

## Physico-chemical properties of spray-dried red pitaya (*Hylocereus polyrhizus*) peel powder during storage

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### Abstract

The physico-chemical properties of spray-dried pitaya peel powders kept at accelerated ( $45 \pm 2^\circ\text{C}$ ) and room temperature ( $28 \pm 2^\circ\text{C}$ ) for 14 weeks and 6 months, respectively were evaluated. Changes in physico-chemical properties of the peel powder were used as indicators of stability, while changes of the betacyanin pigment retention was used to calculate the shelf-life of the powder. Storage temperatures significantly ( $p < 0.05$ ) affected all the studied parameters and Hunter a value had the most significant change. The pigment retention of peel powder was approximately 87% at  $45^\circ\text{C}$  and 89% at room temperature storage. Degradation of betacyanin pigment in the powder followed the first order reaction kinetics with the half-life ( $t_{1/2}$ ) of approximately 76 weeks at  $45^\circ\text{C}$  and 38 months at  $28^\circ\text{C}$ . The spray-dried pitaya peel powder had a solubility of 87 to 92% and low in powder hygroscopicity. The final  $A_w$  of the powder did not exceed 0.6 for both storage temperatures.

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### Keywords

Pitaya peel powder  
Storage stability  
Betacyanin  
Kinetic  
Half-life

### Introduction

Dragon fruit or red pitaya (*Hylocereus polyrhizus*) is farmed in many parts of the world such as in South East Asian region, Bangladesh, China, Australia and Israel, although the fruit is not native to those regions. The fruit is popular because of the numerous nutritional claims reported. In Malaysia, red pitaya is processed into cordials and straight juices apart being eaten as a fresh fruit. The fruit peels are presently discarded during the manufacture of the fruit. Our previous study (Jamilah *et al.*, 2011) showed that the Malaysian pitaya peel was high in pectin, contained a good ratio of insoluble to soluble fibre and had significant amount of betacyanin pigment ( $150.46 \pm 2.19$  mg/100 g). The high content of the betacyanin in the peel was also reported by Harivaindaram *et al.* (2008), Wu *et al.* (2006), and Stintzing *et al.* (2002). Therefore, pitaya peel could be a potential source of functional ingredient containing fibre and betacyanin. However, no attempt has been reported to convert this perishable peel into powder.

Betacyanin stability in several spray-dried fruit powders such as that of *Opuntia stricta* cactus pear juice powder (Obon *et al.*, 2009), *Opuntia ficus-indica* cactus pear juice powder (MoBhammer *et al.*, 2006), *Opuntia streptacantha* cactus pear juice powder (Rodriguez-Hernandez *et al.*, 2005) and *Amaranthus*

*betacyanin* extract (Cai and Corke, 2000) had been reported. However, the stability of the betacyanin pigment in spray dried pitaya peel powder has not been reported. The rate constant and half-life ( $t_{1/2}$ ) of betacyanin degradation in pitaya juice during heating at  $85^\circ\text{C}$  was reported by Herbach *et al.* (2004). The half-life of betacyanin in spray-dried pitaya peel powder may be different from the pitaya juice due to the higher temperature of drying and the overall nature of peel matrix.

Betacyanin pigments are generally susceptible to pH, temperature, oxygen, light and water activity. Kinetic modelling which described the reaction rate as a function of storage time was used to predict changes in a food material during storage (Van Bockel, 1996). Reports on the conversion of peels into value added products are still scarce. Hence, it is important that this database is strengthened. Therefore, this work was attempted with the objective of determining the effect of storage temperature and time on physico-chemical properties of spray-dried pitaya peel powder.

### Materials and Methods

#### Pitaya peel preparation and spray drying

Spray-dried pitaya peel powder was produced according to Jamilah *et al.* (2013). Commercially

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matured red pitaya fruits (*Hylocereus polyrhizus*) were purchased from a farm and transported immediately to the laboratory. Upon arrival, peels were separated manually from the fruits. Peels were homogenized in a Waring blender with water added to facilitate the blending. The homogenate was then filtered to remove pieces of peels which were not well homogenized. Maltodextrin DE10 at 15% was then added to the filtrate. The filtrate was spray-dried in a pilot scale spray dryer (Niro A/S, GEA, Germany) at an inlet air temperature of 165°C and an outlet air temperature of 80°C. The powder produced was collected for the following study.

#### *Storage study*

Low density polyethylene (LDPE) bags measuring 125 x 210 mm (length x diameter) was obtained from a local retail supplier for the storage study. The packaging film was 80 µm thick and had an oxygen transmission rate (OTR) of 60 cc/100 in<sup>2</sup>/24 hr (measured by Oxygen Permeation Analyzer 8000 Illinois Instrument). Approximately 150 g of the powder was packed in each LDPE bag. The bags were then heat-sealed and kept at accelerated (45 ± 2°C; 38% RH) for 14 weeks and sampling was done every forth-night. The other lot was kept at room temperature (26 ± 2°C, 50-70% RH) and sampled on monthly basis until 6 months. All analyses were carried out in triplicates.

#### *Microstructure determination using Scanning Electron Microscopy (SEM)*

The microstructure of the spray dried pitaya peel powder was observed using a scanning electron microscope (SEM) (JEOL JSM-6400, Tokyo, Japan). Peel powder was first attached to SEM stubs using a double-sided adhesive tape. It was then coated with a thin layer of gold and examined by the SEM operated at an accelerating voltage of 15-20 kV (Obon *et al.*, 2009).

#### *Moisture content determination*

The moisture content of the sample was determined by the oven method at 120°C (AOAC, 2007). The weight loss (%) of the powder was recorded after a constant weight was obtained and this was calculated as the moisture content of the powders.

#### *Determination of water activity ( $A_w$ )*

The  $A_w$  of the powder was determined using an Aqua-lab water activity meter (Model CX2, Decagon Devices, Pullman, WA). The water activity meter was calibrated using distilled water according to the manufacturer's guide.

#### *Determination of Hygroscopicity*

Hygroscopicity of pitaya peel powder was measured according to Cai and Corke (2000). A total of 2 g of peel powder was weighed into a pre-weighed petri dish and placed in an airtight desiccator filled with saturated solution of Na<sub>2</sub>SO<sub>4</sub> (81% RH) at 25°C for 1 week. The hygroscopicity of the peel powder was then calculated by weight difference.

#### *Determination of Solubility*

The solubility of the peel powder was carried out according to the Cano-Chauca *et al.* (2005). The peel powder was weighed and homogenized by grinding 1 g of powder in 100 ml of distilled water in a Waring blender for 5 min at high speed. The solution was then centrifuged at 3000 × g for 10 min and the supernatant was collected. An aliquot of 25 ml of the supernatant was transferred to pre-weighed petri dishes and oven-dried at 105°C overnight. The solubility was calculated by weight difference and expressed as percentage.

#### *Hunter a color measurement*

The redness (Hunter a) of the powders was measured by a pre-calibrated Hunter Lab UltraScan PRO Spectrocolorimeter (Hunter Associate Laboratory Inc., Reston, USA). Powder was uniformly packed in the 50 mm glass optical cell before it was placed in the reflectance port for reading.

#### *Determination of Betacyanin Content*

Betacyanin content of spray-dried pitaya peel powder was quantified according to Wybraniec & Mizrahi (2002). Spray-dried peel powder was weighed and diluted with McIlvaine buffer (pH 6.5) to reach an absorption value of 1.0 ± 0.1. Diluted sample was filtered before performing spectrophotometric measurement. Quantification of betacyanins was carried out according to the following equation: [BC (mg/100 g) = (A × DF × MW × V × 100)/(ε × L × W)], where A was the absorption value at 538 nm corrected by the absorption at 600 nm, DF was the dilution factor, MW was molecular weights of betanin (550 g/mol), V was the pigment solution volume (ml), ε was the molar extinction coefficients of betanin (60,000 L mol<sup>-1</sup> cm<sup>-1</sup>), L was the path length of the cuvette and W was the weight of pigment powder in gram.

#### *Evaluation of kinetic modelling and betacyanin pigment storage stability*

Betacyanin pigment stability in pitaya peel powder was expressed in terms of degradation rate constant (*k*) and half-life value (*t*<sub>1/2</sub>) according to Cai *et al.* (1998). The pigment retention (%) in the

peel powder was calculated from betacyanin content measured at zero storage time ( $B_0$ ) and at x time ( $B_x$ ), expressed as pigment retention (%) =  $[(B_x) \times 10^2 / (B_0)]$ . Rate constant ( $k$ ) and half-life value ( $t_{1/2}$ ) were calculated using the regression analysis of  $\ln$  (pigment retention %) versus storage time when plotted on natural logarithmic scale. Degradation rate constants ( $k$ ) were obtained from the slope of the plot and the half-life was calculated as  $t_{1/2} = \ln 2 / k$ .

### Statistical analysis

Minitab statistical package version 14 (2004) was used for statistical analysis. Data obtained from storage analyses were subjected to analysis of variance (ANOVA), followed by the Tukey multiple comparison test to compare the difference among the mean values. Means and standard deviations were reported and the significance was defined at  $p < 0.05$ .

## Results and Discussion

### Microstructure of spray dried powder

The microstructure of the powder is as shown in Figure 1A and 1B. The spray-dried pitaya peel powder consisted of particles of various sizes in the range of 5 - 30  $\mu\text{m}$ . The particles were spherical but appeared shriveled. The shriveled appearance could be due to the slow drying rate during spray drying as commented by Tonon *et al.* (2008) that most powder particles remain shrunk with shriveled surface when inlet air temperature is low due to slow drying. Similar morphology was observed in microcapsules of *Amaranthus* pigment (Cai and Corke, 2000), *Opuntia mucilage* (León-Martínez *et al.*, 2010) and acai fruit (Tonon *et al.*, 2008).

### Moisture content

Moisture content critically influenced the product's storage stability (Yan *et al.*, 2008). The overall adsorbed moisture profiles of pitaya peel powder was significantly ( $p < 0.05$ ) different for both storage temperatures (Figure 2A and 2B). During accelerated storage, a general declining trend of the moisture contents was obtained (Figure 2A). A total of 1.25% moisture content drop was observed after 14 weeks of storage. The gradual decrease of the moisture content in the powder could be due to the outward diffusion of the moisture from the micro environment of the package to the chamber of the oven. On the contrary, a significant increase of 4.61% moisture content was observed in the peel powder kept at room temperature after 6 months of storage (Figure 2B). This could be due to the inward diffusion of moisture, which was eventually absorbed

Table 1. The changes of selected physico-chemical parameters in spray-dried pitaya peel powder throughout accelerated storage at 45°C for 14 weeks <sup>y, z</sup>

Storage time (weeks)	Parameter			
	$A_w$	Hygroscopicity (%)	Solubility (%)	Betacyanin content (mg/100g)
0	0.299 ± 0.008 <sup>A</sup>	27.63 ± 0.27 <sup>A</sup>	89.83 ± 0.51 <sup>A</sup>	64.66 ± 0.56 <sup>A</sup>
2	0.289 ± 0.003 <sup>AB</sup>	27.58 ± 0.10 <sup>A</sup>	92.09 ± 0.19 <sup>B</sup>	61.60 ± 0.64 <sup>B</sup>
4	0.286 ± 0.002 <sup>BC</sup>	27.47 ± 0.27 <sup>A</sup>	92.11 ± 0.68 <sup>B</sup>	60.74 ± 0.56 <sup>BC</sup>
6	0.281 ± 0.002 <sup>BC</sup>	27.83 ± 0.09 <sup>A</sup>	91.63 ± 0.51 <sup>B</sup>	59.40 ± 0.37 <sup>CD</sup>
8	0.280 ± 0.008 <sup>BC</sup>	27.61 ± 0.04 <sup>A</sup>	91.57 ± 1.38 <sup>AB</sup>	58.54 ± 0.42 <sup>DE</sup>
10	0.277 ± 0.002 <sup>BC</sup>	27.55 ± 0.19 <sup>A</sup>	91.26 ± 1.02 <sup>AB</sup>	57.57 ± 0.37 <sup>EF</sup>
12	0.275 ± 0.003 <sup>C</sup>	28.34 ± 0.08 <sup>B</sup>	91.47 ± 0.32 <sup>B</sup>	56.96 ± 0.56 <sup>F</sup>
14	0.276 ± 0.003 <sup>C</sup>	29.15 ± 0.17 <sup>C</sup>	91.76 ± 0.89 <sup>B</sup>	56.22 ± 0.21 <sup>F</sup>

<sup>y</sup> Averages of triplicate analysis.

<sup>z</sup> Values in the column with different uppercase (A-F) were significantly different ( $p < 0.05$ ).

Table 2. The changes of selected physico-chemical parameters in spray-dried pitaya peel powder throughout room temperature storage for 6 months <sup>y, z</sup>

Storage time (months)	Parameter			
	$A_w$	Hygroscopicity (%)	Solubility (%)	Betacyanin Content (mg/100g)
0	0.299 ± 0.008 <sup>A</sup>	27.63 ± 0.27 <sup>A</sup>	89.83 ± 0.51 <sup>A</sup>	64.66 ± 0.56 <sup>A</sup>
1	0.393 ± 0.005 <sup>B</sup>	27.79 ± 0.43 <sup>A</sup>	89.62 ± 0.21 <sup>A</sup>	62.46 ± 0.56 <sup>AB</sup>
2	0.466 ± 0.004 <sup>C</sup>	27.40 ± 0.02 <sup>A</sup>	88.15 ± 0.15 <sup>B</sup>	62.33 ± 0.97 <sup>B</sup>
3	0.488 ± 0.002 <sup>D</sup>	28.78 ± 0.41 <sup>B</sup>	88.54 ± 0.34 <sup>B</sup>	61.60 ± 0.73 <sup>BC</sup>
4	0.533 ± 0.005 <sup>E</sup>	29.53 ± 0.07 <sup>B</sup>	87.97 ± 0.59 <sup>B</sup>	59.77 ± 1.27 <sup>C</sup>
5	0.550 ± 0.004 <sup>F</sup>	31.73 ± 0.40 <sup>C</sup>	86.54 ± 0.37 <sup>D</sup>	59.40 ± 0.73 <sup>C</sup>
6	0.598 ± 0.003 <sup>G</sup>	34.11 ± 0.03 <sup>D</sup>	86.53 ± 0.42 <sup>C</sup>	56.10 ± 0.64 <sup>D</sup>

<sup>y</sup> Averages of triplicate analysis.

<sup>z</sup> Values in the column with different uppercase (A-G) were significantly different ( $p < 0.05$ ).

by the powder particles.

### Water activity

Throughout the accelerated storage (Table 1),  $A_w$  of peel powder decreased with time, which was related to the moisture loss in the powder. The  $A_w$  of powder stored at room temperature significantly ( $p < 0.05$ ) doubled by the end of the 6 months storage (Table 2). These  $A_w$  results were all less than 0.6, which showed that the peel powder was shelf-stable. Caking and clumping were not observed under both storage conditions.

### Hygroscopicity

The hygroscopicity of pitaya peel powder kept at 45°C and room temperature ranged from 27.6 - 29.2% and 27.6 - 34.1%, respectively. These values were lower than those of *Amaranthus* powder produced with DE 10 (40.9%) by Cai and Corke (2000). Although pure betacyanin pigment powder absorbed moisture easily due to the presence of hydrophilic groups in the molecule (Huo and Guo, 1994), however, the addition of maltodextrin was reported to retard caking and decrease the hygroscopicity of powders (Osman and Endut, 2009; Goula and Adamopoulos, 2008; Jaya and Das, 2004; Cai and Corke, 2000). Insignificant increase of the powder hygroscopicity was observed until 10<sup>th</sup> week of the storage; but powder hygroscopicity was increased significantly ( $p < 0.05$ ) thereafter (Table 1). A bigger increase of hygroscopicity in the powder was

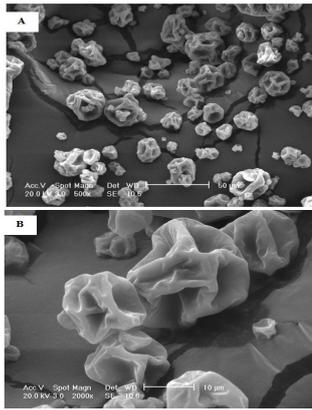


Figure 1. Micrographs of fresh spray-dried pitaya peel particles (165°C inlet air temperature, 80°C outlet air temperature and 15% maltodextrin DE10) in different magnifications: (A) 500x and (B) 2000x.

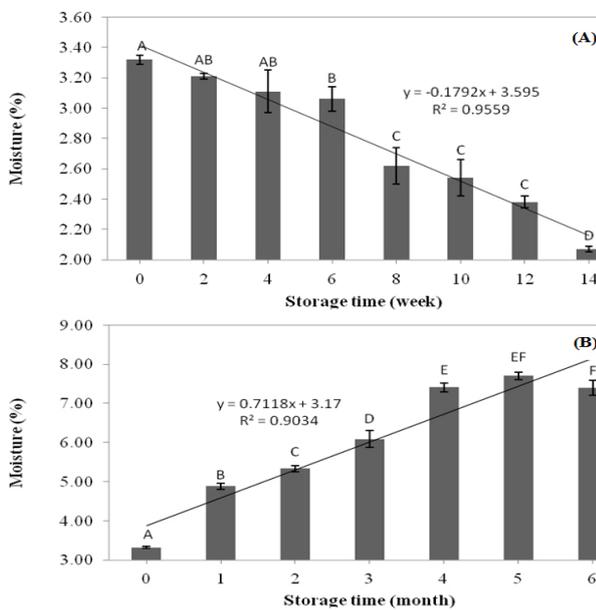


Figure 2. The changes of moisture content in spray-dried pitaya peel powder throughout (A) accelerated storage at 45°C for 14 weeks, and (B) room temperature storage for 6 months [Values in the bar with different uppercase were significantly different ( $p < 0.05$ )].

observed at room temperature and significant change was obtained after 3 months of storage (Table 2). The difference in the increase of the hygroscopicity could be due to the different in the relative humidity of the storage environment which was also reported by Cai and Corke (2000). The hygroscopicity of pitaya peel powder kept at 45°C and room temperature ranged from 27.6% to 29.2% and 27.6% to 34.1%, respectively, which were less hygroscopic than the spray dried *Amaranthus* powder produced with DE 10 (40.9%) by Cai and Corke (2000).

**Solubility**

Negligible increase in the solubility of the powder was obtained for those stored at 45°C (Table 1). The powder had a solubility of 90 to 92%. The increased

Table 3. Storage stability of spray dried pitaya peel powder in accelerated (45°C) and room temperature storage

Storage condition	R <sup>2</sup>	Rate constant $k \times 10^{-3}$	Half-life $t_{1/2}$	Pigment retention (%)
Accelerated 45°C (after 14 weeks)	0.9546	9.1 (week <sup>-1</sup> )	76.15 (weeks)	86.96 ± 0.46
Room temperature (after 6 months)	0.9797	18.1 (month <sup>-1</sup> )	38.29 (months)	89.04 ± 0.79

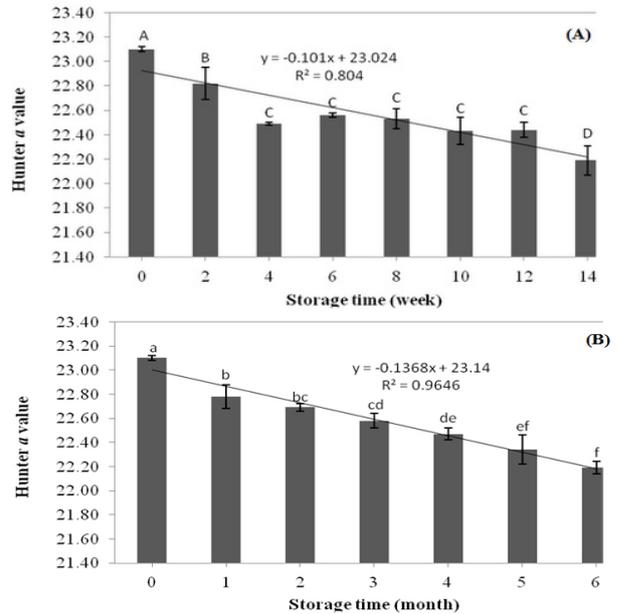


Figure 3. The changes of Hunter a values in spray-dried pitaya peel powder throughout (A) accelerated storage at 45°C for 14 weeks, and (B) room temperature storage for 6 months [Values in the bar with different uppercase were significantly different ( $p < 0.05$ )].

of powder solubility was related to the residual moisture in the powder, being more soluble when the moisture content was low (Goula and Adamopoulos, 2005). An inverse relationship was obtained for moisture content and solubility for powder kept at room temperature (Table 2). All powders kept at room temperature showed continual decreased in solubility and a significant dropped of approximately 3.30% was obtained. As shown in Table 1 and 2, the solubility of pitaya peel powders were similar to that of spray-dried pineapple powder (81.6%) of Abadio *et al.* (2004). The pitaya peel powder was more soluble than the spray-dried tomato powder (17.7 - 26.7%) of Sousa *et al.* (2008).

**Redness color**

Throughout the accelerated storage, Hunter a values (redness) decreased slightly with time, which reflected the characteristic degradation of betacyanin pigment in the powder (Figure 3a). Similar decrement trend was observed for powder kept at room temperature (Figure 3b). According to Herbach *et al.* (2004; 2006), upon prolong heating at elevated temperature, betacyanin was found to decompose

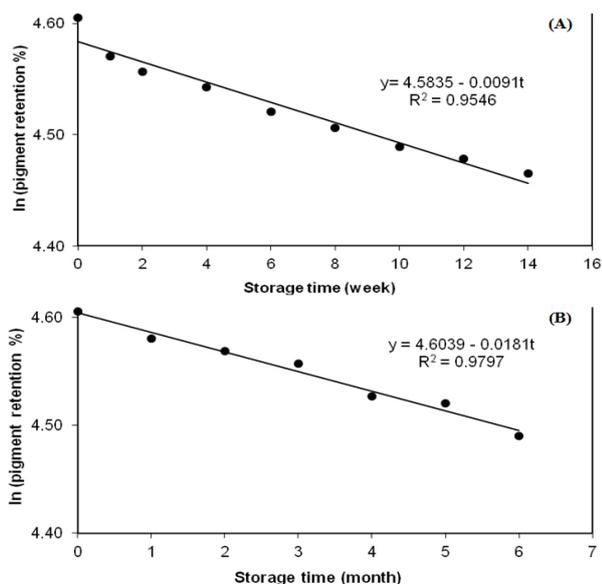


Figure 4. Regression of  $\ln$  (pigment retention %) versus storage time of spray dried pitaya peel powder kept at (A) 45°C for 14 weeks, and (B) room temperature for 6 months.

into yellow degradation products (*cyclo-dopa5-O-b*-glucoside and betalamic acid), leading to a loss in the red color.

#### Kinetic modeling and betacyanin pigment storage stability

The rate constant and the half-life reactions of betacyanin degradation were determined through the kinetic of betacyanin pigment retention of the stored pitaya peel powder as a function of time and temperature. The natural logarithm of the ratio of the betacyanin retention (%) plotted against storage time are as shown in Figure 4A and 4B. The linear line obtained indicated that betacyanin degradation in the powder followed the first-order reaction kinetics. All data were best fitted by a first-order kinetic model [ $\ln C = \ln C_0 - k(t)$ ] with  $R^2 > 0.95$  (Table 3). These findings were consistent with several reports on betacyanin pigment degradation in purple pitaya juice (Herbach *et al.*, 2004), *Celosia* plant (Cai *et al.*, 2001), spray-dried *Amaranthus* powder (Cai and Corke, 2000) and *Amaranthus* pigment (Cai *et al.*, 1998). Higher value of  $k$  indicated greater degradation speed of betacyanin pigment in the peel powder. At the end of 14 weeks storage (Table 3), the rate constant and the half-life value of the packed powder kept at 45°C were calculated to be  $9.1 \times 10^{-3} \text{ week}^{-1}$  and 76.15 weeks, respectively. Betacyanin contents of peel powders gradually decreased throughout 14 weeks of accelerated storage (Table 1), from 64.66 mg/100 g to 56.22 mg/100 g. Generally, natural pigments are susceptible to degradation at higher storage temperature; however, the pitaya peel powder was found to be stable since betacyanin

retention (86.96%) was high even at the end of the storage at 45°C. The retention of betacyanin content (89.04%) of the powder kept at room temperature showed a slow decrease (Table 2 and 3). At the end of the storage period, the rate constant and the half-life value of the powder were  $18.1 \times 10^{-3} \text{ month}^{-1}$  and 38.29 months, respectively. Cai and Corke (2000) reported that the half-life was 43.6 weeks and the retention of betacyanin retention was 88.7% in spray dried *Amaranthus* powder when kept at 25°C for 16 weeks. Therefore, betacyanin pigment in pitaya peel powder should be considered as stable as that in the *Amaranthus* powder, although the degradation of the pigment was carried out under slightly different conditions.

#### Conclusion

Storage conditions significantly ( $p < 0.05$ ) influenced peel powder's moisture content, water activity, solubility, hygroscopicity and color. The half-life of the spray dried pitaya peel powder was predicted to be approximately 76.2 weeks at 45°C and for 38.3 months at room temperature, respectively. The degradation of betacyanin pigment in spray dried pitaya peel powder was according to the first order reaction model and the destruction was greater at 45°C. Betacyanin pigment retention was relatively high (>86%) for both studied storage temperatures.

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