction agent, respectively; with three replications by combination of treatments. The variance analysis was applied to the results and the comparison test of means by Tukey to the sources of variation that resulted significant.

Results and discussion

Yield of pectin. In table 1 values of the extracted pectin of the cortex of passion fruit are showed. It can be seen that the yield in the extraction of pectin incremented at the time that continued the ripening stage of the fruit when employing the saline extract $\text{H}_3\text{PO}_4\text{-(NaPO}_3)_6$. This value of pectin is superior to the one reported by Corona *et al.* (4) with a 13.60% and slightly inferior to the mentioned by Matsumoto *et al.* (13) with a 20%. These obtained results allow to understand that the ripening process caused an increment of soluble pectin in the dissolutions of the

kidnapped agent of the calcium ions, sodium hexametaphosphate, due to the variation in the grade of methyl, it means, the degraded pectin naturally caused by effects of ripening has a small proportion of esterified carboxyl groups, forming insoluble salts with the existent calcium ions, therefore, these are soluble in presence of kidnapper of calcium (22).

The humidity and ashes content are reported in table 2. It is observed that the extracted pectin of the coloring stage V.B. has the highest content of water than the rest of the studied coloring stages. However, all

Table 1. Yield in the obtaining of pectin in different coloring stages by different extracting agents.

Coloring stage	Pectin (%)		
	HCl	H ₃ PO ₄	H ₃ PO ₄ -(NaPO ₃) ₆
Green-white	14.06 ^{bcd} ± 1.90	15.92 ^{ab} ± 1.12	13.35 ^{bcd} ± 1.01
Green-yellow	12.44 ^{cd} ± 0.42	13.41 ^{bcd} ± 0.92	17.96 ^{abc} ± 1.53
Yellow	11.11 ^d ± 1.03	12.57 ^{cd} ± 0.90	18.45 ^a ± 2.19

X±D.E. a = 0.05. Means with different superscripts differ significantly.

the obtained values for this parameter are comparable to those reported by Corona *et al.* (4) and Tandon *et al.* (24), 11.82% and 10.85% respectively; and with the commercial pectin of 10.81%. These results show that at the time that emerges the development phase of the passion fruit, increases the quantity of water. However, in the ripening period, the humidity content reduces particularly in the cortex (21). In ashes, it is seen that the highest

values were found with hexametafosfato of sodium, being the highest the one obtained in the yellow ripening stage. This value is near to the 6.76% reported by Matsumoto *et al.* (13), but far away to the reported by Corona *et al.* (4) of 2.04%. These high quantities of ashes obtained for the use of sodium hexametafosfato, might be due to sodium provided by the salt, and to the chelate action of the saline compound

Table 2. Humidity and ashes analysis of the extracted pectin of the cortex of passion fruit in different coloring stages using several extracting.

Coloring stage	Humidity (%)		
	HCl	H ₃ PO ₄	H ₃ PO ₄ -(NaPO ₃) ₆
Green-white	10.54 ^{bc} ± 0.96	10.63 ^b ± 0.84	10.97 ^a ± 1.02
Green-yellow	10.32 ^d ± 1.20	10.33 ^d ± 0.88	10.69 ^b ± 1.22
Yellow	10.09 ^e ± 0.99	10.23 ^{cd} ± 0.79	10.43 ^{cd} ± 1.34
	Ashes (%)		
Green-white	3.02 ^g ± 0.09	3.10 ^g ± 2.01	6.28 ^c ± 1.11
Green-yellow	3.52 ^f ± 1.01	3.48 ^f ± 0.97	6.66 ^b ± 0.77
Yellow	3.75 ^e ± 0.89	3.97 ^d ± 0.98	6.90 ^a ± 0.99

X±D.E. a = 0.05. Means with the same superscripts do not differ significantly.

on calcium, that is joined to the carboxyl groups of uronic acid of pectin, being more evident in the yellow coloring stage. In table 3, are showed the determined parameters of quality of the pectin preparations. It is observed that the portion of pectin in the coloring stage V.B. had the highest methoxyl content when assaying it with HCl, decreasing toward the last ripening studied stage. These results are superior to those reported by Matsumoto *et al.* (13) and Corona *et al.* (4) of 8.0 and 7.3% respectively. These values allow to say that the methoxyl percentage reduces with the ripening of the fruit as well as by the extracting effect, due

to the rupture of the methyl esters. In relation to the esterification grade, it is seen that when dissolving the fractions of pectin with HCl during the ripening period, the highest values were obtained. On this matter, Corona *et al.* (4), as well as Matsumoto *et al.* (13), report for the dry cortex of passion fruit values of 71.65 and 73.21% respectively, while in the commercial pectin was found 66.50%.

All these pectin extracted from the chemical agent HCl presented an esterification grade very similar between them, which indicates that there is not an important damage in the pectin, because the lost of methoxyl, that were previously

Table 3. Evaluation of the quality of pectin extracted from the cortex of passion fruit in the coloring stage: green-white, green-yellow and yellow by different extracting.

Coloring stage	Content of methoxyl (%)		
	HCl	H ₃ PO ₄	H ₃ PO ₄ -(NaPO ₃) ₆
Green-white	9.90 ^a ± 0.78	8.90 ^b ± 0.95	8.02 ^c ± 1.64
Green-yellow	9.20 ^b ± 0.99	8.42 ^c ± 1.50	7.80 ^d ± 2.10
Yellow	8.62 ^c ± 1.02	7.00 ^e ± 0.87	6.40 ^f ± 1.88
	Esterification grade (%)		
Green-white	72.05 ^a ± 0.06	71.06 ^{ab} ± 0.10	66.50 ^{cd} ± 1.02
Green-yellow	70.58 ^{abc} ± 0.09	70.05 ^{ab} ± 1.00	65.12 ^{de} ± 1.52
Yellow	69.75 ^{bcd} ± 1.01	61.02 ^{ef} ± 2.35	60.55 ^f ± 0.09
	Content of anhydrouronic acid (%)		
Green-white	78.00 ^a ± 0.99	71.11 ^c ± 0.98	70.26 ^c ± 0.89
Green-yellow	74.02 ^b ± 1.24	68.63 ^d ± 1.54	68.24 ^d ± 1.40
Yellow	70.43 ^d ± 1.98	65.15 ^e ± 1.987	60.15 ^f ± 2.54

X±D.E. a = 0.05. Means with the same superscript do not differ significantly.

esterified, is not high. In relation to the content of anhydrouronic acid, the highest percentage is observed in the coloring stage V.B with the extracting agent HCl. The obtained results agree with the expressed by Matsumoto *et al.* (13) and Corona *et al.* (4), of 78.5% and 67.2% respectively; as well as the 68.90% found for the commercial pectin. In base to these results it can be inferred that the extracting agent HCl has a favorable role on the pectin, so the depolarizing action of these mineral acid in the pectin is less drastic. In table 4, the results of the determinations of equivalent weight, gelification time and relative viscosity are observed, comparing these with the reported values for passion fruit and mango of other authors. The forming power of the jelly of pectin in study and its viscosity, allows to say that the extracted pectin of the cortex of passion fruit, is a sub-product with ideal characteristics to be used in the food industry, due to it presented a fast gelation and a strength of desirable gel.

Analysis of calcium, magnesium and sodium. In table 5, concentrations of ions of calcium, magnesium and sodium are showed, presented in the fractions of extracted

pectin with acid. The average values of calcium in the different coloring stage that were studied show the highest concentrations compare to the other ions, besides, it is warned the increment tendency in the quantity of these ions, at the time that increases the ripening. These values are in the interval of the concentrations of calcium reported by Albersheim (1) of 0.11% in pulp of grapefruit and Ferreira (6) from 0.12 to 0.25% in peels of mango. These high percentages of calcium compare to other minerals might be due to the structural role that this metallic ion has, which keeps the integrity of the membranes of the mean blade, and cellular walls joining the groups of free carboxyl from the uronic acid of the pectin, in a pectin way (21).

Spectrometry of infrared. In figure 1 the spectrums of infrared (IR) obtained from samples of commercial pectin of citric (pattern of high methoxyl) are presented, and the vegetal product (pectin) obtained from the process of acid hydrolysis of the dry cortex of passion fruit.

Bands around of 1650 and 1750 cm^{-1} are indicators of groups of free and esterified carboxyl respectively; which are useful for the identification

Table 4. Comparison of the equivalent weight, time of gelification and relative viscosity of pectin.

Equivalent weight (g/eq)	Time of gelification (min.)	Relative viscosity (cp)	Source
1802.2	3:08	0.023	This reseach
1543.2	4:20	-	Corona <i>et al.</i> (4)
1063.5	4:38	0.016	Tandon <i>et al.</i> (24)

Table 5. Content of calcium, magnesium and sodium.

Variables	Coloring stage		
	Green-white	Green-yellow	Yellow
Calcium (%)	0.10	0.14	0.15
Magnesium (%)	0.05	0.06	0.08
Sodium (%)	0.02	0.04	0.04

of pectin with high or low methoxyl (19). These spectrum had similar characteristics, due to these presented a market lengthening in

the groups of esterified carboxyl (bands 1750 cm^{-1}), which indicates the presence of pectin of high methoxyl.

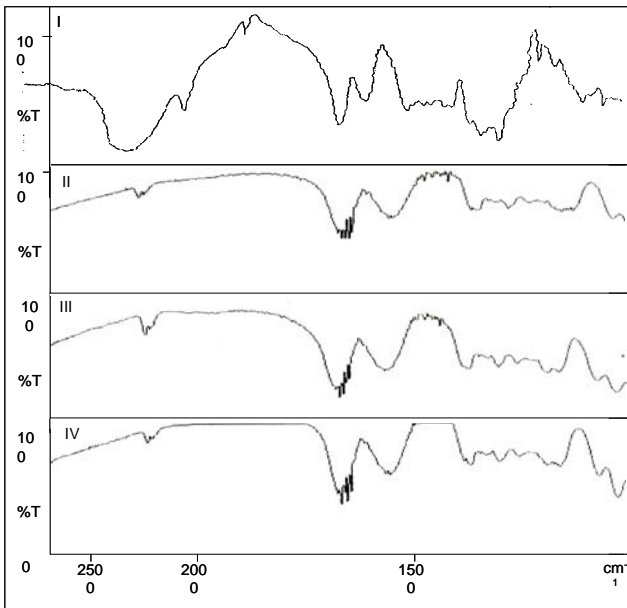


Figure 1. Spectrum of infrared of the extracted pectin from the cortex of passion fruit with HCl. (I) Commercial pectin of citric. (II) Pectin of cortex V.B. (III) Pectin of cortex V.A. (IV) Pectin of cortex (A).

Conclusions

The content and quality of pectin in the cortex of passion fruit differ according to the coloring stage or ripening of the fruit and of the extracting agent.

The cortex of the passion fruit in the yellow coloring stage (A.) had the highest content of pectin (18.45%), but the earliest ripening stage that was studied, the green-white (V.B), showed the pectin with the highest quality, indicating the methoxyl content (9.90%) and the esterification grade (72.05%), this coloring stage appeared to be the optimum for the industrial processing, due to the fast gelation and the strength of the produced gel.

Among the chemical agents used in the extraction, the sodium

hexametaphosphate favored the highest yield of pectin, while the chlorhydric acid resulted more convenient for the quality of it.

The comparisons done between the commercial pectin (Merk) and the pectin extracted from the cortex of the passion fruit, specially the one with the green while coloring stage, indicate that this last is on an acceptable quality rank in function of the content of methoxyl and of anhydrouronic acid.

The pectin extracted from the cortex of passion fruit is a sub-product with ideal characteristics to be destined to the food industries, because it presented a fast gelation and strength of desirable gel.

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