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Optimization of Pectin Extraction from Peel of Dragon Fruit (*Hylocereus polyrhizus*)

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ABSTRACT

Dragon fruit is a popular fruit grown in Malaysia. In order to increase profits for dragon fruit growers and processors, dragon fruit peels, a by-product of dragon fruit processing, were investigated as a source of pectin. Pectin extraction was optimized from this by-product. Pectin was extracted under various conditions: pH 2 to 5; Ethanol Ratios (ER) from 1: 0.5 to 1: 3.5 and extraction periods 30 to 120 min. The highest yield was obtained after 120 min of extraction at pH 3.5, with ER of 0.5. Preliminary characterization of pectin, in terms of degree of esterification (DE) was also carried out in order to investigate the influence of different extraction conditions on the chemical composition of the extracts. Pectin with high DE was obtained at pH 5. The results imply that dragon fruit peels could be a substantial source in food production.

Key words: Pectin yield, degree of esterification, pH, ethanol ratios, citric acid

INTRODUCTION

Pectin or pectic substance, is among the most complex macromolecules found in nature (O'Neill *et al*., 2004). It is a group of polysaccharides that are rich in galacturonic acids (Benen *et al*., 1996). These polysaccharides with extremely diverse set of biopolymers, act as structural elements found within the primary cell wall in dicotyledons (Voragen *et al*., 1995). Pectic substances can be divided into four main categories, namely protopectin, pectic acid, pectinic acid and pectin (Alkorta *et al*., 1998). An important functional property of these biopolymers is their ability to gel in aqueous solutions (Yokoi *et al*., 2002).

In food industry, pectin has been widely applied as thickening, gelling and emulsifying agents for jams, soft drinks, fish and meat products, fruit juice, desserts and dairy products (May, 1990; Ralet *et al*., 2005). Apart from that, pectin is useful in medicinal applications, in which it helps in lowering serum cholesterol level, removing heavy metal ions from the body, stabilizing blood pressure and restoring intestinal functions (Voragen *et al*., 1995) and weight reduction (Jitpukdeebodintra and Jangwang, 2009). These applications directly account for the substantial consumption of pectin worldwide.

Thereafter, with the growing demand for products in food science and nutrition, cosmetics and pharmaceutics (Pilnik, 1990), the worldwide pectin consumption grows constantly and has already exceeded 20,000 tons a year (Ptichkina *et al*., 2008). Generally, the peels and pomace of fruit are disposed as industrial waste or being used for animal feeding, yet pectins have been reported to be

a potential source of pectins (Kim *et al*., 2004; Pedroza-Islas *et al*., 1994; Berardini *et al*., 2005; Pinheiro *et al*., 2008; Kliemann *et al*., 2009). Dried lemon or orange peel, citrus albedo and apple pomace are the main raw materials that have been utilized in pectin production all around the world (May, 1990; Liu *et al*., 2006; Masmoudi *et al*., 2008; Rezzoug *et al*., 2008).

Currently, dragon fruit (*Hylocereus polyrhizus*) or pitaya is a popular fruit in Malaysia due to its attractive appearance and nutritious value. It can be processed into range of food products, such as juice, jam, syrup, ice cream, yogurt, jelly and candy (Wybraniec *et al*., 2001). Thus the dragon fruit peels instantaneously become a convenient and attractive source for the extraction of pectin.

The main objectives of this study were to define optimize conditions for dragon fruit peel pectin extraction in term of yield and preliminarily characterize the obtained products.

MATERIALS AND METHODS

This study was carried out in Universiti Tunku Abdul Rahman from January to December, 2008.

Raw materials: Fresh dragon fruits were purchased from local market. The peels were cut into small pieces and dried at 55°C in oven for 48 h.

Pectin extraction method: Pectin extractions were performed in triplicate. About 10 g of dragon fruit peel was blended with 250 mL distilled water. The extraction of pectin was conducted under seven different pH environments, i.e., 2, 2.5, 3, 3.5, 4, 4.5 and pH 5. The mixture was then acidified by different volumes of 40% citric acid (115, 100, 65, 37, 23, 10 and 0 mL) in order to achieve the desired pH. The acidified mixture of blended peels of dragon fruit was then heated at 60°C. The extraction was carried out in a time-dependent manner at three different incubation times, 30, 60, 120 min in the water bath. The volume of the mixture was remained constant during extraction.

After incubation, the resulting mixture was filtered twice. In the first filtration, the mixture was passed through an ordinary screen with 1 mm mesh size equipped with a layer of cheesecloth. This was followed by the initial filtrate being filtered through a Buchner funnel, equipped with a Whatman No. 1 filter paper and connected to a vacuum pump. Then, the final extract was evaporated to one-fifth of its initial volume by using rotary evaporator (Pagan and Ibarz, 1999; Pagan *et al*., 2001). The concentrated extract was subsequently filtered by a Whatman no. 1 filter paper to remove any excess insoluble impurities completely.

Following that, the concentrated pectin extracts were precipitated with 95% ethanol. The One volume of dragon fruit extract was added with various volumes of ethanol. The ratios of dragon fruit extract to ethanol volume (ER) were 1: 0.5, 1: 1.5, 1: 2.5 and 1: 3.5. The mono-phase system of extraction mixture turned into a two-phase system, which was then kept at room temperature for 24 h to allow pectin floatation (Kalapathy and Proctor, 2001) and to facilitate the equilibrium between colloid-liquid states.

The precipitated solution was centrifuged for 15 min at 9000 rpm to separate the precipitated material totally. Thereafter, the precipitated materials were washed with 45% ethanol to eliminate impurities such as monosaccharide and disaccharide (Pagan and Ibraz, 1999; Pagan *et al*., 2001). The solution was centrifuged again at 9000 rpm for 15 min to separate pectin substances from liquid phase. The washing and centrifuging operations were repeated few times to obtained a completely clarified upper layer of liquid phase. Each pectin samples that was subjected to different extraction parameters, were freeze-dried.

Pectin characterization: Each of the dried pectin was weighed. Pectin yield was calculated as mass of dried extracted pectin/ mass of dried dragon fruit peel.

The degree of esterification (DE) of each pectin samples, extracted under varying conditions, was determined by using the method of Mizote *et al.* (1975) with minor modifications. About 50 mg of the powder was moistened with 65% isopropanol and dissolved in 10 mL of distilled water. Then, the resulting pectin was titrated with 0.1 N NaOH solution (a mL) to pH 7.5. The solution was added with 30 mL of 0.1 N NaOH and left for 30 min, followed by the addition of 30 mL of 0.1 N sulfuric acid. The pectin solution was then titrated again with 0.1 N NaOH (b mL) to pH 7.5. Degree of esterification (DE) of the each extracted pectin was calculated by using the formula of DE (%) = (b/*a b*)×100% (Jiang *et al.*, 2005). Analysis of each were performed in duplicate.

RESULTS

Yield of pectin: The pectin yield (%) with various parameters is shown in Fig. 1a-c. According to Fig. 1a, the highest pectin yields were obtained at pH 3.5. At this optimum pH, the yield was peaked at the ER of 0.5 regardless of extraction time. The yield ranged from 10.40, to 16.76%. This pH also resulted high yields for ER = 1.5 and 2.5. It was observed that at optimum pH of 3.5, 120 min of extraction gave the highest percentage of pectin yield. Most of the pectin yield was found that to be relatively higher at 120 min of extraction time as compared with 30 min and 60 min at different ERs (Fig. 1a). Apart from that, pectins extracted at pH 5 were slightly less. The yields were 9.83, 11.56 and 13.87% at extraction time of 30, 60 and 120 min, respectively for ER = 0.5 (Fig. 1b). However, at pH 2, ER of 3.5 was needed in order to obtain high yield of pectin (Fig. 1c). Pectin yields obtained at other pH values were unsatisfied (Data not shown). The influence of ER on the trend of pectin yield became inconsistent when pH was above 3.5 (Data not shown).

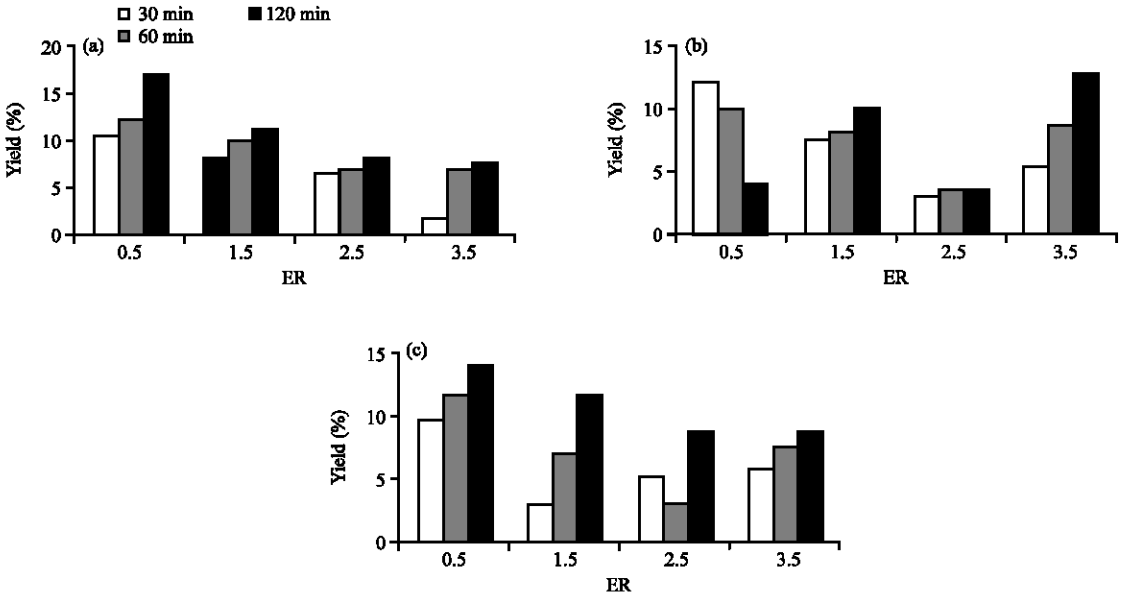

Fig. 1: (a) Pectin yield (%) at pH 3.5, different ERs and extraction times; (b) pectin yield (%) at pH 5, different ERs and extraction times; and (c) pectin yield (%) at pH 2, different ERs and extraction times

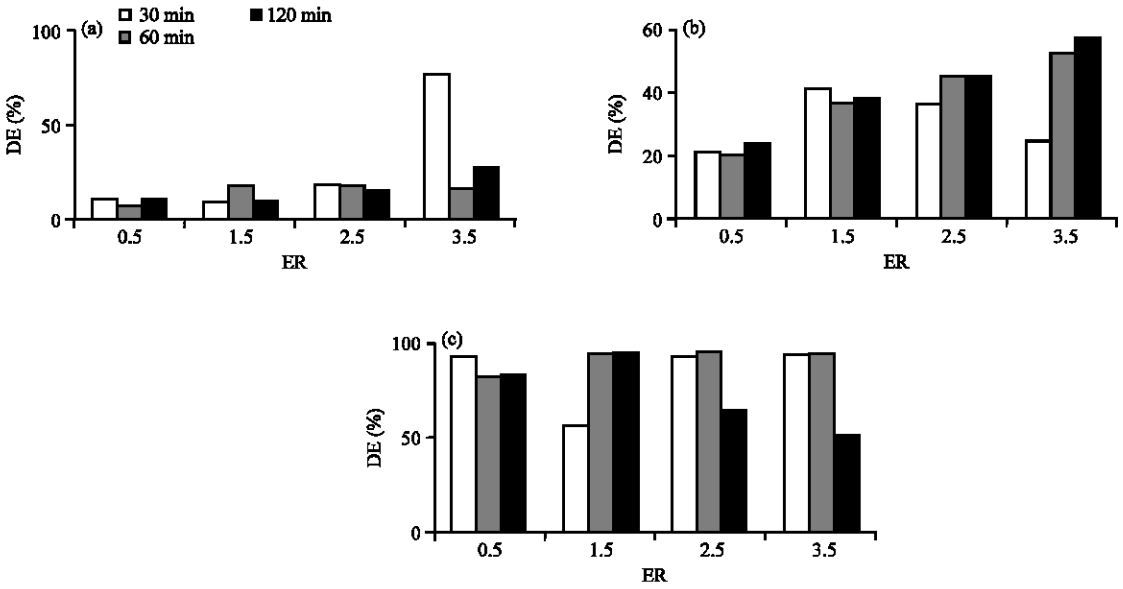

Fig. 2: (a) Degree of esterification (% DE) of pectin extraction at pH 2, different ERs and extraction times; (b) degree of esterification (% DE) of pectin extraction at pH 3.5, different ERs and extraction times; and (c) degree of esterification (% DE) of pectin extraction at pH 5, different ERs and extraction times

Degree of Esterification (DE) of extracted pectin: With reference to Fig. 2a, it could be deduced that when ER = 0.5, the lowest DEs which were obtained at pH 2, ranged from 6.52% to 10.49%. Optimum pH for extraction yield resulted in 19.07 to 56.60% DE (Fig. 2b). On the other hand, DE values obtained at pH 5 were comparatively high, ranging from 81.78 to 92.10% (Fig. 2c). It was found that high DE could be obtained with ER = 3.5 in high acidic condition (pH 2) (Fig. 2a) and ER = 0.5 in low acidic condition (pH 5) (Fig. 2c). The obtained results indicate that DE of pectin is proportional to the increase of pH.

DISCUSSION

Dragon fruit peels as a potential pectin source: At present, most of the main pectin producers utilize citrus peel or apple pomace, both by-products from juice (or cider) manufacturing as raw materials. Apple pomace contains 10 to 15% of pectin on a dry matter basis while citrus peel contains of 20 to 30% (May, 1990). Present results indicate that pectin yield form dragon fruit peels (~17%) (Fig. 1a) is equivalent with apple pomace and slightly lower than citrus peel. In fact, dragon fruit peels contain higher amount of pectin than other food industry by-products, such as cocoa husks (Mollea *et al.*, 2008), peach pomace (Faravash and Ashtiani, 2008) and sunflower head residues (Iglesias and Lozano, 2004).

Influence of pH on pectin yield: pH is an important factor to be controlled in pectin extraction (Pagan and Ibarz, 1999; Joye and Luzio, 2000; Kalapathy and Protor, 2001; Pagan *et al.*, 2001; Levigne *et al.*, 2002). Extraction with strong acid gives lower pectin yields whereas high yields can be obtained when the strength of the acid is weak (Kalapathy and Proctor, 2001). Generally, pectin gel gives the highest gel strength at pH 3.2 to 3.5 (Thakur *et al.*, 1997; Hoefler, 1999) and the

optimum pH for pectin extraction falls between pH 2 to 2.8 (Masmoudi *et al.*, 2008; Mollea *et al.*, 2008; Wai *et al.*, 2009). Present study indicated that pH 3.5 is the most suitable pH for obtaining high yield of pectin from dragon fruit peel which is close to this optimum range. However, the optimum pH in present study contradicts the results of Wang *et al.* (2007) and Yujaroen *et al.* (2008) which reported an optimum pH of 1.01 and 6, respectively.

Influence of ER on pectin yield: Pectin precipitation is governed by the addition of suitable amount of alcohol (ER) into pectin solution, which can increase the yield (Arthey and Ashurst, 1996). The optimum pectin precipitation pH (p-pH) ranged from 2.8 to 3.5 and the minimum solubility of pectin in solution is obtained at p-pH of 3.5 (Kalapathy and Proctor, 2001). Present results agree to the above findings.

Faravash and Ashtiani (2008) found that the precipitation of pectin could not occur with very low ER and the maximum extraction yield was obtained at the ER of 1.5. Contrary to the finding of Faravash and Ashtiani (2008), present results indicated that high yield could be obtained at low ER of 0.5 if precipitation of pectin occurred at the optimum pH range suggested by Kalapathy and Proctor (2001). In fact, it was observed that increase of ER could lead to a drop of pectin yield at this pH range in our study.

Influence of extraction time on pectin yield: Previous studies have shown that the highest yield of pectin was recorded under the highest temperature and lowest pH (Pagan and Ibarz, 1999). Alternatively, the longer the extraction time, the higher the pectin obtained under a constant pH and temperature (Pagan *et al.*, 2001; Mollea *et al.*, 2008). Present results provided an idea that majority of the highest yields of pectin could be obtained at 120 min, thereby displaying an agreement to the discovery of Pagan *et al.* (2001) and Faravash and Ashtiani (2008). However, some studies showed that optimum pectin yield could be obtained in 1 h (Mollea *et al.*, 2008; Wai *et al.*, 2009) or even 10 min (Kliemann *et al.*, 2009).

Influence of pH on DE of extracted pectin: The pH is an important parameter to be considered in pectin extraction (Pagan *et al.*, 2001). In this study, it was found that the increase in pH resulted in the increase of DE. Present results contradict the finding of Pinheiro *et al.* (2008) that lower acid concentration results in production of pectin with high degree of esterification. From application point of view, only pectins with DE <60% are usually employed in the food industry (Ptichkina *et al.*, 2008) where they can be used as gelling agent in low sugar products, such as low calories jams and jellies (Iglesias and Lozano, 2004). Hence, this study suggested that pH 3.5 favored the extraction of pectin suitable for food industry without sacrificing the yield.

Influence of ER on DE of extracted pectin: Generally, high DE could be obtained at different conditions in this study. The DE was directly correlated with the increase of ER when pectin was extracted at the optimum pH of 3.5 for 60 and 120 min. This correlation was not observed at other conditions. Thus, our results agree to the finding of Kalapathy and Proctor (2001) but partially contrary to the work of Faravash and Ashtiani (2008) that ER strongly affects DE.

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