Characterization of Green-Extracted Orange Peel Pectin

Ngoc Lieu Le[1], Thuy Thi Thanh Tran[2], Linh Tran Khanh Vu[2]*
[1] *International University - Vietnam National University Ho Chi Minh City*
Quarter 6, Linh Trung Ward, Thu Duc District, Ho Chi Minh City, Viet Nam
[2] *Ho Chi Minh City University of Technology and Education*
No. 1 Vo Van Ngan Street, Linh Chieu Ward, Thu Duc District, Ho Chi Minh City, Viet Nam
Received: February 14, 2019; Accepted: June 22, 2020

Abstract

This study primarily investigated the possibility of extracting pectin from orange peels using water as a green solvent. The pectin powder was characterized. The results show that the extracted pectin had acceptable moisture content (8,21%) and high purity with the total anhydrouronic acid content of 82,7%. The degree of esterification and methoxyl content w 25,54% and 3,72%, respectively, which indicates the obtained pectin belongs to low methoxyl pectin able to form gel in the present of metal ions. The Fourier Transform Infrared (FTIR) spectrum re-verified its chemical structure. The work suggests that water can be an environmentally friendly solvent for pectin extraction.

Keywords: orange peel, pectin, FTIR, degree of esterification

1. Introduction

Orange processing industry is one of the most food waste sources. During the production of orange can, approximately 50% of the fruit weight can be canned, the rest is considered by-products, which consist of peel, core and frit [1]. These wastes may contain significant amounts of valuable compounds such as sugars, organic acids, essential oils, antioxidants, antimicrobial agents and fibers, and hence transforming them into commercial products can add values and reduce the environmental burden of waste disposal [2]. One of most important substances in citrus waste is pectin [3]. The world demand for pectin exceeds 30,000 tons annually and is predicted to increase 4-5% annum [4]. As a result, recovery of pectin from citrus waste has had commercial interests.

Pectin is a complex carbohydrate comprising 1,4-α-linked galacturonic acid and 1,2-linked rhamnose with side chains of either 1,4-linked β-D-galactose or 1,5-α-linked L-arabinose. Part of C-6 carboxyl units in the galacturonic acid chain are esterified with methoxyl groups or exist in the form of uronic acid salt [5]. Pectin has been demonstrated for its health benefits such as anti-inflammatory, anti-cancer, and hypocholesterolemic activities [6]. In addition, due to its good capability of gelling and thickening, it has been extensively used as a food additive to vary food texture and rheology. Another potential application of pectin in food industry is as a fat replacer in spreads, dressings, ice cream, emulsified meat products, cookies, etc. [6].

Although pectin exists in the cell walls of most plants, the natural sources for commercial pectin production are relatively limited. That is because the functional properties of pectin depend on molecular weight and degree of methoxylation, and these parameters can vary with material sources and the conditions for pectin extraction [7]. Currently, apple pomace and citrus are the main sources for commercial pectin production. In addition, beetroot and sunflower head residue have also been considered for pectin extraction [8]. Generally, conventional method for pectin production comprises of two main steps, extraction of pectin using mineral acids and subsequently recycle by precipitation with ethanol [9]. However, acid extraction method may cause increased production cost and problems for the environment. Hence, extraction of pectin without mineral acid could become a more environmentally friendly process [10].

In this study, orange peels in Vietnam were considered as material for pectin production. Water was used as a green solvent for pectin extraction. The chemical properties and characteristics of the obtained pectin powder were then verified.

[*] Corresponding author: Tel.: (+84) 966.955.46
Email: linhvtk@hcmute.edu.vn

2. Materials and Methods

2.1. Materials

Orange peels were collected from juice producers in Ho Chi Minh City and transferred to the laboratory for experiments. After removing the outer skin, the peels were washed and dried at 50°C until reaching 10-15% moisture content. The dried peels were then ground and sieved with a mesh size of 0.6 - 1.5mm. The peel powders were kept in zip bags at room temperature until further analyses.

2.2. Pectin extraction

The pectin extraction procedure was carried out according to the report of Hosseini et al. [10]. The peel powder was dissolved in distilled water at the ratio of 25:1 (v/w) and heated up at 95°C for 90 min using a water bath. The mixture was then filtered by a cheese cloth and centrifuged at 4000 rpm for 15 min. After that, the supernatant was precipitated by ethanol at the ratio of 1:2 (v/v) for 30 min and then cooled to 7°C for 20 h. The precipitate was filtered by a filter paper and washed twice with ethanol. Subsequently, the obtained precipitate was dried at 50°C in a hot air oven until all moisture was removed. The samples were then cooled and stored in small plastic sample bags until further analyses.

2.3. The characterization of the extracted pectin

The moisture and ash contents of the pectin powder were analyzed according to AOAC with the numbers of 934.01 and 930.30, respectively. The equivalent weight, total anhydrouronic acid content (AUA) and methoxyl content (MeO) were determined using the methods described by Azad et al. [11]. The degree of esterification (DE) was then calculated on the basis of methoxyl and AUA contents [11]. The yield of pectin was determined according to process given by Azad et al. [11], which was calculated by the weight of obtained pectin over the weight of used sample in percentage. All the results were the average of triplicate and were expressed as mean values with standard deviations.

The chemical structure of the extracted pectin was analyzed using a Fourier Transform Infrared (FTIR) system (FTIR-8400S, Shimadzu, Japan) at the Research and Development Center for Radiation Technology (VINAGAMMA). The sample was scanned in the range of 400 – 4000 cm^{-1} with a resolution of 4 cm^{-1}. The obtained spectrum was analyzed using the software SigmaPlot (Version 10.0).

3. Results and Discussion

3.1. Yield, moisture and ash contents

Table 1 lists the yield, moisture and ash contents of the extracted pectin. It can be seen that the extraction yield was 15.79%, which was nearly equal to the one obtained by Hosseini et al. [10] (18.35%) for pectin extraction from orange peels using distilled water. The obtained extraction yield was also comparable or higher than the yields of pectin extracted from other sources such as ambarella (16%) [12], mango peel (4.6% to 18.5%), bael fruit (5% to 17 %) [13] and passion fruit (7.5%) [14], but lower than those from golden apple (22%) [15] and beet pulp (22.4%) [16].

As shown in Table 1, the moisture content of the extracted pectin was 8.21 ± 0.02%. It should be noted that the obtained moisture content still meets the criteria for ambient storage (<12%) [17]. The ash content of the extracted pectin was 0.57 ± 0.02 %, which was similar to the one reported by Mesbahi et al. [16] for pectin from citrus fruits (0.55%) and lower than that from beet pulp (4.91%). According to Ismail et al. [18], low ash content in pectin powder is desirable for good gel formation and should be lower than 10%. Therefore, the extracted pectin in this study had acceptable value of ash content.

Table 1. The yield, moisture and ash contents of the extracted pectin

Parameters	Value
Moisture (%)	8.21 ± 0.02
Ash (%)	0.57 ± 0.02
Yield (%) (based on dry solid)	15.79 ± 0.11

3.2. Characterization of the extracted pectin

Table 2 indicates the equivalent weight, total anhydrouronic acid content (AUA), methoxyl content (MeO) and degree of esterification (DE) of the extracted pectin. The equivalent weight was 285.7 ± 3.92, which was similar to the value reported for lemon pectin (254) [19] but lower than those reported for the pectin from apple pomace (833 – 1666) [20], dragon fruit peel (476 – 714) [18] and cocoa husks (511 – 645) [21]. The value of equivalent weight was used in the calculation of AUA and DE.

The degree of esterification (DE), also known as degree of methoxylation, is generally used to classify pectin including low-methoxyl pectin (LMP, DE < 50%) and high-methoxyl pectin (HMP, DE > 50%). The DE value is directly related to the gelling mechanism: HMP forms gel in an acidic medium and at sugar concentrations above 55% whereas LMP forms gel with the aid of metal cations (commonly calcium ion) [10]. This also leads to

different applications of LMP and HMP in which the former is used in jams and marmalades while the latter is used in products with high sugar content (55 – 65 wt%) [22]. Table 2 shows that the DE of the extracted pectin was 25.54 ± 0.09%, which was similar to the value reported for pectin extracted from orange peel by distilled water (23%) [9], but lower than those from lemon pomace (33 - 79%) [11] and dragon fruit peel (31- 47%) [18] using conventional methods. This implies that DE values depend on material sources and extraction method. The extracted pectin in this study can be classified as having low degree of esterification (LMP).

Another value characterizing gel-forming mechanism of pectin is methoxyl content (MeO). It varies in the range of 0.2 to 12% depending on used materials and extraction procedures [19]. Table 2 displays that the methoxyl content of the extracted pectin was 3.72 ± 0.03% which was lower than those from lemon pomace (4.24 – 10.25%) [11], comparable with those from dragon fruit peel (2.98 – 4.34%) [18] and higher than that from lemon peel using citric acid for extraction (2.3%) [19]. This result reconfirmed the low methoxyl property of the extracted pectin.

On the other hand, the AUA content indicates the purity of pectin, which should not be less than 65% [23]. The AUA content of the pectin extracted from orange peel using distilled water in this study was 82.70 ± 1.15 %, which was comparable to the value reported by Hosseini et al. [10] for the pectin extracted from similar material and solvent (84.5%), but higher than those reported for the pectin extracted from orange peel using microwave (71%) [24], lemon pomace (73%) [11], dragon fruit peel (45 – 52%) [18]. This result suggested that the obtained pectin had high purity.

Table 2. Chemical characteristics of the extracted pectin

Parameters	Value
Equivalent weight	285.7 ± 3.92
MeO content (%)	3.72 ± 0.03
AUA content (%)	82.70 ± 1.55
DE (%)	25.54 ± 0.50

3.3. FTIR spectrum of the extracted pectin

To verify the chemical structure of the extracted pectin, FTIR was used for analysis in the range of 400 – 4000 cm^{-1} as shown in Figure 1. The broad and strong peak between 3000 and 3700 cm^{-1} is assigned to O–H stretching absorption which is attributed to the vibration of inter- and intramolecular hydrogen bonds of galacturonic acid units in the pectin structure [25]. The peak at 2935.2 cm^{-1} corresponds to C–H vibrations, which include the stretching and bending vibrations of CH, CH_2, and CH_3 groups. The peak at 1750 cm^{-1} is due to stretching vibrations of ester carbonyl C=O groups (COOH and $COOCH_3$), which is important in identifying and quantifying pectin. Carboxylate groups in pectin display two bands, an asymmetrical stretching band at 1629 cm^{-1} and a weaker symmetric stretching band at 1409cm^{-1}, where the latter corresponds to the characteristic of polygalacturonic acid in pectin samples [25]. The absorption bands in the range of 1100 – 1200 cm^{-1} indicate ether (R-O-R) bonds and C-C rings in pectin. In addition, the absorption pattern between 1200 and 800 cm^{-1} is referred as the "finger-print" which is unique to a compound (i.e. pectin) but usually hard to interpret. In summary, the spectrum of the extracted powder is similar with those reported for pectin which reconfirms its identification.

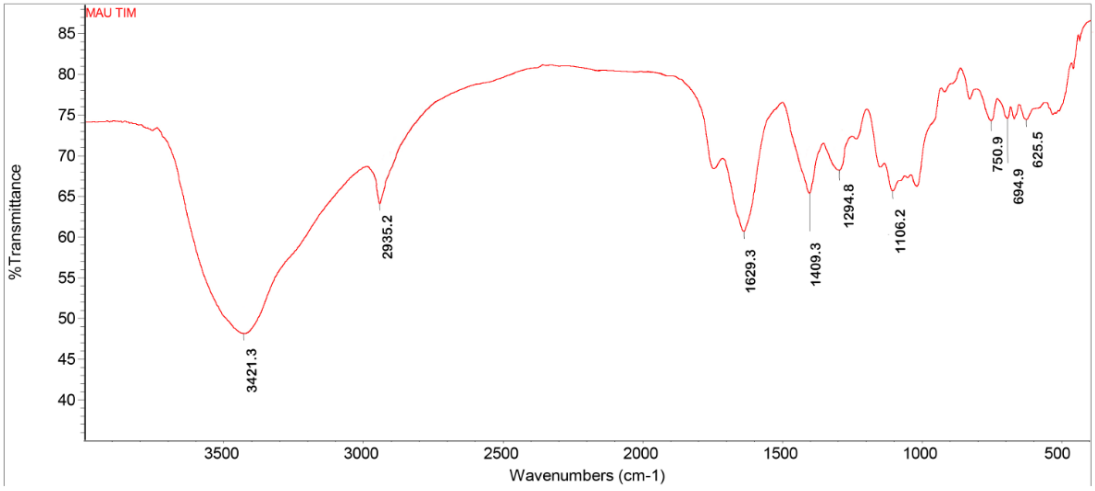

Fig. 1. FTIR spectrum of the extracted pectin.

4. Conclusion

In this work, pectin powder was extracted from orange peel by using water. The pectin powder was characterized. The obtained pectin had low equivalent weight but high purity. It had low methoxyl content and degree of esterification, suggesting gel formation in the presence of metal cations. The FTIR analysis re-verified the chemical structure of the extracted pectin. This study indicates that water is also a suitable solvent for pectin extraction which is environmentally friendly. Further investigation should be done on optimization of extraction conditions to obtain the highest yield of pectin.

References

[1] Prihatin A, Shiguo C, Xingqian Y: Pectin-enriched material from mandarin orange byproducts as a potential fat replacer in cookies. International Journal on Advanced Science, Engineering and Information Technology (2015) 5:31-35.

[2] Martínez R, Torres P, Meneses MA, Figueroa JG, Pérez-Álvarez JA, Viuda-Martos M: Chemical, technological and in vitro antioxidant properties of mango, guava, pineapple and passion fruit dietary fibre concentrate. Food Chemistry (2012) 135:1520-1526.

[3] Cho CW, Lee DY, Kim CW: Concentration and purification of soluble pectin from mandarin peels using crossflow microfiltration system. Carbohydrate Polymers (2003) 54:21-26.

[4] Yeoh S, Shi J, Langrish T: Comparisons between different techniques for water-based extraction of pectin from orange peels. Desalination (2008) 218:229-237.

[5] Ciriminna R, Chavarría-Hernández N, Inés Rodríguez Hernández A, Pagliaro M: Pectin: A new perspective from the biorefinery standpoint. Biofuels, Bioproducts and Biorefining (2015) 9:368-377.

[6] Lim J, Ko S, Lee S: Use of Yuja (Citrus junos) pectin as a fat replacer in baked foods. Food Science and Biotechnology (2014) 23:1837-1841.

[7] Thakur BR, Singh RK, Handa AK, Rao M: Chemistry and uses of pectin—a review. Critical Reviews in Food Science & Nutrition (1997) 37:47-73.

[8] Miyamoto A, Chang K: Extraction and physicochemical characterization of pectin from sunflower head residues. Journal of Food Science (1992) 57:1439-1443.

[9] Ma S, Yu SJ, Zheng XL, Wang XX, Bao QD, Guo XM: Extraction, characterization and spontaneous emulsifying properties of pectin from sugar beet pulp. Carbohydrate polymers (2013) 98:750-753.

[10] Hosseini SS, Khodaiyan F, Yarmand MS: Aqueous extraction of pectin from sour orange peel and its preliminary physicochemical properties. International journal of biological macromolecules (2016) 82:920-926.

[11] Azad A, Ali M, Akter MS, Rahman MJ, Ahmed M: Isolation and characterization of pectin extracted from lemon pomace during ripening. Journal of Food and Nutrition Sciences (2014) 2:30-35.

[12] Koubala B, Mbome L, Kansci G, Mbiapo FT, Crepeau MJ, Thibault JF, Ralet MC: Physicochemical properties of pectins from ambarella peels (Spondias cytherea) obtained using different extraction conditions. Food Chemistry (2008) 106:1202-1207.

[13] Maskey B, Dhakal D, Pradhananga M, Shrestha NK: Extraction and process optimization of bael fruit pectin. Food Science & Nutrition (2018) 6:1927-1932.

[14] Yapo BM: Pectin quantity, composition and physicochemical behaviour as influenced by the purification process. Food Research International (2009) 42:1197-1202.

[15] Rha HJ, Bae IY, Lee S, Yoo S-H, Chang P-S, Lee HG: Enhancement of anti-radical activity of pectin from apple pomace by hydroxamation. Food Hydrocolloids (2011) 25:545-548.

[16] Mesbahi G, Jamalian J, Farahnaky A: A comparative study on functional properties of beet and citrus pectins in food systems. Food Hydrocolloids (2005) 19:731-738.

[17] Ismail NSM, Ramli N, Hani NM, Meon Z: Extraction and characterization of pectin from dragon fruit (Hylocereus polyrhizus) using various extraction conditions. Sains Malaysiana (2012) 41:41-45.

[18] JECFA F: Pectin, 71st JECFA. Monograph 2009, 7:1-5.

[19] Kanmani P, Dhivya E, Aravind J, Kumaresan K: Extraction and analysis of pectin from citrus peels: augmenting the yield from citrus limon using statistical experimental design. Iranica Journal of Energy & Environment (2014) 5:303-312.

[20] Kumar A, Chauhan GS: Extraction and characterization of pectin from apple pomace and its evaluation as lipase (steapsin) inhibitor. Carbohydrate Polymers (2010) 82:454-459.

[21] Ramli N: Effect of ammonium oxalate and acetic acid at several extraction time and pH on some physicochemical properties of pectin from cocoa husks (Theobroma cacao). African Journal of Food Science (2011) 5:790-798.

[22] Pilnik W, Voragen A: Gelling agents (pectins) from plants for the food industry. Advances in plant cell biochemistry and biotechnology (USA) 1992.

[23] Codex FC: Food chemicals codex. Washington, DC: National Academies Press; 1996.

[24] Hosseini SS, Khodaiyan F, Yarmand MS: Optimization of microwave assisted extraction of pectin from sour orange peel and its physicochemical properties. Carbohydrate polymers (2016) 140:59-65.

[25] Chen Y, Zhang JG, Sun HJ, Wei ZJ: Pectin from Abelmoschus esculentus: Optimization of extraction and rheological properties. International journal of biological macromolecules (2014) 70:498-505.