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Effect of process parameters on pectin extraction from dragon fruit (*Hylocereus polyrhizus*) peels via chemical and physical treatment

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ABSTRACT

Dragon fruit (*Hylocereus polyrhizus*) peel was found to be one of the potential sources of pectin. The combination of physical and chemical treatments namely mild ultrasound, stirring and citric acid has been applied with the aim to get the high yield of pectin during extraction. The influence of several process namely agitation, temperature, time, pH and liquid solid ratio during extraction process were investigated. The one factor at a time (OFAT) method was used to determine the possible best levels of each factor during extraction. The pectin obtained from these experiments was compared in term of yield based on dry weight. The highest yield of pectin 42.5% (w/w) was obtained when the extraction was carried out at agitation, temperature, time, pH and solid liquid ratio of 250 rpm 70ºC, 120 min, pH of 1.5 and 1:10 (w/v), respectively.

INTRODUCTION

Industries of pectin production receive a rapid growth due to high demand in food and pharmaceutical industries. Williat *et al*., (2006) reported that worldwide annual consumption of pectin is estimated 45 million kilogrammes. Pectin is known as high-value functional food ingredient widely used in food industries as thickening, gelling and emulsifying agents for jams, drinks and dairy product. It is also used in fillings, sweets and as a source of dietary fibre (Srivastava and Malviya, 2011). Although pectin did not contribute significantly to the nutrition, but pectin is a natural part to the human diet which good to the human intestine. Furthermore, consumption of pectin has been shown to stabilize blood pressure and reduce serum cholesterol level and glucose level (Srivastava and Malviya, 2011,Rubio-Senent *et al*., 2015,Kim, 2005).

According to the previous work, source of pectin were from apple (Wikiera *et al*., 2015), citrus fruit (Yeoh *et al*., 2008), lemon pulp (Rashad *et al*., 2011), kiwifruit (Yuliarti *et al*., 2015), passion fruit peel (Pinheiro *et al*., 2008), sugar beet pulp (Li *et al*., 2015), grape pomace (Minjares-Fuentes *et al*., 2014) and some other sources. This study was focusing on utilizing dragon fruit (*Hylocereus polyrhizus*) peels as source of pectin. *Hylocereus polyrhizus* (*H.polyrhizus*) has red pulp with margenta skin received a great attention as added-value to natural colourant, as nutritional value for food product besides, it also has good antioxidant activity (Muhammad *et al*., 2014). The fruit peels and pomace which has been used for the production of natural coloring agent and juice could led to the accumulation of the peels as waste material. So, extraction of the valuable product which is pectin gives the additional value to the disposed peel.

Pectin is a complex set of polysaccharides that are naturally abundant in the non-woody parts of almost terrestrial plants. Pectin concentrations is found higher in the plant middle lamella and gradually decrease driving towards plasma membrane. These polysaccharides could constitute by at least 17 monosaccharides

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which galacturonic acid is the major one, followed by either galactose or arabinose and this sugar are concentrated in different structural elements which known as pectic polysaccharides (Yapo, 2009). Several of pectic polysaccharides can be detected in the plant cell wall include: homogalacturonan (HG), xylogalacturonan (XG), apiogalacturonan (AG), rhamnogalacturonan I (RGI) and rhamnogalacturonan II (RGII).

There is various method has been used in pectin extraction in order to enhance the efficiency, reduced the extraction time and increase yield. Generally, pectin extraction can be carried out in three different ways including chemical, physical, enzymatic treatment or also by the combination of these methods (Panouillé *et al*., 2006). Current extraction process has focused on high reproducibility with low cost process simultaneous to emphasis the concept of environment and human-friendly method. Conventional method was using hot acid extraction but due to exposure to the corrosive environment in industries, researcher has given attention to the physical method of ultrasonic and microwave assisted extraction (Bagherian *et al*., 2011). However, in order to maintain physicochemical characteristic of the pectin extract as pH value give the high impact to the characterization of pectin, the combined method of the physical and chemical method has been introduced like chemical ultrasound assisted (Minjares-Fuentes *et al*., 2014).

The goal of this study is to study the best condition of the pectin extraction from *H. polyrhizus* peels using bath sonication, agitation with propeller and citric acid as the extraction solvent. Theoretically, extraction of pectin involves two types of physical phenomena: diffusion through the cell walls and washing out the cell content once the walls are broken. In order to give the best of extraction process, combined method of ultrasound technique to help cell broke (Minjares-Fuentes *et al*., 2014), stirring with propeller stirrer to diffuse the cell content into the solvent which is citric acid solution. Citric acid was chosen as it is known to give less damaging effects on the extracted pectin (Kurita *et al*., 2008, Yapo, 2009). The extraction process was performed by one factor at a time (OFAT) method considering the extraction in different agitation rate, temperature, time, pH and liquid solid ratio (LSR). The pectin extracts were measured in term of cumulative yield.

Methodology:

2.1 Materials and chemicals:

Dragon fruits with red epicarp and red pulp (*H. polyrhizus*) were obtained from a fruit farm in Gambang, Pahang. Citrus pectin was purchased from Sigma-Aldrich (USA). All chemicals (≥99.5% citric acid and 95% ethanol) were purchased from Sigma–Aldrich (USA) and were used as received or otherwise stated.

2.2 Sample preparation:

The whole peel (epicarp) of each dragon fruit was separated from the pulp immediately after the arrival of the fruits in the laboratory conditioned at 25ºC. The peels were chopped into 1 cm^2 pieces using a stainless steel knife and were kept in -20ºC freezer until used.

2.3 Pectin extraction:

Pectin was extracted using citric acid as extraction solvent and assisted with propeller and sonicator bath with internal dimensions of 24.0 cm x 13.6 cm x 10.0 cm , frequency of 132 kHz and power output of 80 W. The extraction procedure was performed using one factor at a time (OFAT) which itself provide 31 experiments in every run. The evaluated parameters in this experiment were tabulated in Table 1.

Table 1: Variables of OFAT analysis.

Parameter	
Agitation, rpm	50
	100
	150
	200
	250
	300
	350
Temperature, ºC	30
	40
	50
	60
	70
	80
Time, h	30
	60
	90
	120
	150
	180
	210

	240
pH	1.5
	2.5
	3.5
	4.5
	5.5
LSR (w/v)	1:5
	1:10
	1:15
	1:20
	1.25

The hot acid extract was filtered through metal sieve. The filtrate was cooled to 4ºC. Pectin-containing aqueous extract was coagulated by using 1: 2 samples to 95% ethanol at 4ºC and left for 4 h to allow the pectin reach equilibrium colloid-liquid state and float on the surface (Faravash and Ashtiani, 2007). The gelatinous pectin flocculants was skimmed off. The extracted pectin was purified by washing in 200 ml 95% ethanol at room temperature and then pressed on a nylon cloth to remove the residual acid. Extracted pectin was dried to a constant weight in an oven at 50ºC. Each of extraction conditions was carried out in triplicates.

2.4 Yield:

The dried pectin obtained was calculated using equation (1) and (2) respectively (Muhammad *et al*., 2014):

$$Y_f(\%) = 100[{}^{P}/_{DF}] \quad (1)$$

$$Y(\%) = Y_f/(100 - moisture\ content\ of\ DF) \quad (2)$$

where Y_f is the extracted pectin yield on fresh and Y on dry weight basis, P is the amount of dried extracted pectin in g and DF is the weight of fresh dragon fruit peel (g).

RESULTS AND DISCUSSION

Percentage of pectin yield was monitor regards to the influence of agitation rate. This parameter was varied between 50 and 350 rpm. Figure 1 shows the effect of agitation rate on pectin yield. The results obtained shows that the variation of agitation rate to a certain point showed a considerable increase in pectin yield. The yield of pectin keeps on increase with further increase of agitation rate. This situation is due to the fact that increase stirring rate may reduce the thickness of the diffusion layer which can enhanced the extraction process. However, the influence of agitation rate was less significant at 250 rpm to 350 rpm with the yield increase of about 1.03% (w/w) only. A medium agitation rate was set at 250 rpm throughout the studies as not much different on pectin yield observed at its high level. In addition, the lower agitation rate is more beneficial as energy consumed is lesser.

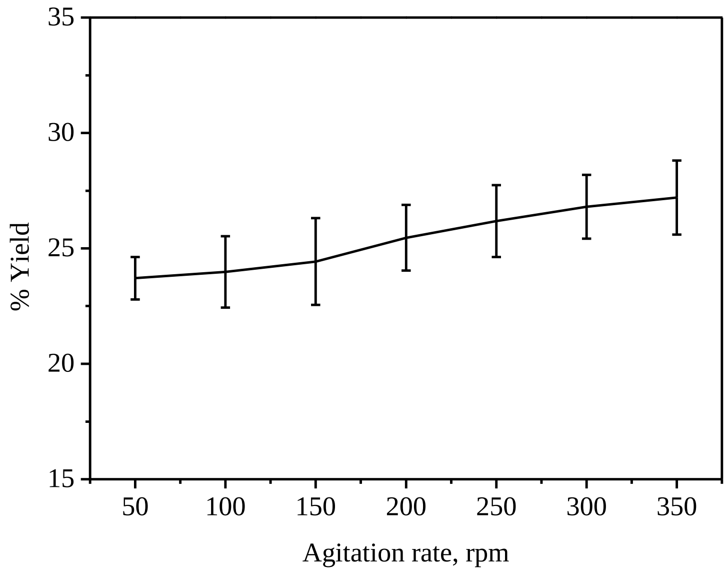

Fig. 1: Effect of different agitation rates on pectin yield. The extraction time, temperature, pH and LSR were fixed at 120 min, 80ºC, pH 2.0 and 1:5, respectively.

A set of temperature ranging from 30-80ºC were applied to study the influence of temperature on pectin extraction from dragon fruit peels. As shown in Figure 2, the yield of pectin increase with increase in temperature from 30-70ºC and highest pectin yield obtained was 34.8% (w/w). The yield decreased on further increase of temperature to 80ºC reach to 26.18% (w/w). The decrease of pectin yield is due to the decrease of power output from the sonicator bath where at lower ultrasound intensity with high temperature the cavitations will occur due to the elevation of the vapor pressure of the heated liquid (Raso *et al.*, 1999). Cavitations will lead to the reduction of shear forces hence reduced the recovery of pectin during extraction (Xu *et al*., 2014). A significant amount of pectin was obtained within temperature ranging from 30-70ºC and thus the possible optimum temperature was chosen to be at 70ºC for subsequent experiments.

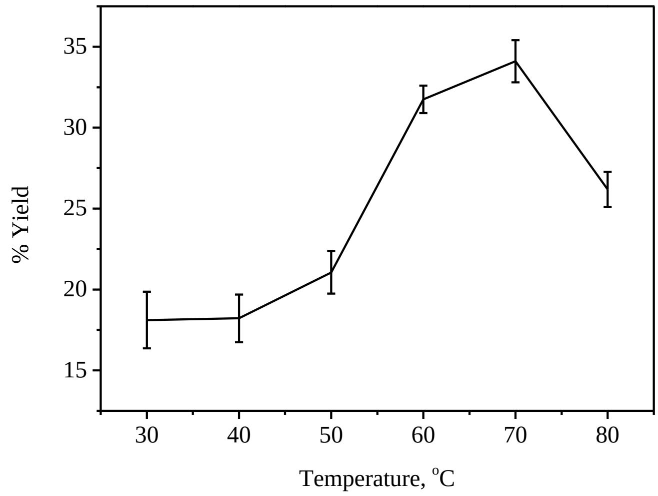

Fig. 2: Effect of different extraction temperature on pectin yield. The extraction agitation rate, time, temperature, pH and LSR were fixed at 250 rpm, 120 min, pH 2.0 and 1:5, respectively.

Pectin was recovered from dragon fruit peels at various levels of reaction times varying from 30-240 min in order to get the maximum pectin yield. The influence of reaction time on pectin extraction at fixed temperature 70ºC, agitation rate 250 rpm, pH 2.0, LSR 1:5 were shown in Figure 3. In the first 60 min of extraction, the pectin yield had shown no significant increase with almost stagnant value of about 26.5% (w/w). Although, there was no significant increase but the yield of pectin obtain at that time is comparable to the previous study reported by Muhammad K. *et al*. (2014) and Xu Y. *et al*. (2014). When reaction time was increased from 60 to 120 min, the yield linearly increased with a maximum yield value of 34% (w/w). But, when the reaction time was further increased, the depletion on the yield occur which may due to the thermal degradation of the soluble pectin when expose to the long extraction time at high temperature (Woo *et al*., 2010). This study shown that 30 -150 min of reaction time was found to be the most suitable range for further study by FFD for screening the significant variables to achieve the highest pectin production.

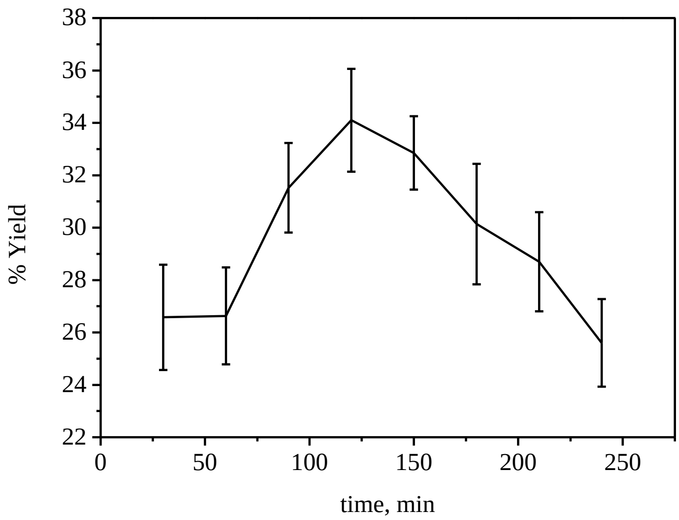

Fig. 3: Effect of different extraction time on pectin yield. The agitation rate, temperature, pH and LSR were fixed at 250 rpm, 70ºC, pH 2.0 and 1:5, respectively.

Different pH values ranging from 1.5-5.5 were chosen to find out the best pH condition. Figure 4 represent the effect of pH on pectin extraction at 120 min, agitation rate 250 rpm, and temperature 70ºC, LSR 1:5. The highest yield of pectin extract was 36% (w/w) at pH values 1.5. Extraction in acidic condition at low pH gives the best value to obtained high pectin yield. This might due to the used of citric acid that gives less damaging effect on the pectin extraction (Yapo, 2009). Furthermore, extraction in an acid medium with pH less than 2.0 gives the stable linkages between galacturonic acid residues and increase the hydrolysis of pectin neutral sugar thus enriched the galacturonic content in pectin produced (Yapo, 2009, Minjares-Fuentes *et al*., ,2014). Although there are not much different among all those five pH value used but the yield of pectin can be seen increase quite significant as the pH decrease from pH 3.5 to pH 2.5 which is 0.62% compare to pH 4.5 to pH 3.5 which is 0.09% only. However, pH 2.0 was chosen throughout the study as there is only slight different on the yield between pH 1.5 and pH 2.5 which is 0.12% only.

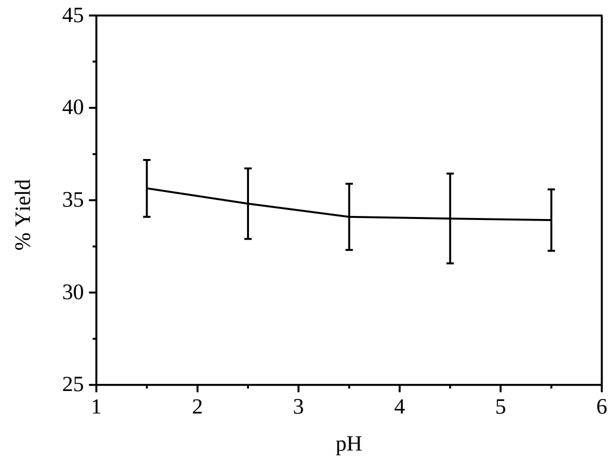

Fig. 4: Effect of different extraction pH on pectin yield. The agitation rate, temperature, time and LSR were fixed at 250 rpm, 70ºC, 120 min, and 1:5, respectively.

There were 5 different levels of liquid solid ratio (LSR) ranging from 5-25 ml/g of citric acid solution/ dragon fruit peel. It was observed that a maximum pectin yield of 42.5% (w/w) was obtained with LSR 10 ml/g. The yield was increased firstly and gradually decline with increasing LSR probably due to increasing of dissolving capacity when LSR is increased but reduced the separation ability of pectin from the solution (Li *et al*., 2015). As for the other reason, when the LSR increase from 10-25 ml/g, the degradation of pectin increase with the decrease of pectin concentration in the solution. This was due to the low content of raw material that provided less protection for the dissolved pectin and facilitated to the degradation of pectin (Xu *et al*., 2014) Due to pectin is type of natural polymer, this situation support the reason of the increase of the degradation rate polymer with decrease of polymer concentration (Kanwal *et al*., 2000).

Overall experiment has shown that dragon fruit peels contain higher amount of pectin with 42.5% (w/w) of pectin yield compared to the extraction of dragon fruit peels from previous study (Muhammad *et al*., 2014) and also compared to some others source from the previous study (Minjares-Fuentes *et al*., 2014). The combination of physical and chemical treatment in this study showed a high efficiency for the extraction of pectin which regard to the percentage of pectin yield.

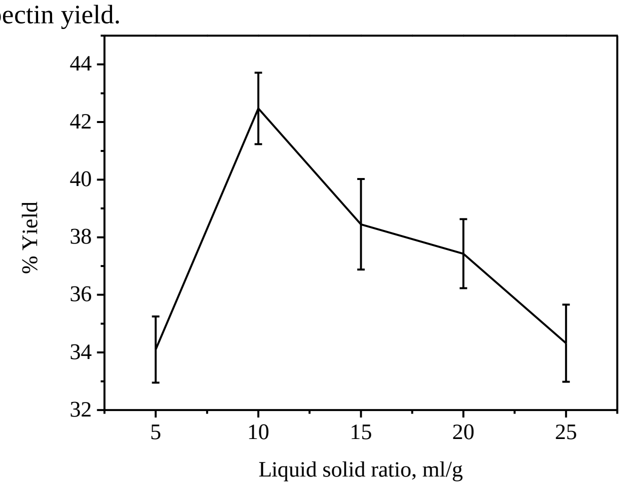

Fig. 5: Effect of different extraction LSR on pectin yield. The agitation rate, temperature, time and pH were fixed at 250 rpm, 70ºC, 120 min and pH 2.0, respectively.

Conclusion:

This study showed technique applied in this study provides high extractability of pectin from *H. polyrhizus* peels with different extraction parameters using OFAT analysis. The highest yield of pectin 42.5% (w/w) was obtained when the extraction was carried out at agitation, temperature, time, pH and solid liquid ratio of 250 rpm 70ºC, 120 min, pH of 1.5 and 1:10 (w/v), respectively. However, the physic-chemical characteristics of the pectin should be perform to further understand its benefit for technological and health aspect.

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