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PECTINS FROM DRAGON FRUIT (*Hylocereus polyrhizus*) PEEL

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ABSTRACT

The extraction of pectin from a by-product of dragon fruit processing, was identified as an alternative source for commercial pectin. In this work, dried alcohol-insoluble residues (AIR) of dragon fruit peels were treated separately with ammonium oxalate/oxalic acid 0.25 %, pH 4.6, 85ºC; HCl 0.03 M, pH 1.5, 85ºC; and deionised water, 75ºC. The pectin obtained from these methods were compared in term of yield, color, gelling characteristic and chemical structure. The highest yield for the extracted pectin from dragon fruit peels was 20.1 % (dry weight basis) by ammonium oxalate/oxalic acid extraction, contained 11.2 % moisture and 6.9 % ash. Extraction by deionised water yielded 15.4 % pectin, 11.3 % moisture and 11.6 % ash. Whereas, the acid extraction gave the lowest yield 15 %), 11.1 % moisture and 12 % ash. The amount of pectin from all extraction conditions were comparable to pectin obtained from commercial apple (12 %) or citrus (25 %). Gel hardness test was performed for gelling properties measurement. The Fourier Transform Infrared Spectroscopy (FTIR) was useful in the identifying dragon fruit pectins. Different conditions used in the extraction do not show a difference in the pectin structure. With a good recovery yield and gelling properties, ammonium oxalate-extracted dragon fruit pectin present good characteristics to be exploited industrially as food additive.

ABSTRAK

Pengekstrakan pektin daripada sisa pemprosesan buah naga telah dikenal pasti sebagai sumber alternatif bagi pektin komersial. Dalam kajian ini, pepejal tak larut alkohol (AIR) kering daripada kulit buah naga diperlakukan secara berasingan dengan ammonium oksalat/asid oksalik 0.25 %, pH 4.6, 85ºC; HCl 0.03 M, pH 1.5, 85ºC; dan air ternyahion, 75ºC. Pektin yang diperolehi melalui kaedah ini dibandingkan dari segi hasil, warna, ciri penggelan dan struktur kimia. Hasil pektin tertinggi daripada kulit buah naga ialah 20.1 % (berat kering) menggunakan ekstraksi ammonium oksalat/asid oksalik, mengandungi 11.2 % air dan 6.9 % abu. Ekstraksi menggunakan air ternyahion menghasilkan 15.4 % pektin, 11.3 % air dan 11.6 % abu. Manakala, ekstraksi asid memberikan hasil yang terendah (15 %), 11.1 % air dan 12 % abu. Pektin yang terhasil daripada semua keadaan pengekstrakan adalah setanding dengan pektin epal (12 %) atau sitrus (25 %) komersial. Ujian kekerasan gel dilakukan bagi mengukur sifat penggelan. Spektroskopi inframerah (FTIR) amat berguna bagi mengenal pasti pektin buah naga. Keadaan berbeza yang digunakan dalam pengekstrakan tidak menunjukkan perbezaan dalam struktur pektin. Dengan perolehan hasil serta ciri penggelan yang baik, pektin buah naga hasil pengekstrakan menggunakan ammonium oksalat/asid oksalik mempunyai ciri yang baik untuk diterokai secara industri sebagai bahan tambah makanan.

Key words: Dragon fruit; Pectin; Alcohol insoluble residue; Extraction conditions

INTRODUCTION

Dragon fruit (*Hylocereus polyrhizus)* or red pitaya is a tropical fruit belonging to the cactus family, Cactaceae. Dragon fruit is also called 'buah naga' or 'buah mata naga' in Malaysia and has a quite pleasant taste. The peels of the fruit are virtually treated as waste part during the peeling process in the juice industry. These peels will create an environmental problem if it is not properly handled. Therefore, the research was conducted to utilize these waste materials as a source of pectin.

Pectin is the methylated ester of polygalacturonic acid that contains 1,4-linked α-D-galacturonic acid residues (Levigne *et al.*, 2002). It is commonly found in the cell walls and the middle lamellae of higher plants. Pectin is a gelling agent in jams and jellies that has been widely applied in food industry as a thickener, an emulsifier, a texturizer and stabilizer.

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Pectin can be obtained from many sources with variation of yield percentages. Commercial pectins are mostly extracted from citrus peels and apple pomace by using acid extraction method with the yield about 25 and 12 % pectin, respectively (Kalapathy & Proctor, 2001). Other sources of pectin include sugar beet, sunflower head residue, cocoa husk, soy hull, mango peels and banana peels. However, there is still no research done on the extraction of pectin from dragon fruit.

The extraction of pectin basically involves the aqueous extraction of pectin from the raw material (plant), isolation of the extracted pectin, purification and followed by drying process. The yield of pectin usually depend on the extraction conditions such as temperature, the extraction time, the pH, and also the types of extraction solvent used (Yeoh *et al.*, 2008). Before the extraction begins, alcohol insoluble residue (AIR) is prepared to remove low molecular weight compounds, including any trace of free galacturonic acid (Emaga *et al.*, 2008). Pectin can be divided into two types based on the degree of esterification (DE) of the pectin: high methoxyl pectin (DE >50 %) and low methoxyl pectin (DE<50 %).

The objective of this study were (i) to evaluate the impact of different extraction conditions on the yield of dragon fruit pectin, (ii) to measure the gelling properties of the extracted pectin and (iii) to define the structure of pectin using infrared spectroscopy (FTIR).

MATERIALS AND METHODS

Sampling and sample treatment

The dragon fruit peels were obtained from MARDI. The peels of the fruits were removed and chopped into pieces of 1 cm^2 using a stainless steel knife and dried in an air convection oven at 50°C. The dried peels were then grounded in the laboratory dry blender. Alcohol insoluble residue (AIR) was prepared by treating the dried peels four times with isopropanol (85 vol. %) at 70°C for 20 min following the procedure that modified from (Koubala *et al.*, 2008).

Extraction procedure

Three different extraction conditions by Koubala *et al*. (2008b) were applied and compared to find the best condition to recover pectin from dragon fruit. Solvents used were ammonium oxalate (0.25 %) pH 4.6 $\pm$ 0.01 (using oxalic acid) at 85°C for 1 h, Hydrochloric acid (0.03 M; pH 1.49 $\pm$ 0.02) at 85°C for 1 h, and deionised water at 75°C for 1 h. About 50 g of AIR was stirred with 800 ml of each of the above extracting solutions. The extracts were separated from the AIR residue by filtering through a nylon cloth, and the pectin was coagulated with isopropanol. Pectin coagulated was then purified by washing several times with isopropanol and acetone before drying in an oven at 40°C for a few hours to constant weight and then grounded finely. The yield obtained was reported as % yield (g dried pectin per g dried peels). Each of the extraction conditions were carried out in triplicate. This pectin was used for further analysis.

Ash and Moisture content

Ash content was determined by weighing 1 g of pectin in a tared crucible and ignited slowly, then heated in muffle furnace at 600°C for 4 hours. The residue was cooled in desiccators and weighed to constant weight (AOAC 1980). In determining the moisture content, 1 g of pectin was weighed and dried at 100°C for 4 hours to constant weight (AOAC 1980). The test was performed in triplicate and the average values were reported.

Color measurement

The color parameters of pectin samples were determined using Chroma meter DR-400 Minolta by measuring L*, a* and b* values in CIE system. Before each measurement the colorimeter was standardized with the reference white plates provided with the equipment. The test was performed in triplicate and the average values were reported.

Pectin gels preparation

Gels were prepared according to method that modified from Fu and Rao (2001). Dispersion containing 1 % pectin (extracted with ammonium oxalate and deionised water; and commercial citrus pectin), 10 % sucrose and 0.15 % calcium (Ca^{2+}). Plain gels (i.e. without sucrose) containing only Ca^{2+} were also studied. Weighted amount of pectin and sucrose were dissolved in solution of 0.1 N NaCl with stirring. The total volume of gel prepared in this study was 40 ml. The pH adjusted to 4 using either 0.1 N NaOH or 0.1 N HCl. Calcium was added at 60°C while stirring for 10 min and left at room temperature for 24 hours before being tested for gel hardness.

Gel hardness measurement

Hardness was measured with 0.5-inch radius cylindrical probe (P/0.5R) using Texture Analyzer (EZtest/AGS-H) SHIMADZU. Gel hardness is defined as the force required for penetration of standard probe into the gel (Ebrahim *et al.*, 2007). The sample container (diameter of 2 inches) was placed centrally under the standard probe and penetration test was started. The probe penetrates

into the gel to a depth of 4mm with a cell loading of 5 kg (Norziah *et al*., 2000). All analyses were performed three replicate samples.

Structural analyses

FTIR spectra was used to obtain information on chemical structure. The fourier transform infrared data was obtained using Perkin Elmer, GX spectrum model with wavelength ranging from 4000-400 cm^{-1}.

RESULTS AND DISCUSSION

Extraction yields

The yield of the extracted dragon fruit pectin varied from 15 to 20.1 % of the dry weight of peels, depending on the extraction condition used. A summary of results is shown in Table 1. The highest yield of pectin was 20.1 % obtained using ammonium oxalate/oxalic acid extraction. Ammonium oxalate is a calcium chelating agent that helps the pectin to be released from the cell wall (Yeoh *et al.*, 2008). The extraction with deionised water yielded 15.4 % pectin. Whereas, extraction with 0.03 M HCl resulted in the lowest yield (15 %) pectin. In comparison, yields for ambarella peels (10-13 %) that was obtained using the same conditions was lower under water extraction (Koubala *et al.*, 2008b). The pectin obtained in this study was more than 10 % which make it feasible for commercial use.

Ash and moisture content

The moisture content in all samples (Table 1) was quite high (11.1-11.3 %) compared with the pectin from soy hull and galgal (*Citrus pseudolimon* Tan) peels which is 6 -7 % and 6 – 8 % respectively (Attri & Maini, 1996; Kalapathy & Proctor 2001). The maximum limit of moisture content in pectin should not more than 12 % (FAO JECFA 2009). Pectin should have as lower as possible moisture content for safe storage. From table 1, it was found that the pectin extracted by ammonium oxalate gave the lowest ash content which make it more favourable for gel formation. However, ash content in acid and water extracted pectin was significantly higher. In other study, soy hull pectin contains 1.2 to 3.2 % of ash (Kalapathy & Proctor, 2001). The maximum limit of ash content for quality criteria is 10% (Mohamadzadeh *et al*., 2010)

Color values of pectin

Color values and their standard deviation values of powders of dragon fruit pectin that extracted by various extraction conditions and commercial apple pectin are presented in Table 2. Commercial apple pectin had highest lightness (L*) and yellowness (b*) compared to dragon fruit pectin. Water extracted pectin had highest redness (a*). There were shows significant differences for all samples tested in this study. It was suggested that ammonium oxalate extracted pectin had similar color to that of apple pectin with high lightness and yellowness. While water extracted pectin had the most negative effect on appearance among the extractions method.

Gelling properties

Evaluations show that addition of sucrose has the positive effect on pectin gel properties (Figure 1). Pectin gel that added with sucrose has higher hardness value than plain pectin gel. Theoretically, addition of sugar will decrease hydration of the pectin by competing for water. Thus, decrease the ability of pectin to stay in dispersed state. When cooled, these unstable dispersing of less hydrated pectin forms gel (Oakenfull, 1991).

From the result, ammonium oxalate extracted pectin has higher hardness value than water

Table 1. Extraction yields (% dry peels) for dragon fruit (*hylocereus polyrhizus)*[a]

	Ammonium oxalate/oxalic acid 0.25%	0.03M HCl	Deionised water
Yield of pectin, %	20.14+0.43[a]	14.96+0.36[b]	15.37+0.44[b]
Moisture, %	11.19+0.25[a]	11.13+0.7[a]	11.33+0.69[a]
Ash content, %	6.88+0.42[b]	11.95+1.55[a]	11.55+0.13[a]

[a-b] Mean value from triplicate measurement+standard deviation. Value with different superscripts are significantly different (p<0.05).
*Kalapathy & Proctor (2001)

Table 2. Color parameter of L*, a* and b* of dragon fruit pectin extracted by ammonium oxalate/oxalic acid, HCl and deionised water compared with commercial apple pectin.

Pectin samples	L*	a*	b*
Ammonium oxalate/oxalic acid 0.25%	65.56 ± 0.71[c]	5.16 ± 0.09[b]	15.50 ± 0.04[b]
0.03 M HCl	74.81 ± 0.08[b]	3.02 ± 0.06[d]	7.74 ± 0.12[c]
Deionised water	53.83 ± 0.63[d]	11.04 ± 0.24[a]	4.16 ± 0.38[d]
Apple pectin	83.95 ± 0.53[a]	4.01 ± 0.05[c]	19.81 ± 0.29[a]

[a-b] Mean value from triplicate measurement+standard deviation. Value with different superscripts are significantly different (p<0.05).

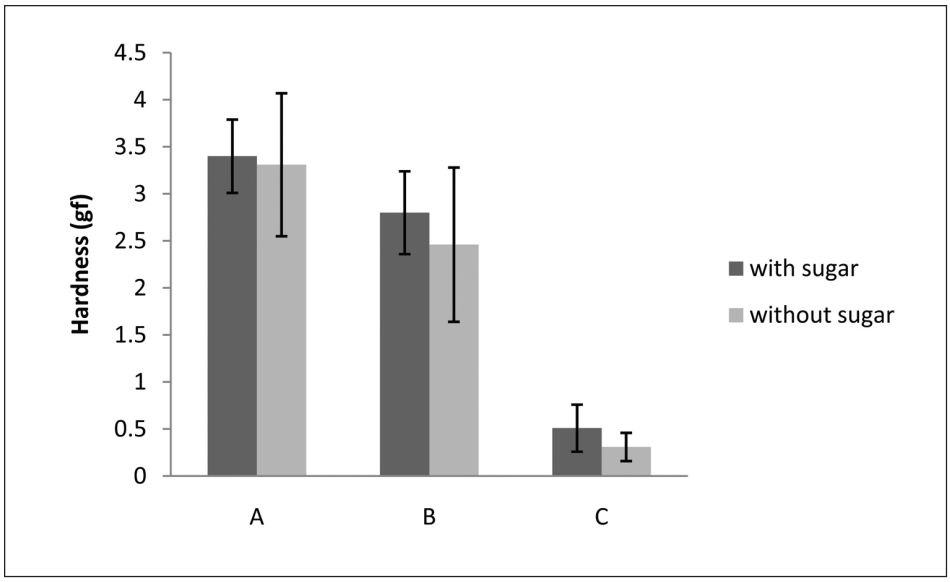

Fig. 1. Effect of extraction condition on gel hardness. (A)- Commercial citrus pectin, (B)- Ammonium oxalate extracted pectin, (C)-Water extracted pectin.

extracted pectin that only form a sol rather than gel. It also shows that the gelling quality of the ammonium oxalate extracted pectin was comparable to that of commercial citrus pectin. HCl extracted pectin was not tested for hardness because there was no gelation was observed. This result was supported by Koubala *et al*. (2009). In his work, no gelation was observed with HCl-extracted ambarella pectin due to low intrinsic viscosity and also low content of galacturonic acid. The weak gelation of water and HCl extracted pectin observed in this work could be due to the high ash content in the pectin.

Dragon fruit pectin structure

The bands in the region between 1000-2000 cm^{-1} of FTIR spectra are typically used to identify the major chemical groups in the pectins (Kalapathy & Proctor, 2001). It determines the functional groups of the pectin and provides structural information. The infrared spectra of pectin produced by extraction with ammonium oxalate, 0.03 M HCl and deionised water is compared to commercial apple pectin in Figure 2. The carbonyl bands at 1630-1650 and 1740-1760 cm^{-1} indicates the free and esterified carboxyl groups, respectively (Gnanasambandam &

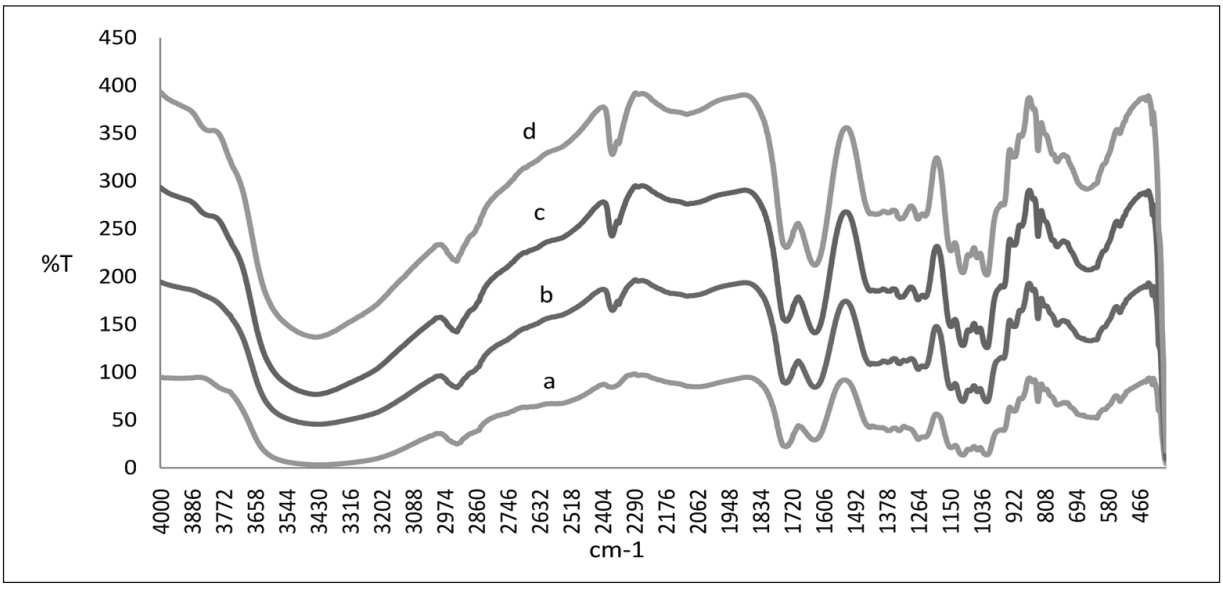

Fig. 2. FTIR spectra of (a) apple pectin from Sigma Inc., (b) dragon fruit pectin produced by ammonium oxalate extraction, (c) dragon fruit pectin produced by 0.03M HCl extraction, (d) dragon fruit pectin produced by deionised water extraction.

Proctor, 1999). The increase in (DE) values will also increase the intensities and band area of esterified carboxyl groups. It could be used to compare the different types of pectin. It was observed that the FTIR spectra of all the extracted dragon fruit pectins is similar to that of the commercial apple pectin. Different extraction conditions do not give significant differences in FTIR spectra. The broader band, from 2400 to 3600 cm^{-1} was due to absorbed moisture in the pectin samples.

CONCLUSION

Results of the study obtained indicated that the dragon fruit peels is rich in pectins. The extraction conditions have major impact on the extraction yields but did not have significant difference in pectins structure. Ammonium oxalate extracted pectin exhibit better gelling properties than water or HCl extracted pectin. Further investigations need to be directed at the characterization of the extracted pectin in order to determine the quality of the pectin as food additive.

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REFERENCES

AOAC. 1980. Official Methods of Analysis of the Official Analytical Chemists. 13th ed, Washington D.C.

Attri, B.L. & Maini, S.B. 1996. Pectin from galgal (*Citrus pseudolimon* Tan) peel. *Bioresource Technology*, **55(1)**: 89-91.

Ebrahimi, B., Alemzadeh, I. & Seifkordi, S.K. 2007. Investigation into some parameter affecting pectin gel quality. *Scientia Iranica*, **14**: 174-179.

Emaga, H.T., Ronkart, S.N., Robert, C., Wathelet, B. & Paquot, M. 2008. Characterisation of pectins extracted from banana peels (Musa AAA) under different conditions using an experimental design. *Food Chemistry*, **108**: 463-471.

FAO JECFA. 2009. Pectin. *Monographs* 7.

Fu, J-T. & Rao, M.A. 2001. Rheology and structure development during gelation of low-methoxyl pectin gel: the effect of sucrose. *Food Hydrocolloid* **15**: 93-100.

Gnanasambandam, R. & Proctor, A. 1999. Preparation of soy hull pectin. *Food Chemistry*, **65**: 461-467.

Kalapathy, U. & Proctor, A. 2001. Effect of acid extraction and alcohol precipitation conditions on the yield and purity of soy hull pectin. *Food Chemistry*, **73**: 393-396.

Koubala, B.B., Mbome, L.I., Kansci, G., Mbiapo, F.T., Crepeau, M.J., Thibault, J.F. & Ralet, M.C. 2008. Physicochemical properties of pectins from ambarella peels (*Spondias cytherea*) obtained using different extraction conditions. *Food Chemistry*, **106**: 1202-1207.

Koubala, B.B., Kansci, G., Garnier, C., Mbome, L.I., Duran, S., Thibault, J-F. & Ralet, M-C. 2009. Rheological and high gelling properties of mango (*Mangifera indica*) and ambarella (*Spondias cytherea*) peel pectins. International Journal of Food Science & Technology, **44**: 1809-1817.

Levigne, S., Ralet, M.C. & Thibault, J.F. 2002. Characterisation of pectins extracted from fresh sugar beet under different conditions using an experimental design. *Carbohydrate Polymers*, **49**: 145-153.

Miyamoto, A. & Chang, K.C. 1992. Extraction and physicochemical characterization of pectins from sunflower head residue. *Can. Inst. Food Technol*., **57**: 1439-1445.

Mohamadzadeh, J., Sadeghi-Mahoonak, A.R., Yaghbani, M. & Aalami, M. 2010. Extraction of pectin from sunflower head residues of selected Iranian cultivars. *World Applied Sciences Journal*, **8(1)**: 21-24.

Mollea, C., Chiampo, F. & Conti, R. 2008. Extraction and characterization of pectins from cocoa husks: A preliminary study. *Food Chemistry*, **107(3)**: 1353-1356.

Norziah, M.H., Fang, E.O. & Abd Karim, A. 2000. Extraction and characterization of pectin from pomelo fruit peels. *Gums and Stabilisers for the Food Industry*, **10**: 27-36.

Oakenfull, D.G. 1991. The chemistry of high-methoxyl pectins. In R.H Walter (Ed), *The chemistry and technology of pectin*. New York: Academic Press.

Yapo, B.M., Robert, C., Etienne, I., Wathelet, B. & Paquot, M. 2007. Effect of extraction conditions on the yield, purity and surface properties of sugar beet pulp pectin extracts. *Food Chemistry*, **100(4)**: 1356-1364.

Yeoh, S., Shi, J. & Langrish, T.A.G. 2008. Comparisons between different techniques for water-based extraction of pectin from orange peels. *Desalination*, **218**: 229-237.

Zainudin, M. 2005. Buah naga tanaman berpotensi tinggi. *Agromedia*, MARDI. 4-9 pp.