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## Impact of temperature and relative humidity on physicochemical properties of the spray dried red flesh pitaya powder during storage

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### ABSTRACT

This study was carried out to evaluate the changes in physicochemical properties of pitaya powder produced by spray-drying under the different temperature storage at 5-7°C, room temperature, and 50°C for 40 days. The results showed that delta E of color increased but the content of betacyanin and total phenolic decreased following temperature increase. The degradation of betacyanin and total phenolic content in all samples during storage fitted the first-order reaction. Moreover, the behavior of moisture content in pitaya powder under different relative humidity was investigated. The isotherm curve of pitaya powder was built ( $y = 3,2655e0,0106x$ ). The result also showed the GAB equation can be used to calculate the MO of the sample better than the BET equation.

## 1. INTRODUCTION

The flesh and peel of the pitaya contain the beneficial compound including vitamin C, polyphenol, especially betacyanin — the compound giving red or purple-red color for pitaya. (Jaafar et al., 2009; Le et al., 2021). Therefore, the previous studies were researched to encapsulate betacyanin in the extract of peel and flesh by using spray drying (Tze et al., 2012; Bakar et al., 2012; Lee et al., 2013). The authors focus research on the optimization of spray-dried red flesh pitaya powder processing such as food aid wall material, inlet, and outlet drying temperature, pump speed was found in the previous studies. However, the information about the change of physicochemical properties, antioxidant compounds during storage, especially red flesh pitaya powder produced from flesh and peel was still limited.

The bioactive compounds such as betacyanin, polyphenol can be changed by temperature, relative humidity, these factors can promote the degradation

of them (Gengatharan et al., 2105; Ali et al., 2018). Therefore, an understanding of the causes of deterioration and the appropriate storage conditions for food products are important issues needed to be comprehended. Furthermore, many studies report the sorption characteristics for a variety of fruit powders (Kha et al., 2015; Muzaffar & Kumar, 2016), but not for pitaya fruit powder. This study is aimed to investigate the stability of the pitaya powder under a variety of storage conditions (i.e relative humidity and temperature), in terms of color, betacyanin, polyphenol, moisture content, and water activity. Kinetic parameters and moisture sorption isotherms were also examined to predict the shelf-life of this product.

## 2. MATERIALS AND METHODS

### 2.1. Material and chemical

Red- flesh pitaya (*Hylocereus polyrhizus*) were purchased at Thu Duc wholesale market, Ho Chi Minh City. The selected fruit was evenly red peel with no physical injures and fungi.

Chemical: Folin – Ciocalteu 99,5% (Merck, Germany), sodium carbonate 98%, gallic acid, potassium acetate (CH<sub>3</sub>COOK), magnesium nitrate (Mg(NO<sub>3</sub>)<sub>2</sub>), sodium chloride (NaCl), and potassium chloride (KCl), sodium hydroxide (NaOH) were purchased from Xilong, China.

## 2.2. Sample preparation

Juice extract preparation: After washing, the flesh and peel of pitaya (prepared in section 2.1) were separated by hand. Then, the flesh was crushed by a blender (MJ-70M, Panasonic, Japan), and the extract was mixed well with distilled water in the ratio 1:1 (w/w). The mixture was hydrolyzed by using enzyme 0,2% Pectinex Ultra SP-L(w/w) for 1 hour at 45 °C by using a water bath (Schutzurt, Germany). The peel of the pitaya part was blended (Philip, Netherland) with distilled water in the ratio 1:1 (w/w). The mixture was hydrolyzed with 0,2% Pectinex Ultra SP-L (w/w) at 45°C for 90 minutes. Both flesh extract and peel extract were used for spray drying on the sample day.

Powder preparation: The flesh extract and peel extract were mixed with the ratio of 4:1 (w/w). Maltodextrin DE10 at 15% was added to the mixture and stirred continuously until maltodextrin dissolved completely. After that, the mixture was homogenized at 5000 rpm for 5 minutes (T18 digital Ultra Turrax homogenizer, IKA, Germany). The mixture was then dried in a spray dryer at an inlet temperature of 150°C. The inlet flow rate is 500 mL/h, the pump rotation speed was 2.5 rpm, and the compressed air pressure was 2.1 kgf /cm<sup>2</sup>. The spray-dried powder was collected and kept in polyethylene (PE) bags coated aluminum at -18°C until further analysis.

## 2.3. Effect of temperature storage on physicochemical properties of pitaya powder

An aliquot 5 g of pitaya powder (prepared in section 2.2) was kept in a polyethylene bag coated aluminum 7×12 cm (0.16 mm of thickness). The samples were stored at accelerated temperature include 50°C, room temperature (30 ± 1°C), refrigerated temperature (5 ± 1°C) for 0, 5, 10, 15, 20, 30, and 40 days. At each time interval, the samples at each storage condition were analyzed the physicochemical properties such as water activity, moisture content, betacyanin content, total phenolic content, and color values measurement. All experiments were carried out in three replicates.

## 2.4. Effect of relative humidity on the physicochemical properties of pitaya powder

Preparing a series of hermetic glass desiccators (700mL) containing available saturated solutions of sodium hydroxide (NaOH), potassium acetate (CH<sub>3</sub>COOK), magnesium nitrate (Mg(NO<sub>3</sub>)<sub>2</sub>), sodium chloride (NaCl), and potassium chloride (KCl), providing a relative humidity were 10%, 23%, 51%, 76%, and 84%, respectively. The environment for this study was prepared for 24 hours before conducting the experiment. After that, an aliquot of 2 g flesh and peel pitaya powder (prepared in section 2.2) was put in a polyethylene bag that was not sealed. All the desiccators were tightly closed and stored at room temperature. Additionally, after being stored at room temperature for 14 days, the samples were analyzed for moisture content, water activity, betacyanin content, and total phenolic content. All experiments were carried out in three replicates.

## 2.5. Analytical method

### 2.5.1. Determination of moisture content

The moisture content of the powder was determined by using an MX-50 moisture analyzer. One gram of powder was put on aluminum dish after pressing “reset”. Then press “Start”, heating unit starts to dry the sample at 130°C. The instrument shuts off and moisture was obtained on the screen when the sample no longer lost weight.

### 2.5.2. Water activity

A sample cup was half-filled with pitaya powder, then the water activity was measured using a water activity meter (Aqualab Series 3, Decagon, WA, USA).

### 2.5.3. Determination of betacyanin content

The betacyanin quantification was determined by using a spectrophotometer (Wong et al., 2015). One gram of pitaya powder was mixed with 25 g of distilled water. The solution was filtered and kept in test tubes wrapped with aluminum foil to prevent light penetration. The filtered solution's absorption value was analyzed by a spectrophotometer (Jascor V730, Japan) at a wavelength of 538 nm. Betacyanin content (BC) was calculated by the following equation:

$$\text{Betacyanin content (mg/100 g dry matter)} = \frac{A \times DF \times V \times MW \times 100}{\epsilon \times L \times W(100\% - \text{Moisture content}\%)}$$

Where: A: the absorption value. DF: the dilution factor. V: sample solution (mL). MW: the molecular weight of betanin (550g/mol).  $\epsilon$ : the molar extinction coefficient of betanin ( $\epsilon = 60000 \text{ L} \times \text{mol}^{-1} \times \text{cm}^{-1}$  in  $\text{H}_2\text{O}$ ). L: the path length of the cuvette (L = 1 cm). W: the weight of the sample (g). 100: the coefficient converts from g to 100g.

#### 2.5.4. Determination of total phenolic content

The amount of total phenolic content was determined using Folin-Ciocalteu's reagent. Absorption spectrum of polyphenol was determined at a wavelength of 765 nm (Lim et al., 2007). Add 1 ml of distilled water to a mixture of 1 ml of the

$$\text{TPC} = \frac{(y - b) \times V \times DF \times 100}{a \times m \times (100\% - \text{moisture content}\%) \times 1000}$$

Where y: OD of the sample. a and b are the coefficient in the standard curve. V: volume of extracted solution. DF: dilution factor. m: mass of sample (g). 100/1000: the coefficient converts from  $\mu\text{g/g}$  to  $\text{mg}/100\text{g}$ .

#### 2.5.5. Color measurement

The color of pitaya powder samples was determined using a Chroma mater CR – 400 (Konica Minolta, Japan). The results were expressed as  $L^*$ ,  $a^*$ , and  $b^*$  values. Total color difference samples were calculated by the formula as follows:

$$\Delta E = \sqrt{(L_o^* - L^*)^2 + (a_o^* - a^*)^2 + (b_o^* - b^*)^2}$$

Where  $L_o^*$ ,  $a_o^*$  and  $b_o^*$  are the values of the samples at zero time, and  $L^*$ ,  $a^*$  and  $b^*$  the measured values of each sample after storage (Kha et al., 2015).

#### 2.5.6. Calculation of kinetic parameters

The kinetic change of powder was reported to follow first-order reaction rate constants (Kha et al., 2015) using the following equations: First-order kinetic reaction:  $\ln C = \ln C_o - kt$ . Where C is the concentration at time t;  $C_o$  is the concentration at time zero; k is the degradation rate constant ( $\text{day}^{-1}$ ) is obtained from the slope of a plot of the natural log of  $C/C_o$  vs. time, and t is storage time (Days). The half-life was calculated at a specific temperature by the equation:  $t_{1/2} = \ln 2/k$

#### 2.5.7. Sorption moisture content

The monolayer moisture content  $M_o$  (db, %) was calculated using the Brunauer–Emmett–Teller

diluted sample and 0.5 mL of Folin- Ciocalteu 10%. The mixture solution was vortexed and prevented light for 6 minutes. Continue, the mixture was added 1,5 mL of  $\text{Na}_2\text{CO}_3$  20% and 1 mL of distilled water and shake well. The solution was left in the dark for 60 minutes and the absorbance was determined spectroscopy at 765 nm (distilled water was used as blank). Gallic acid solution at different concentrations ( $\mu\text{g/ml}$ ) was used to build a standard curve. Total phenolic contents (TPC) were expressed in terms of gallic acid equivalent  $\text{mgGAE}/100 \text{ g}$  of dry matter (dm)

(BET) and Guggenheim – Anderson de Boer (GAB) equations as follows (Koç et al., 2010).

$$\text{BET: } M_o = \frac{M_C C A_w}{(1 - A_w)[1 + (C - 1)A_w]}$$

$$\text{GAB: } M_o = \frac{M_C C_o K_G A_w}{(1 - K_G A_w)(1 - K_G A_w + C_G K_G A_w)}$$

Where,  $M_C$  is moisture content of powders expressed in g per 100 g sample;  $M_o$  is g of water equivalent to monomolecular layer adsorbed per 100 g sample;  $a_w$  is water activity at moisture content  $M_C$ ; C is BET constant;  $C_o$ ,  $K_G$ , and  $C_G$  are GAB equation parameters.

### 2.6. Statistical analysis

All experiment was reported as mean  $\pm$  standard deviation. Data and results were analyzed by using JMP 10.0 and Excel 2013.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of temperature storage on physicochemical properties of pitaya powder

The degradation rate k ( $\text{day}^{-1}$ ) of samples was kept at  $5^\circ\text{C}$ , room temperature and  $50^\circ\text{C}$  were 0.0041, 0.0044, and 0.0106, respectively (Figure 1 and Table 1). The predicted time for betacyanin content to be reduced by 50% at  $5\text{-}7^\circ\text{C}$ , room temperature ( $29\text{-}31^\circ\text{C}$ ), and  $50^\circ\text{C}$  were 169, 157, and 71 days, which indicates high-temperature storage gave the half-life ( $t_{1/2}$ ) of product reduced. This phenomenon could be explained the betacyanin at high temperatures can be converted into different isomer compounds or be decomposed (Gengatharan

et al., 2015). The high temperature leads to the decline of the compound betacyanin that has also been demonstrated in another previous study about the pitaya such as spray-dried study, puree (Liaotrakoon et al., 2013; Ee et al., 2014).

The total phenolic content behavior changed under different temperatures similar to betacyanin compound. At room temperature, the degradation rate of polyphenol ( $k=0.004 \text{ day}^{-1}$ ) approximately 2 times smaller than samples stored at 50°C ( $k=0.009 \text{ day}^{-1}$ ). On the other hand, the degradation rate of polyphenol at 5°C ( $k=0.0032 \text{ day}^{-1}$ ) is lower than when stored at a higher temperature. For polyphenol, the  $t_{1/2}$  value was 217 days at 5°C, 173

days at room temperature, and 85 days at 50°C. The stability of phenolic compounds appears to be greatly affected at temperatures higher than 50°C. This is shown that temperature is one of the most factors that influence the total phenolic content in powder. That is consistent with the results presented in previous studies on polyphenols degradation (Moldovan et al., 2016). The decrease of the total phenolic content of the sample at high temperatures may be due to the increased oxidation reaction of bioactive components. The rates of these oxidation reactions increase as a result of temperature increases.

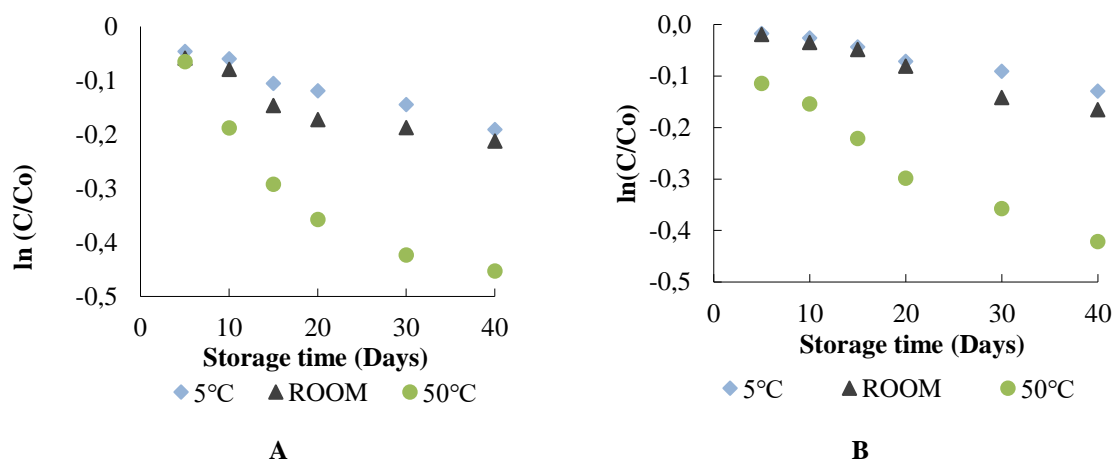


Figure 1. First-order degradation plots for (A) betacyanin content and (B) total phenolic content in pitaya powders under different temperatures such as 5-7°C, room temperature (29-31°C), and 50°C

Table 1. Kinetic parameters of first order for bioactive compounds degradation in pitaya powders under different storage conditions

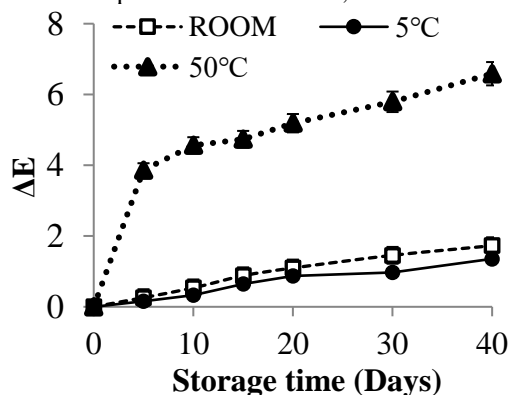
Temperature (°C)	Equation	R <sup>2</sup>	t <sub>1/2</sub> (day)	K (day <sup>-1</sup> )
<b>Betacyanin content (mg/100g dm)</b>				
50	Y= -0.0106x - 0.0855	0.87	65	0.0106
Room	Y= -0.0044x - 0.0554	0.86	158	0.0044
5	Y= -0.0041x - 0.0301	0.97	169	0.0041
<b>Total phenolic content (mgGAE/100g dm)</b>				
50	Y= -0.009x - 0.0817	0.97	85	0.0106
Room	Y= -0.004x + 0.0066	0.90	173	0.0040
5	Y= -0.0032x + 0.0017	0.98	217	0.0032

Note: Room temperature: 29-31°C

Perceivable changes in color ( $\Delta E^*$ ) are an important indicator of the changes in food quality during processing and storage. The difference in perceivable color can be classified as  $\Delta E$  more than 3 (very distinct), less than 3 more than 1.5 (distinct), and less than 1.5 (small difference) (Gengatharan et al., 2105). The  $\Delta E$  values of the sample after 40 days stored at 5°C, room temperature, and 50°C were

1.35, 1.72, and 6.8, respectively (Figure 2). It indicates that the color of the samples was kept at 5-7°C and room temperature (29-31°C) was slightly different from the initial sample; in contrast, the sample stored at 50°C had a sharp difference (Figure 3). The color changes in a sample may be due to the susceptibility of the red-colored betanin compounds to form yellow-colored betaxanthins. Similarly,

other studies have also reported the color of the sample, which one containing betacyanin was significantly affected by high storage temperature and times (Gengatharan et al., 2015). In this study, the room temperature was chosen, because it can



Note: Room temperature: 29-31°C

Figure 2. The different color indexes of pitaya powder under different storage temperatures

### 3.2. Effect of relative humidity on the physicochemical properties of pitaya powder

Generally, the EMC values the environment's relative humidity increased (10-84%) leading to the water activity value (0.22-0.78) and moisture content value (3.67-8.27) of pitaya powder increased (Figure 4). The samples kept at RH<51% reach water activity <0,44 and moisture content <3.39%; which indicates that the pitaya powder could be stable physicochemical and microbial at RH<51%. The sorption isotherm curve of pitaya powder under different RH had behavior similar to the other spray dried fruit powder such as grapes, apricots, apples, and potatoes (Kaymak-Ertekin et al., 2004), tomato (Goula et al., 2008), and tamarind (Muzaffar & Kumar, 2016). The initial moisture content of the sample (4.21%) was lower than the relative humidity of the environment (10-84%), due to the sample absorbing water from the environment during the storage period. Besides that, this phenomenon can be attributed to the hydrophilic nature of maltodextrin in spray-dried pitaya powder.

Figure 5 illustrates the relative humidity affected on the total phenolic content and betacyanin content of the sample after 14 days of storage. Generally, the

stability well the physicochemical properties of pitaya powder. In addition, it is the temperature usually applied on the industrial scale for the dried product's storage, transportation.

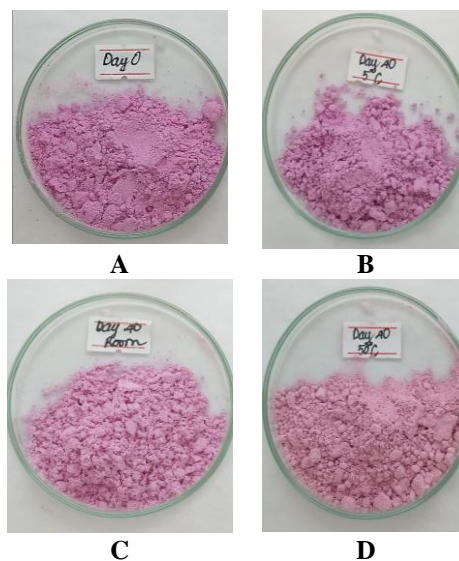
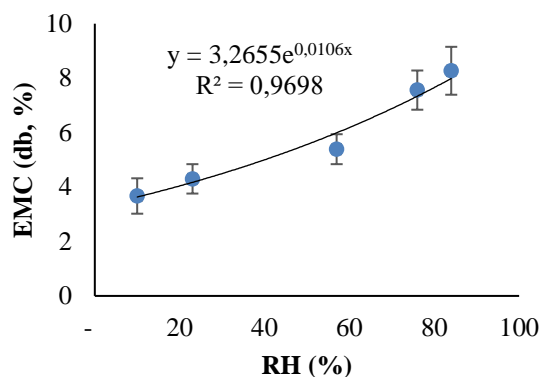


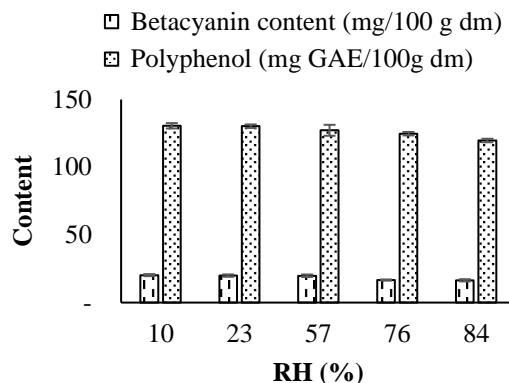
Figure 3. The pitaya powder under (A) before and after 40 days stored at (B) 5-7°C, (C) room temperature (29-31°C), and (D) 50°C

total phenolic content decreased (from 130.56 to 119.69 mg GAE/100g dm) and betacyanin content (from 20.29 to 16.47 mg/100g dm) under relative humidity from 10% to 84%. Whereas the degradation rate of these compounds decreased slightly at RH<51% and sharply decreased at RH>51%. At RH> 51%, the powder caking phenomenon occurred and samples got the color bright pink; in contrast, the samples did not clump and had a pinkish color. The samples were kept at RH<51% to help the physicochemical stability of pitaya powder.

The monolayer moisture content ( $M_0$ ) shows the amount of water at the food surface, which affects the longest time period of product with minimum quality loss (Goula et al. 2008). The sample's monolayer moisture content was stored at room temperature was 2.2 ( $R^2=0.93$ , BET) and 4.3% ( $R^2=0.99$ , GAB). Basic on  $R^2$  value, the GAB equation was more accurate for predicting  $M_0$  than the BET equation. The pitaya powder sample used in the present study had an initial moisture content of  $4.21 \pm 0.35$ , which was close to the  $M_0$  value. The initial moisture content of a product was advised to equal or slightly above  $M_0$  value in order to prolong shelf life (Fellows et al., 2009).



**Figure 4. Sorption isotherm curve of spray-dried red flesh pitaya powder at room temperature**



**Figure 5. Effect of relative humidity (RH) on betacyanin content and total phenolic content in spray-dried flesh and peel pitaya powder after 14 days kept at room temperature (29-31°C)**

#### 4. CONCLUSION

This study found the first-order kinetic reaction for the degradation of the bioactive compounds in red flesh pitaya powder. For betacyanin, the  $t_{1/2}$  value of sample stored at 50°C, room temperature, 5-7°C were 71, 157, and 169 days, respectively. The  $t_{1/2}$  value for total phenolic content is higher than betacyanin; specifically, the values were obtained at 50°C, room temperature, 5-7°C were 85, 173, and 217 days, respectively. Moreover, the isotherm curves of the red flesh pitaya powder stored at room

temperature had the appearance as parts of sigmoidal shapes ( $y = 3,2655e^{0,0106x}$ ). The GAB equation was more accurate for predicting  $M_0$  than the BET equation. The sample keeps good physicochemical properties at temperature 5-7°C or RH<51%.

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