CHEMICAL ENGINEERING TRANSACTIONS
VOL. 56, 2017
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet
Guest Editors: Jiří Jaromír Klemeš, Peng Yen Liew, Wai Shin Ho, Jeng Shiun Lim

ISBN 978-88-95608-47-1; **ISSN** 2283-9216
DOI: 10.3303/CET1756135

Extraction and Characterisation of Pectin from Dragon Fruit (Hylocereus Polyrhizus) Peels

Dayang Norulfairuz Abang Zaidel*[,a], Jamaeyah Md Rashid[a], Nurul Hazirah Hamidon[a], Liza Md. Salleh[a], Angzzas Sari Mohd Kassim[b]

[a]Department of Bioprocess and Polymer Engineering, Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor
[b]Department of Chemical Engineering Technology, Faculty of Engineering Technology, University Tun Hussein Onn Malaysia, Batu Pahat, Johor
dnorulfairuz@utm.my

Pectins are complex carbohydrate molecules that are used in numerous food applications as a gelling agent, thickener, stabiliser, and emulsifier. Dragon fruit (Hylocereus polyrhizus) is one of the tropical fruits that belong to the cactus family, Cactaceae. Since the peels of dragon fruit are often discarded as waste, it would be an advantage to convert it into a value-added product such as pectin. The objective of this study was to investigate the extraction of pectin from dragon fruit peels under different extraction time using hot water extraction method. The dragon fruit peels were extracted using distilled water at 80 °C with different extraction time of 20, 40, 60 and 80 min. The extracted pectin was characterised by its yield, moisture and ash content, degree of esterification and antioxidant activity. Determination of moisture and ash content was conducted using AOAC standard method. The determination of the degree of esterification of pectin was performed using Fourier Transform Infrared Spectroscopy (FTIR). DPPH assay was used to determine the antioxidant activity of the pectin extract. Based on the result, the yield of pectin decreases (20.34 to 16.20 %) with the increase of extraction time, moisture contents were between 4 to 6 % while ash contents were between 7 to 10 %. Pectin from dragon fruit peels was determined as low methoxyl pectin and has high percentage of antioxidant activity with low value of inhibition concentration (IC_{50}) (0.0063 to 0.0080 mg/mL). 60 min extraction sample exhibits the highest antioxidant activity (81.91 % at 40 µg/mL), followed by 80 min extraction (81.68 % at 40 µg/mL), 40 min extraction (81.38 % at 40 µg/mL) and 20 min extraction (81.31 % at 40 µg/mL).

1. Introduction

Dragon fruit (Hylocereus polyrhizus) or red pitaya is one of the tropical fruits that belong to the cactus family, Cactaceae. It has high amounts of vitamin C which contributes to the high antioxidant properties and water-soluble fibre (Jaafar et al., 2009). The polyphenolic components of dragon fruit and its antioxidant activity have been reported by Tenore et al. (2012). Studies have shown that a mature dragon fruit contains considerable amount of total soluble solids, rich in organic acids (Stintzing et al., 2003), protein (Le Bellec et al., 2006), anthocyanins (Abang Zaidel et al., 2015) and other minerals like potassium, magnesium, calcium and vitamin C. Dragon fruits are widely grown around Malaysia and its peels are always discarded prior consumption of the fruits. This will create waste that could be transformed into a value-added product by using it as an alternative source to extract pectin.

Pectins are complex carbohydrate molecules that are used mainly as gelling agents in the food and beverage industry. Pectins are also used in pharmaceutical and cosmetics. Pectin is mainly composed of α(1, 4)-D-galacturonic residue, with various degrees of methyl esterification. Industrial pectin is extracted from citrus peels and apple pomace with multiple stage physical chemical process characterised by an extraction step with hot dilute mineral acid and recovery through alcohol precipitation (May, 1990). Citrus and apple are not locally available in Malaysia. Dragon fruit is a potential alternative source of pectin that is locally abundant.

Please cite this article as: Zaidel D.N.A., Rashid J.M., Hamidon N.H., Salleh L.M., Kassim A.S.M., 2017, Extraction and characterization of pectin from dragon fruit (hylocereus polyrhizus) peels, Chemical Engineering Transactions, 56, 805-810 DOI:10.3303/CET1756135

Different sources of pectin have different extraction conditions. The extraction conditions vary with the nature of raw material and process economics. It is important to select a suitable extraction condition according to the chemical characteristic of pectin and its properties in order to have high pectin yields without compromising the quality. Depending on the chemical characteristics of pectin, it is used in numerous food applications as a gelling agent, thickener, stabiliser, and emulsifier.
The objectives of this study were (i) to investigate the extraction of pectin from dragon fruit peels using hot water extraction method, (ii) to characterise the properties of pectin extracted from dragon fruit peels.

2. Experimental procedures

2.1 Materials and reagents

Dragon fruits were obtained from a local fruit market in Taman Universiti, Skudai. All reagents were obtained from Sigma-Aldrich.

2.2 Sample preparation

The peels of red dragon fruit were cut into small pieces and washed thoroughly with tap water. The sample was dried in an oven at 60 °C overnight until constant weight. The dried sample was ground into a powder form using a dry blender. The sample was stored in a desiccator until further use.

2.3 Extraction of pectin

Dried sample (8 g) was added with 250 mL of distilled water and was heated to 80 °C for 20, 40, 60 and 80 min. The suspensions were filtered using cheesecloth to separate the residues. The filtrate was then coagulated with 90 % ethanol and stirred regularly for 15 min, and then the pectin was washed with ethanol. The pectin was dried in oven at 50 °C for few hours until constant weight. The final weights were recorded and pectin yield were calculated.

2.4 Determination of pectin yield

Pectin yield was calculated as the ratio of the weight of dried pectin to the dried powder taken for extraction for each extraction time.

2.5 Determination of moisture and ash content

For the determination of ash content, 1 g of pectin was weighed in tared crucible and were heated in muffled furnace at 600 °C for 4 h. The residues left were cooled in desiccator and weighed until it reached a constant weight. For the determination of moisture content, 1 g of pectin were weighed and dried at 100 °C in oven for 4 h until constant weight (AOAC, 1980).

2.6 Determination of degree of esterification

The degree of esterification (DE) was determined by using the Fourier Transform Infrared (FTIR) spectroscopy method adapted from Singthong et al. (2004). DE is defined as the percentage of number of esterified carboxylic groups over number of total carboxylic groups. The samples were analysed using Fourier FTIR spectrophotometer (Perkin Elmer RX1 FT-IR, USA) in order to confirm the identity of dragon fruit pectin and estimate the DE. Commercial pectin with known DE of 26 % and 59 % from citrus pectin were used as standard. The spectra were recorded in the absorbance range from 4,000 to 400 cm^{-1}(mid-infrared region).

2.6 Determination of antioxidant activity

Antioxidant activity was determined using DPPH (2,2-diphenyl-1 picrylhydrazyl) assay (Bedawey et al., 2010). Preparation of DPPH solution was adapted from Molyneux (2003) with minor modification. DPPH was mixed with 10 µg/mL methanol. Then, 1 mL of 10 µg/mL of extract was pipetted into 3 mL DPPH solution with concentration 10 µg/mL (1:3) to initiate the reaction. After stored at dark place for 30 min, the absorbance was read at wavelength 517 ηm by using spectrophotometer UV-Vis (Hewlett Packard 8435, USA). Methanol was used as a blank and DPPH solution 10 µg/mL as standard. The experiment was repeated with 20, 30, 40 and 50 µg/mL for the sample and blank. Antioxidant activity of the extract was determined based on the reduction of DPPH absorbance by calculating percentage of antioxidant activity or known as radical scavenging activity or percentage of inhibition as shown in Eq(1). The value for inhibition concentration (IC_{50}) was calculated by graph of percentage of antioxidant versus series of concentration.

$$\text{Antioxidant activity (\%)} = \frac{\text{Absorbance of blank} - \text{Absorbance of sample}}{\text{Absorbance of blank}} \times 100\ \% \quad (1)$$

3. Results and discussion

3.1 Pectin yield

Figure 1 shows the percentage of yield pectin extracted with different extraction time of 20, 40, 60, and 80 min. The yield of pectin decreased along with the increasing of extraction time. The lowest yield, which was 16.20 %, was obtained from sample extracted for 80 min. The highest yield (20.34 %) was obtained when heated for 20 min. Generally, the duration of hot water extraction influenced the pectin yield. The longer the extraction time, the lower the pectin obtained. In this research, there was no addition of dilute acid during extraction, so the yield obtained was low. This is because addition of acid during extraction helps to protect the pectin from losing some of its branching and chain length. Lower pH values negatively affect the galacturonic acid content of pectin, but increases the pectin yield.

3.2 Determination of moisture and ash content

The moisture contents of pectin extracted with different extraction time were illustrated in Figure 1. The lowest moisture content obtained was from 20 min extraction which was 4.37 %. The result showed that moisture content increases as the extraction time increases. But there was an exception for 60 min extraction time where the moisture content was highest at 5.50 %. Overall, the moisture contents were between 4 to 5 % which can be considered as low moisture content. According to Muhamadzadeh et al. (2010), pectin should have as low moisture content as possible for safe storage and to inhibit the growth of microorganisms that can affect the pectin quality due to the production of pectinase enzymes.

The result for ash content for pectin extracted shows that as the extraction time increases, the ash content decreases. The highest ash content was at 20 min extraction which was 10.02 % and the lowest ash content was at 80 min extraction (7.10 %). The value obtained was below than 10 % thus can be concluded as low ash content. Low ash content of below 10 % and maximum limit of ash content of 10 % are one of the good criteria for gel formation (Ismail et al., 2012). The ash content indicates the purity of the pectin. The lower the ash content, the higher the purity of the pectin.

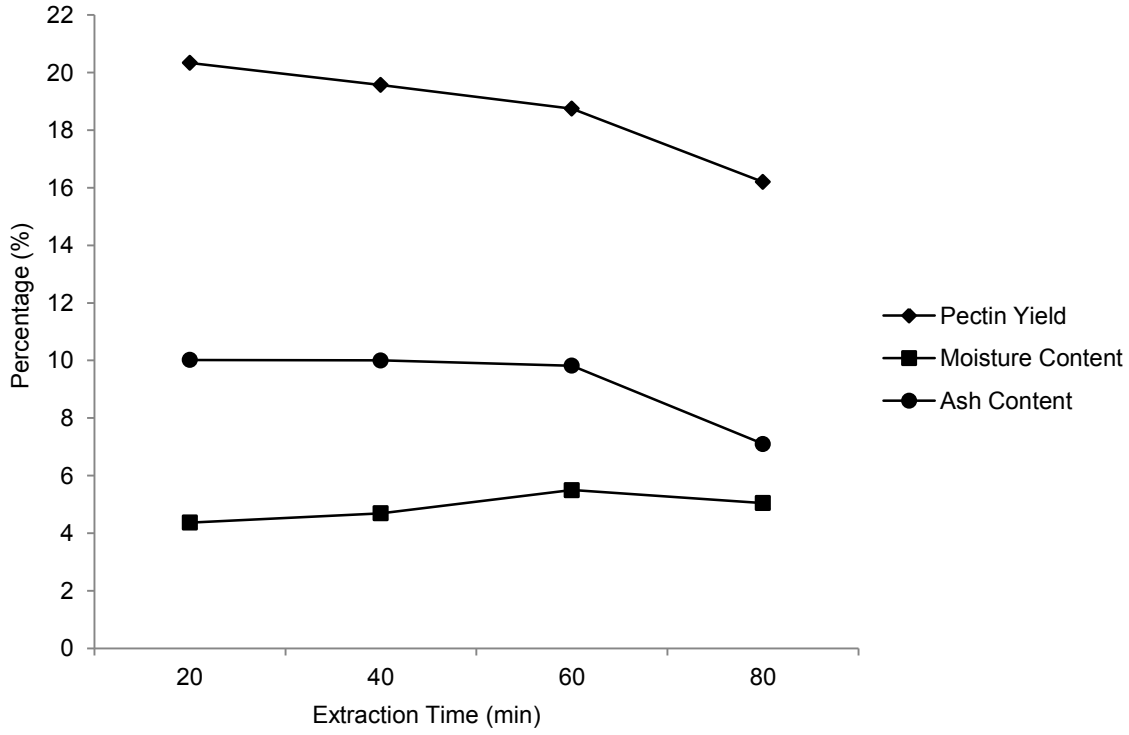

Figure 1: Graph of yield, moisture content and ash content of pectin extracted with different extraction time

3.3 Determination of degree of esterification

Figure 2 shows the FTIR spectra of pectin extracted with different extraction times, commercial citrus pectin 26 % and 59 % DE. FTIR spectra in the wave number between 800 and 1300 cm^{-1} is considered as the finger print region for carbohydrates, which allows the identification of major chemical group in polysaccharides. To know whether the sample obtained were pectin or not, comparison were made with commercial pectin. The result shows that the absorption pattern of samples was similar with commercial pectin, so the identity of samples obtained were pectin. Further analysis showed that broader band of absorption between 3,600 and 3,000 cm^{-1} was due to O-H stretching where an absorption band at 2,900 cm^{-1} was due to C-H stretching of CH_2 groups. Absorption bands appeared at 1,745 and 1,600 cm^{-1} were assigned to the stretching vibrations of ester carbonyl (C=O) groups and carboxyl ions (COO-). A weaker absorption at 1,745 cm^{-1} coupled with a higher absorption at 1,600 cm^{-1} indicated that the pectin extracted for 20, 40, 60 and 80 min were low methoxyl pectins (DE < 50 %) when compared with commercial citrus pectin DE 29 % at region at 1,745 and 1,600 cm^{-1}. The results indicated that all pectin extracted have DE lower than 29 %.

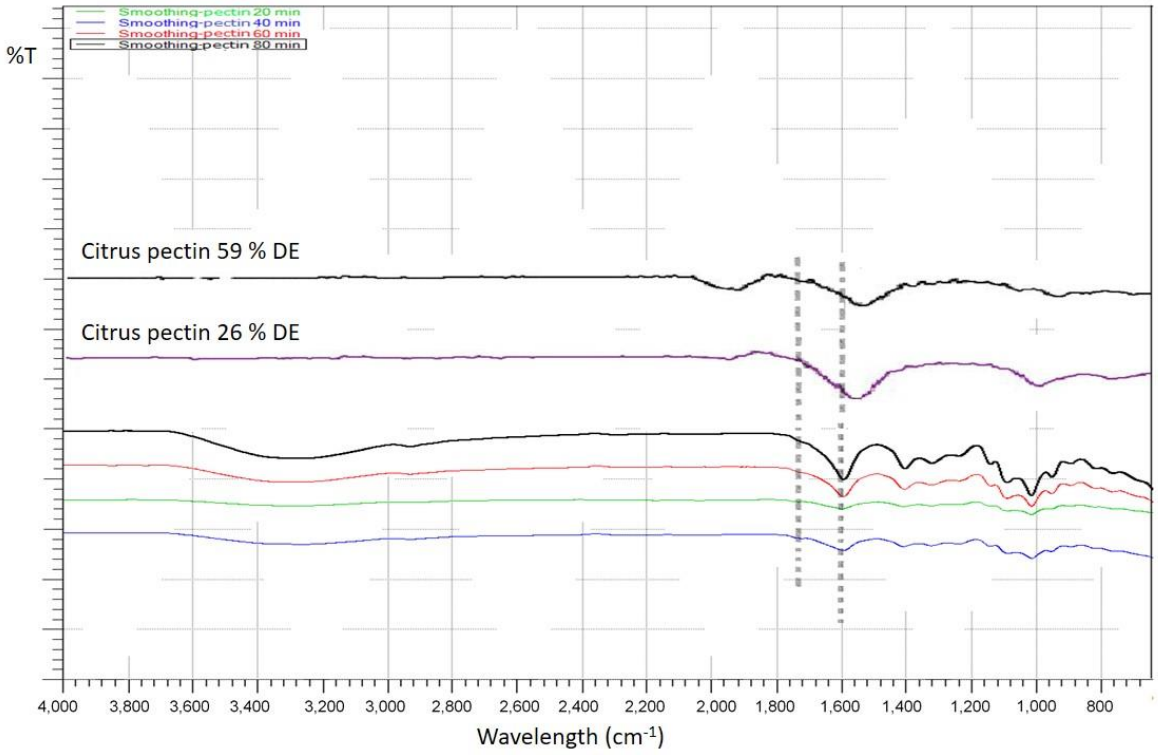

Figure 2: FTIR spectra of pectin extracted at 20, 40, 60, 80 min extraction time, commercial citrus pectin 26 % and 59 % DE

Figure 3 shows the comparison of antioxidant activity versus concentration for 20, 40, 60 and 80 min extraction. The antioxidant activity increases with the increasing of concentration of extract. 60 min extraction sample exhibited the highest antioxidant activity (81.91 % at 40 µg/mL), followed by 80 min extraction (81.68 % at 40 µg/mL), 40 min extraction (81.38 % at 40 µg/mL) and 20 min extraction (81.31 % at 40 µg/mL) showed the weakest antioxidant activity. Based on the result, the value of antioxidant activity of all samples were similar which means that different extraction time does not affect the antioxidant activity at concentration 40 µg/mL. This DPPH assay measures the ability of sample to donate hydrogen to DPPH radical. In present study, all samples demonstrated purple bleaching reaction at increasing concentrations, showing the presence of compounds responsible as free radical scavengers which reduced the initial DPPH concentration. Dragon fruit's peel extract has high radical scavenging activity or antioxidant activity showing that they contain high amount of radical scavenging compound. In terms of IC_{50}, the highest value was shown by 20 min extraction (0.008 mg/mL) followed by 80 min extraction (0.0071 mg/mL), 40 min extraction (0.0065 mg/mL), and the lowest was 60 min extraction (0.0063 mg/mL). For 60 min extraction, it showed the lowest value of IC_{50} which means that it had more capability to scavenge the free radicals. IC_{50} value for positive control ascorbic acid is

0.00502 mg/mL which is lesser than the lowest IC_{50} value of the extract (0.0063 mg/mL) proving that the extract had slightly lesser free radical scavenging potential than ascorbic acid.

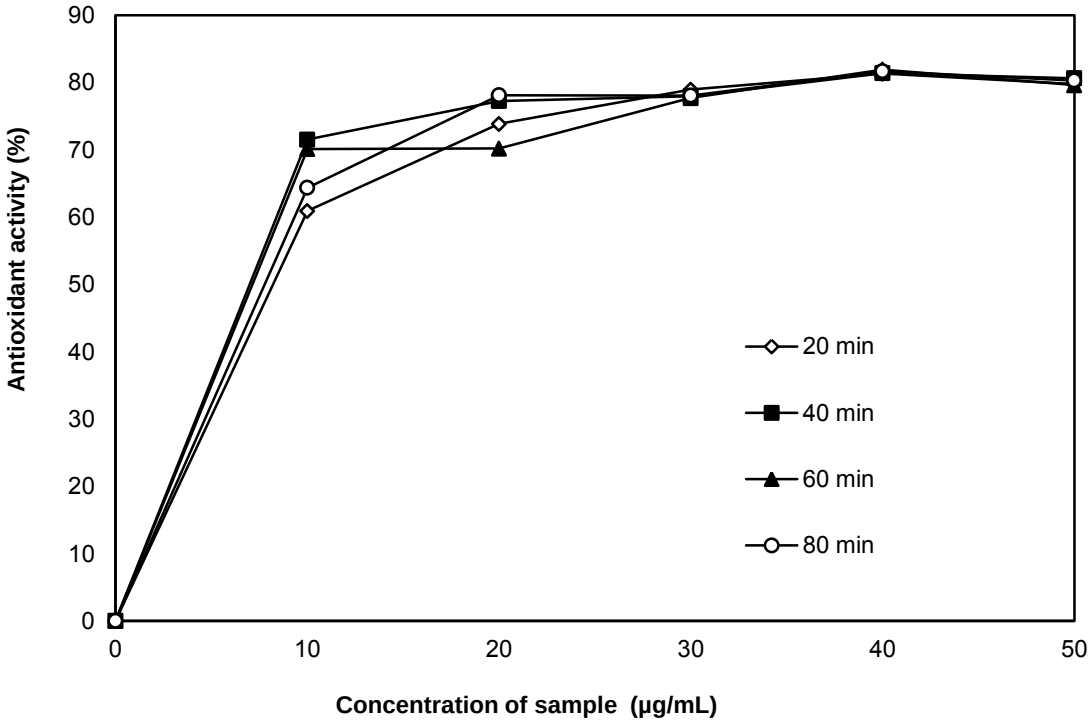

Figure 3: Graph of antioxidant activity (%) against different concentration of pectin (µg/mL) extracted at 20, 40, 60 and 80 min extraction time

4. Conclusions

As conclusion, pectin yield in this study ranges from 16 to 20 % for different extraction time. The moisture content of pectin did not exceed 6 % and can be considered as low content. As extraction time increases, the ash content of pectin decreases. The ash contents obtained were in the range of 10 %. From FTIR analysis, it showed that all pectins extracted with different extraction time were low methoxyl pectin, DE < 50 % when compared with commercial citrus pectin of 26 % DE. The highest antioxidant activity was shown by 40 µg/mL concentration of pectin extracted for 60 min. The extraction conditions give impact on the extraction yield and its characteristics. Further investigations need to be directed in order to know the best condition for the extraction process.

Acknowledgments

The authors would like to acknowledge Universiti Teknologi Malaysia and Ministry of Higher Education for the financial support under Fundamental Research Grant Scheme with Project No R.J130000.7844.4F447.

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