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Effect of Extraction Conditions on Pectin Yield Extracted from Sweet Potato Peels Residues using Hydrochloric Acid

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Pectins are complex polysaccharides obtained from higher plants which are structurally composed mainly of α-1, 4-linked-D-galacturonic acid (Gal A). Pectin has a good property of gelling, thickening and emulsification thus it is used widely in food and beverage industry. The applications of pectin in food industry are developing thus the demand of pectin is also increasing. Sweet potato residue is a potential local source of pectin. The peels of sweet potato are usually discarded prior to the consumption of the fruit. In Malaysia, starch industries and small local business are generating a lot of residual waste from sweet potato. Converting the waste into a value-added product such as pectin would bring great advantage in term of environmental concern and profit. Effects of extraction conditions such as extraction temperature (60 – 100 °C), extraction time (60 – 180 min) and solution pH (1 – 3) on the extraction yield of pectin (%) from sweet potato residues were investigated in this study. The extracting solvent used was hydrochloric acid. The highest yield was 2.59 % which was obtained at extraction temperature 90 °C, extraction time 60 min and solution pH 1.5. All three extraction conditions showed significant effect on the yield of pectin (%).

1. Introduction

Pectin is a naturally occurring biopolymer that has been used successfully for many years in the food and beverage industry as a thickening agent, gelling agent and colloidal stabiliser (Takamine et al., 2007). Pectin is a linear polysaccharide that consists mainly of D-galacturonic acid (GalA) units (Mukhiddinov et al., 2000) joined by α-(1-4) glycosidic linkage. Commercially, pectin is exclusively extracted by apple pomace and citrus peel that are by-products of juice manufacturing. According to the literature, fresh weight of plant material accomplishes 0.5 - 4.0% of pectin substances (Faravash and Ashtiani, 2008). Extraction of pectin with acid solution is the most convenient approach for the industrial (May, 1990). The pectin is extracted by treating the raw material with hot dilute mineral acid at pH about 2. Although pectin is available commonly in most plants, the source for commercial pectin is limited to apple pomace and citrus peel. In recent years, various valuable sources were studied for pectin manufacturing, such as sugar beet (Levigne et al., 2002), banana peels (Emaga et al., 2008), mangosteen rind (Gan and Latiff, 2011), cacao pod husk (Vriesmanna et al., 2011) and passion fruit peels (Pinheiro et al., 2008). Therefore, the research interest on pectin has been increasingly stimulated because of its abundant supply and functional properties. Recent study has reported that industrial potato waste contain appreciable amount of rhamnogalacturonan I (hairy region of pectin) (Byg et al., 2012). This finding opens the possibility to investigate the potential usage of other crop residue material such as sweet potato residue, as an alternative pectin source. In Malaysia, abundance of sweet potato residues is produced from large starch processing industry and even small local business. Very often the residues are discarded as waste, which in turn causes environmental pollution and waste of resources. It was reported that sweet potato residues contain about 15 % pectin on a dry matter basis (Mei et al., 2010). By using sweet potato residues as an alternative source for pectin, the residues can be converted into a value added product that is profitable as well as reducing pollution to the environment.

Previous study on extraction of pectin from sweet potato residues using citric acid obtained the highest yield of 65.8 % of pectin (Hamidon and Abang Zaidel, 2016). When extracting pectin from sweet potato residues using hydrochloric acid at temperature 90 °C for 1 h, the highest yield obtained was 74.43 % (Abang Zaidel et al.,

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2014). The yield and characteristic of extracted pectin varies depending on the extraction factors; extraction time, extraction temperature, extraction method and types of acid used as extracting solvent.
The objective of this study is to investigate the effect of extraction conditions to extract pectin from sweet potato residues to the yield of pectin extracted. The extraction conditions of interest were; extraction temperature (°C), solution pH and extraction time (min). The extraction solvent used in was hydrochloric acid.
This paper includes the method of extracting pectin from sweet potato residues and the dried cell wall material preparation step prior to the extraction, the simple experimental design, discussion of the results obtained and finally the conclusions.

2. Experimental procedures

This section covers the details of the experimental procedures including the materials, reagents and equipment that were used in this research.

2.1 Materials and reagents

Sweet potato residues were obtained from a local snack stall in Taman Universiti, Skudai. Heat-stable α-amylase (Termamyl 120 type LS) and amyloglucosidase (EC 3.2.1.3 from Aspergillus niger, 30 - 60 units per mg protein) for enzymatic hydrolysis and all reagents were obtained from Sigma-Aldrich.

2.2 Sample preparation

Sweet potato peels residues were washed with tap water. The residues were then sliced into small pieces and macerated in a Waring blender (Scientific Industries, NY). The slurry was filtered through double layers of cheesecloth to separate the residue from the starch milk. The residue was rinsed thoroughly with running water until the water ran clear, and was then dried in a forced-air dryer (Whisper 500, Philip, Germany) at 45 °C for 18 h. The dried residue was ground before it is used for enzymatic hydrolysis.

2.3 Dried cell wall material preparation

Dried cell wall material is prepared according to a previous enzymatic hydrolysis method by Abang Zaidel et al. (2015). Ground dried sweet potato residue (10 g) was suspended in distilled water (200 mL) and boiled for 5 min. The suspension was kept at 80 °C, and 0.5 mL of heat-stable α-amylase was added, and then incubated for 30 min to hydrolyze the residual starch. The mixture was centrifuged at 3,000 rpm for 10 min, supernatant was discarded and digestion of the residue was repeated with 0.5 mL amyloglucosidase, and then incubated at 55 °C for 30 min at pH 4.5. The mixture was filtered using two layers of cheesecloth. The residue was washed with distilled water, methanol and acetone, successively, and oven dried for 48 h at temperature 60 °C.

2.4 Pectin extraction

Pectin was extracted according to a previous method by Abang Zaidel et al. (2015). The cell wall materials were dispersed in distilled water under specific conditions of extraction temperature, time and solution pH as tabulated in Table 1. The solution pH was adjusted using citric acid. After the extraction, the suspensions were centrifuged at 10 °C for 15 min at 10,000 rpm (Kubota Model, Germany). The liquid fraction containing extracted pectin materials was neutralized with 32 % NaOH, then the same volume of 95 % ethanol was added and the mixture was stirred for 5 minutes and then stored at 4 °C for 12 h. The mixture was then centrifuged at 10,000 rpm for 15 min and the pectin residue washed successively with 70, 80, 90 % ethanol. The extracted pectin was dried in a freeze dryer for 24 h, ground and stored in a desiccator until further analysis.

Table 1: Experimental design

Manipulating Variables	Range					Constant Variables		
						Temperature (°C)	Time (min)	pH
Extraction temperature (°C)	60	70	80	90	100	-	60	1.5
Extraction time (min)	60	90	120	150	180	90	-	1.5
Solution pH	1	1.5	2.0	2.5	3.0	90	60	-

2.5 Analysis of yield of pectin

The weight of extracted pectin was measured and the yield of pectin was determined using the following Eq(1).

$$\text{Yield} = \frac{\text{Weight of extracted pectin (g)}}{\text{Weight of dried sweet potato cell wall material (g)}} \times 100\ \% \qquad (1)$$

3. Results and discussion

In this section, the results of effect of temperature, solution pH and extraction time to the yield of pectin obtained from the study is presented and discussed. Each of the variables is further discussed in individual sub-section.

3.1 Effect of temperature

Effect of extraction temperature was investigated by manipulating temperature in range of 60 to 100 °C at constant time of 60 min and solution pH kept at 1.5. A study by Hamidon and Abang Zaidel (2016) investigated the optimal conditions of pectin extraction from sweet potato residues using citric acid. The range of temperature used in the said study was 60 to 100 °C. Figure 1 shows the relationship of extraction temperature on the yield of pectin. The yield of pectin increases with increasing temperature until it reaches point temperature 90 °C where the highest yield of 2.59 % pectin is obtained.

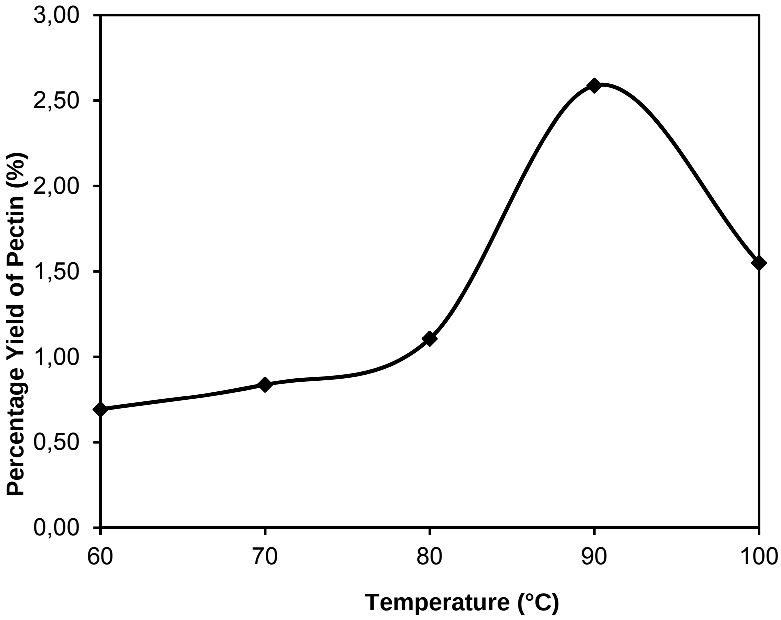

Figure 1: Graph showing correlation between extraction temperature (°C) and yield of pectin (%) at pH 1.50 and extraction time 60 min.

The result was in agreement with that of standard acid extraction method, in which the yield of pectin increased with increasing temperature and an elevated temperature of about 90 °C is commonly employed to extract pectin (Masmoudi et al., 2008). Maximum temperature of 100 °C gives lower yield of pectin. Extraction with very high temperature could induce the hydrolysis of pectin to short-chain sugars, which cannot be precipitated using ethanol thus decreasing the yield of pectin (Canteri-Schemin et al., 2005). Foo et al. (2016) in their study obtained the highest yield of pectin (19.2 %) from sweet potato at temperature 90 °C. However, the yield obtained in this study is fairly low compared to the study. This may be attributed to the variation of sweet potato residues used in both study.

3.2 Effect of solution pH

Pectin extractions were conducted using solution pH 1 to 3 at temperature 90 °C and time 60 min to study the effect of solution pH on the yield of pectin. Xue et al. (2011) in their optimization study of pectin extraction from citrus peels carried out a preliminary study prior to the optimization using range of pH 1 to 3. Abang Zaidel et al. (2015) studied the effect of concentration of acid on the yield of sweet potato pectin at constant temperature 90 °C and time 60 min.

Figure 2 depicts the effect of solution pH on the yield of pectin extracted from sweet potato residues. It can be observed that the yield of pectin decreases when the pH increases especially at point pH 2. Generally, higher pectin is extracted from sweet potato residues at pH lower than 2. This may be due to the fact that high temperature and low pH might prompt the disruption of hydrogen bonds and ester linkages between pectin and cell wall and then increase the rate of diffusion and pectin extraction (Renard et al., 1995; Cho and Hwang, 2000; Masmoudi et al., 2008).

Based on previous studies, approximately temperature of 90 °C and solution pH of 2 to 3 were often utilised as extraction conditions for pectin (Kliemann et al., 2009). However, in this study, pectin yield is generally higher at pH lower than 2. The yield of pectin at pH 3 is almost near to 0 % which suggests that pectin extraction is not suitable to be conducted at pH higher than 3. The highest yield is obtained at pH 1 and it decreases slightly when the pH increases to 1.5. The difference of yield between pH 1.5 and 2 is very large while between pH 2 to 3 only varies slightly. The best pH to use is 1 to 1.5 and further study should focus on extraction of pectin at pH range of 1 to 2 only.

Another optimization study by Zhang et al. (2013) discovered that the best solution pH to extract pectin is at pH 1.71 when the temperature and time are kept constant at 92.98 °C and 2.23 h and the yield of the pectin obtained is 5.08 %. The results above indicated that appropriate values of extracting variables were necessary to obtain high pectin yield.

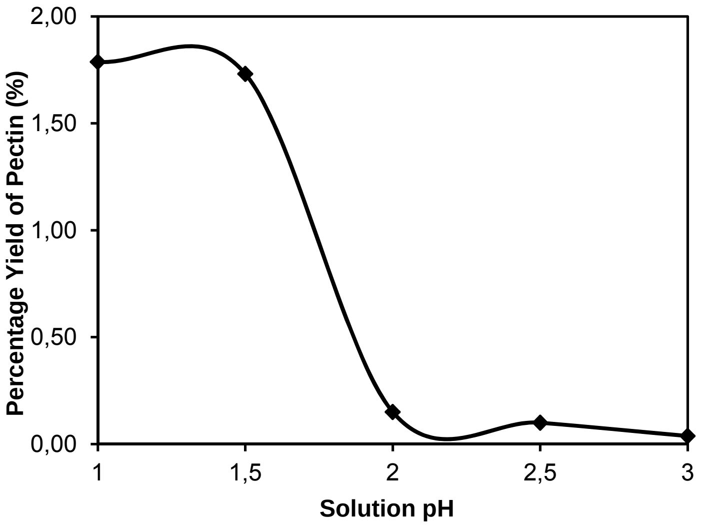

Figure 2: Graph showing the effect of solution pH to the yield of pectin (%) at constant extraction temperature 90 °C and extraction time 60 min.

3.3 Effect of extraction time

Effect of extraction time on the yield of pectin is studied within the range 60 to 180 min at constant temperature 90 °C and solution pH 1.5. The range used is similar to a study of pectin extraction conducted by Zhang et al. (2013).

The study obtained the highest yield of pectin at extraction time 132 min. Figure 3 depicts the relationship of extraction time and yield of pectin. The yield decreases gradually as the extraction temperature increases from 60 to 180 min. The highest yield of 2.59 % is obtained when the extraction time is 60 min. Prolonged extraction time might induce the digestion of pectin thus making it difficult to be precipitated by ethanol which lowers the yield of pectin. During extraction process of pectin, the period of when acid is added to the substrate and the ethanol precipitation should be as short as possible in order to prevent the acid from breaking down the glycoside and ester linkage (Xue et al., 2011). This could negatively affect the molecular weight of pectin and its gelling property. The result obtained in this study is similar to Xue et al. (2011) that discovered the best extraction time to extract pectin from citrus peel is around 60 to 70 min.

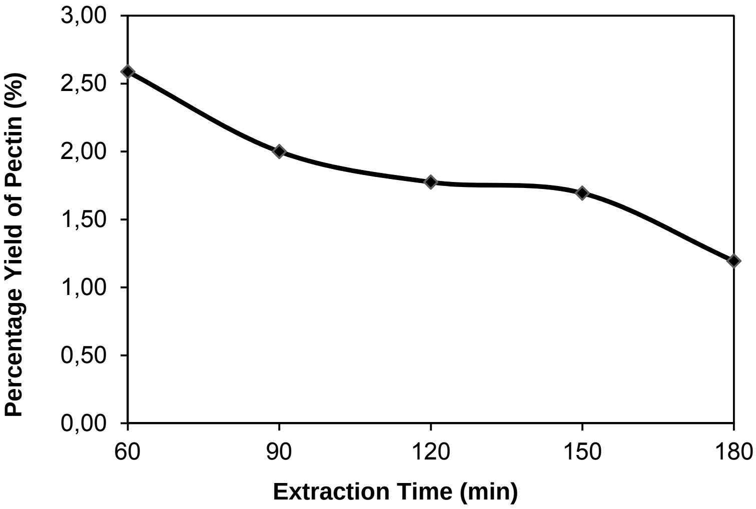

Figure 3: Graph showing the effect of extraction time to the yield of pectin (%) at constant extraction temperature 90 °C and solution pH 1.5.

4. Conclusions

As the conclusion, higher temperature is good when extracting pectin however very high temperature could also induce digestion of pectin during the extraction process. Pectin yield is generally higher at lower solution pH. Extended extraction time could also affects negatively on the yield of pectin. The best extraction conditions obtained in this study is temperature 90 °C, extraction time 60 min and solution pH 1.5 which yields 2.59 % of pectin. The highest yield of pectin extracted was low compared to a study by Abang Zaidel et al. (2014) which obtained the highest yield of 74.43 %. Foo et al. (2016) reported that 19.17 % of pectin was extracted from sweet potato using hydrochloric acid while extraction of pectin using citric acid by Hamidon and Abang Zaidel (2016) yields higher pectin (65.8 %). This shows that the yield of pectin extracted is also highly dependent on the type of acid used as extracting solvent. It is also wise to consider the variation of sample of the substrate used to extract pectin when comparing the yield obtained in the study.

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