

EXTRACTION AND CHARACTERIZATION OF PECTIN FROM ORANGE, WATERMELON AND BANANA FRUIT PEEL WASTE

Mohd Zaki Sukor^{*1,a}, Muhd. Irsyad Roslan^{2, b}, Muhd. Imran Salleh^{3,c}, and Mohammad Abdullah^{4,d}

^{1,2,3,4}College of Engineering, Universiti Teknologi MARA, Cawangan Johor, Kampus Pasir Gudang, Bandar Seri Alam, 81750 Masai, Johor.

^amdzaki@uitm.edu.my, ^bmuirsyadroslan@gmail.com, ^cimran115215@gmail.com, ^dmoham3767@uitm.edu.my

*Corresponding author's
email :
mdzaki@uitm.edu.my

Abstract

Fruit peels are an important commercial supply of pectin, hence the goal of this work was to extract pectin from them. Pectin is a typical gelling agent for jams and jellies and is widely used in medicines and the food processing industry. Following the extraction process, the effect of various parameters on pectin extraction and detail applications was investigated. Banana, orange, and watermelon peels were used as fruit peels. The extraction solvents were hydrochloric acid and sulphuric acid, which were placed in a water bath. The pectin yield of the extracted pectin by hydrochloric acid and sulphuric acid ranged from 0.145 to 2.5% and 0.16 to 8.5% respectively. These results indicated that more pectin was retained using sulphuric acid as solvent.

Keywords: Fruit peels; Pectin; Extraction; Hydrochloric acid; Sulphuric acid

1.0 INTRODUCTION

Pectin could be found in the primary cell wall and intercellular of plants. Pectin contains anionic polysaccharides and may vary depending on various factors such as species, variety, tissue, maturity, plant part and growing condition. Mostly, pectin was extracted from fruit peel waste and is generally highly concentrated in legumes and citrus fruits than cereals. The fruits that contain a high level of pectin are apple, grapefruit, orange and apricot. Other sources of pectin are bananas, beets, cabbage, carrot, and watermelon [1]. Figure 1 shows the chemical structure of the pectin.

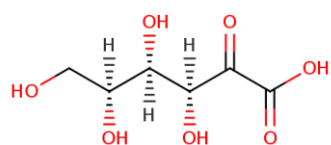


Fig. 1. Chemical structure of pectin.

Pectin is mainly composed of $\alpha(1, 4)$ -Dgalacturonic residue, with different degrees of methyl esterification from each source, such as fruits [2]. The amount of methoxyl groups in pectic carbohydrates influences the degree of methoxylation (DM) of pectin, which varies depending on plant type and fruit ripening stage [3]. There are two classification of pectin; high methoxyl pectins (HMP) and low methoxyl pectins (LMP). HMP becomes gels when heated in solutions with an acidic pH and sugar contents more significant than 55%, but LMP gels only when calcium ions are present [4].

Pectin has been highly valuable to the cooking industry as a whole. It has been found consumption in general texture modifiers, gelling agent, thickening agent and stabilisers in food [5]. It is widely used in jams, jellies, desserts, yoghurts, confectionaries, and anti-diarrheal agents, among other things. However, new pectin applications have recently become quite

essential. The most notable application is in the where pectin has found widespread use in heavy metal pollution treatment regimens. Furthermore, by encapsulation, pectin can operate as an excipient for a variety of pharmacological actives. Pectin encapsulation is highly efficient and thus increasing medication efficacy [6].

Several methods to extract pectin has been reported in the many literatures. Pectin can be extracted through sequential extraction from alcohol-insoluble solids (AIS) [7] and ultrasonic treatment [8]. It was studied to be able to establish conditions in which pectin degradation can be minimised. Pectin extracted under these conditions was shown to have a higher yield while retaining the drainage qualities of the extracted fruit mass [9].

Extraction of pectin through the direct boiling extraction method was also known to be the most straightforward and oldest method for extracting pectin substance. However, it also produces a minimal amount of pectin (Alamineh, 2018). Direct boiling is also the most usual technique used by many industries. This approach requires roughly 2 hours to achieve a low good yield of pectin, making it a time demanding operation. There is also the possibility of pectin thermal deterioration due to repeated heating [10].

According to the results of a flash pectin extraction from orange using microwave heating under pressure, the molar mass, size, and intrinsic viscosity were all increased when compared to pectin extracted using the direct boiling method [11]. It is undeniable that this technique consistently results in an excellent performance, but the cost of the process is significantly higher than the cost of other techniques.

Acid extraction method can also extract a reasonable amount of pectin, albeit it is one of the conventional methods. Acidified water is used with a range of pH up to 2, then the solution mixture was heated in a water bath at temperatures of 80°C for roughly 2 hours. The pectin substances was precipitated by using ethanol and centrifuge [5]. The most commonly acidifying materials used are hydrochloric acid (HCL), citric acid (C₆H₈O₇), nitric acid (HNO₃), ammonium oxalate (C₂H₈N₂O₄) and oxalic acid (C₂H₂O₄).

The objective of this study is to investigate the yield gain of pectin extracted by the acid extraction method. The characterisation of pectin was also observed and measured, including colour, acidity, solubility, and degree of esterification using FTIR. The result from the different inorganic acid solvent used is also compared.

2.0 EXPERIMENTAL

Raw material preparation. Fruit wastes such as of orange, watermelon and banana peels were collected from restaurant and pastries in Pasir Gudang. The peels were washed before it was cut to

treatment of wastewater effluents, small pieces. After cutting the peel to small pieces, the peel was grinded by using grinding machine. Distilled water, the acid use which is HCl and H₂SO₄, ethanol and acetone were prepared.

Pretreatment of shredded fruit peel. The main objective of pretreatment of fruit peel is to eliminate the shell that protects the cellulose, to reduce the crystallite size of the cellulose, and to break this shell in order for the enzyme to access the substrate. Prior to shredding the fruit peels, pretreatment heating was carried out inside a waterbath at a temperature of 90°C.

Experimental setup & procedure. The sample acquire had to be prepared and in good condition for water bath heating, filtration, precipitation and centrifuge of pectin. The samples of each fruit peel were measured to 20 grams. After that, the measured fruit peels were mixed with 250ml of distilled water and 2.5ml of HCl before heating for 2 hours using a water bath. Afterwards, the mixture was cooled down before filtrate to get the solution of pectin. After filtration, the solution was precipitated by adding ethanol with a ratio of 2:1 (by volume). The solution was then centrifuged for 10 minutes and 5000rpm at 0°C to get gel pectin. The gel pectin was washed using 200ml of acetone before being filtered on a nylon cloth to remove the residual acid and universal salt. Then, pectin was dried in an oven for 1 hour.

Characterisation of extracted pectin. The colour of the dried pectin was observed by visual observation. The colour of banana, orange and watermelon pectin were brown, orange and colourless green respectively. The yield of the pectin was determined by using the equation (1).

$$\text{Yield, \%} = \frac{\text{mass of pectin}}{\text{Mass of peels}} \times 100 \quad (1)$$

3.0 RESULTS AND DISCUSSION

Colour. The colour of the pectin was determined visually. Pectin colour is crucial because it determines the appearance of the gel produced. According to Table 1, the pectin colour of orange, banana, and watermelon peels was orange, chocolate, and colourless, respectively.

Dry amorphous powders will tend to scatter light and so appear as a whitish powder. Organic molecules with colour tend to have benzene groups or azo type bonds or generally delocalised bonds. These tend to absorb in the visible spectrum. As it is broken down into the large biopolymer, smaller molecules that go into the solution are formed and display these electronic properties that give colour. The same process of breaking down large molecules happens in the sweetening of fruit. The concentration of the pectin will affect the colour because of the absorbance intensity [12].

Table 1. Colour of pectin extracted

Type of fruit peels	Precipitating Agent	
	HCl	H ₂ SO ₄
Orange	Yellowish	Yellowish
Banana	Brownish	Brownish
Variable 2	Light green	Light green

Yield of Pectin. The yield of the pectin which was extracted using a different precipitating agent was presented in Table 2. The highest yield of the pectin using HCl was an orange with 2.5 % followed by banana and watermelon which only 0.3 % and 0.145%, respectively. When using sulphuric acid as a precipitating agent, pectin's yield increased drastically for all fruit peels. However, orange peels still yield the highest amount of pectin which is 8.5% compared to both pectin yield from banana and watermelon which are 2.0% and 0.16%, respectively.

In terms of yield gain, it was found that sulphuric acid gives the most yield of pectin on the 3 fruits extracted. This is because of the presence of sulphate ions in the soaking process [12]. Hirt & Jones [13] also prove that sulphate ions play important roles and increase the extraction of pectin.

Table 2. Yield of pectin using a different precipitating agent on each sample.

Type of fruit peels	Yield of pectin (%)	
	HCl	H ₂ SO ₄
Orange	2.5	8.5
Banana	0.3	2.0
Variable 2	0.145	0.16

Fourier-transform infrared spectroscopy (FTIR). The following conclusions were reached after comparing the spectra of six samples, as illustrated in Figure 2. When compared to the other samples, the increase in absorption bands at 3307.96 cm⁻¹ for orange peels treated with HCl has the largest broadband. The key chemical groups in pectin were identified and structural information about pectin was obtained using FTIR spectra in the 4000-400 cm⁻¹ range.

By comparing the FTIR spectra in the following distinctive regions, the spectral data acquired were analysed. The O-H stretching band was identified in a region between 3100-3600 cm⁻¹ and the C-H stretching bands between 2800-3000 cm⁻¹. The 'fingerprint' region of the spectra (under 2000 cm⁻¹), contributing to the resonant absorption energy of the pyranose cycle vibrations (950-1200 cm⁻¹) and carboxylic groups in the region of 1200-1800 cm⁻¹ [14].

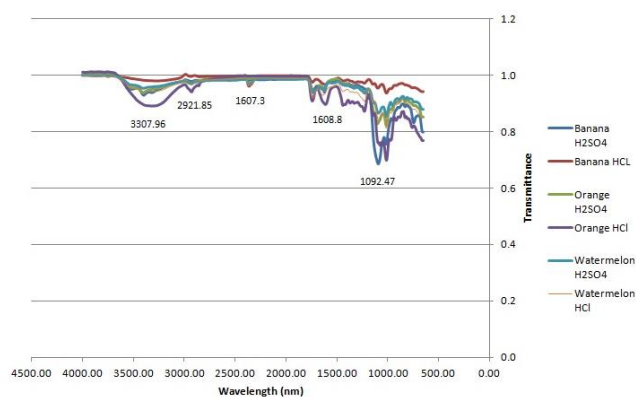


Fig. 1. The FTIR analysis on pectin extracted by using on HCl an H₂SO₄ on banana, orange, and watermelon.

4.0 CONCLUSION

It can be concluded that the types of solvent used can greatly affect the yield of pectin. H₂SO₄ solvent shows a higher yield of pectin compared to HCL. The other factor that can be included is the types of fruit. Different types of fruit show a different yield of pectin despite the same amount of solvent used in the experiment. From these 3 fruit peels (banana, orange and watermelon), orange is shown to yield the highest amount of pectin compared to the rest.

Acknowledgement

Authors would like to acknowledge the Faculty of Chemical Engineering UiTM Pasir Gudang for providing the facilities for laboratory work.

References

- [1] T. H. Emaga, C. Robert, N. S. Ronkart, B. Wathelet, and M. Paquot, "Dietary fibre components and pectin chemical features of peels during ripening in banana and plantain varieties," vol. 99, pp. 4346-4354, 2008.
- [2] N. S. M. Ismail, N. Ramli, N. M. Hani, and Z. Meon, "Extraction and characterization of pectin from dragon fruit (*Hylocereus polyrhizus*) using various extraction conditions," *Sains Malaysiana*, vol. 41, no. 1, pp. 41-45, 2012.
- [3] E. G. Maxwell, N. J. Belshaw, K. W. Waldron, and V. J. Morris, "Pectin - An emerging new bioactive food polysaccharide," *Trends Food Sci. Technol.*, vol. 24, no. 2, pp. 64-73, 2012.
- [4] D. F. Torralbo, K. A. Batista, M. C. B. Di-Medeiros, and K. F. Fernandes, "Extraction and partial characterization of *Solanum lycocarpum* pectin," *Food Hydrocoll.*, vol. 27, no. 2, pp. 378-383, 2012.

- [5] E. Abebe Alamineh, "Extraction of Pectin from Orange Peels and Characterizing Its Physical and Chemical Properties," *Am. J. Appl. Chem.*, vol. 6, no. 2, p. 51, 2018.
- [6] R. K. Mishra, A. K. Banthia, and A. B. A. Majeed, "Pectin Based Formulations Biomedical Applications: A Review," vol. 5, no. 4, pp. 1–7, 2012.
- [7] E. S. Andreani, S. Karboune, and L. Liu, "Extraction and characterization of cell wall polysaccharides from cranberry (*Vaccinium macrocarpon* var . Stevens) pomace," vol. 267, no. January, 2021.
- [8] Y. Yang, Z. Wang, D. Hu, K. Xiao, and J. Y. Wu, "Efficient extraction of pectin from sisal waste by combined enzymatic and ultrasonic process," *Food Hydrocoll.*, vol. 79, pp. 189–196, 2018.
- [9] I. Panchev, N. Kirchev, and C. Kratchanov, "Improving pectin technology. II. Extraction using ultrasonic treatment," *Int. J. Food Sci. Technol.*, vol. 23, no. 4, pp. 337–341, 1988.
- [10] J. Zheng, H. Li, D. Wang, R. Li, S. Wang, and B. Ling, "Radio frequency assisted extraction of pectin from apple pomace: Process optimization and comparison with microwave and conventional methods," *Food Hydrocoll.*, vol. 121, no. April, p. 107031, 2021.
- [11] Y. Zouambia, K. Youcef Ettoumi, M. Krea, and N. Moulai-Mostefa, "A new approach for pectin extraction: Electromagnetic induction heating," *Arabian Journal of Chemistry*, vol. 10, no. 4, pp. 480–487, 2017.
- [12] P. Dervisi, J. Lamb, and I. Zabetakis, "High pressure processing in jam manufacture: Effects on textural and colour properties," *Food Chem.*, vol. 73, no. 1, pp. 85–91, 2001.
- [13] S. Hirt and O. G. Jones, "Effects of chloride, thiocyanate and sulphate salts on β -lactoglobulin-pectin associative complexes," *Int. J. Food Sci. Technol.*, vol. 49, no. 11, pp. 2391–2398, 2014.
- [14] J. Singthong, S. W. Cui, S. Ningsanond, and H. Douglas Goff, "Structural characterization, degree of esterification and some gelling properties of Krueo Ma Noy (*Cissampelos pareira*) pectin," *Carbohydr. Polym.*, vol. 58, no. 4, pp. 391–400, 2004.