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Optimization of the encapsulation of the mixture made from flesh and peel pitaya extract by spray drying

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ABSTRACT

In this study, the optimal conditions for spray drying of the extract made from flesh and peel of red pitaya were tested using the central composite design (CCD). The response surface methodology was used to evaluate the effect of inlet air drying temperature (145 – 155°C) and concentration of maltodextrin (13-17% w/w) on the physicochemical properties of the powder. The regression models accurately predicted the retention of betacyanin ($R^2=0.94$) and total phenolic ($R^2=0.95$). The optimal conditions for spray drying were determined as inlet air drying temperature at 148°C and 14.62% maltodextrin. Under these conditions, the spray-dried powder achieved retention of betacyanin of 68.04% and retention of total phenolic of 84.17%. The optimized sample achieved a moisture content of 4.41%, the water activity of 0.39, and the water solubility index of 97.41%.

1. INTRODUCTION

The pitaya has a high bioactive compound such as vitamins C, fiber, betacyanin, and polyphenols (Nurul & Asmah, 2014). Besides, the flesh of the fruit has an attractive colors including red, purple-red color. Many authors have studied red flesh pitaya microencapsulation by spray-drying (Lee et al., 2013; Tze et al., 2012). However, the ingredient most commonly used was flesh. The previous study showed that peel takes 25-30% in whole fruit weight. It meant, after processing, a large number of peels were discarded, which put a high pressure on the environment. Moreover, several studies observed pitaya peel had antioxidant capacity and a potential source of natural colorants (Wu et al., 2006; Stintzing et al., 2002).

There are numerous reports finding inlet temperature and maltodextrin concentration are one most of the main factors, that strongly influence

spray-dried powder's quality (Bakar et al., 2013; Phisut, 2012; Goula & Adamopoulos, 2008). However, there is limited scientific information on how these factors can affect the physicochemical properties of red pitaya powder made from a mixture include flesh and peel extracts.

In many studies, response surface methodology (RSM) has been widely applied to optimize dry processing (Bakar et al., 2013; Tan et al., 2015). Therefore, the present study was carried out to understand the influence of inlet temperature and maltodextrin concentration on retention of betacyanin and total phenolic of red pitaya fruit powder by using RSM. The physical properties of optimal powder as moisture content, water activity, and solubility were evaluated.

2. MATERIALS AND METHODS

2.1. Material and chemical

Red-flesh pitaya fruits (*Hylocereus polyrhizus*) were purchased at Thu Duc wholesale market, Ho Chi Minh city. The selected fruit was free of physical injuries, rotten. The selected fruits were separated peel and flesh, next, each part was packed by polyamide bags coated aluminum and stored at -18°C.

Chemical: Gallic acid, Folin – Ciocalteu 99,5% (Merck, Germany), sodium carbonate 98% (Xilong, China).

2.2. Sample preparation

1.1.1. Extraction preparation

The flesh after thawing was ground by a juicer (MJ-70M, Panasonic, Japan), then mixed with water to the ratio of 1:1 (w/w). Pectinex Ultra SP-L of 0.2% (weight/weight of flesh) concentration was added. The puree was hydrolyzed at pH natural (4.0-4.5), temperature 45°C for 60 minutes in a water bath (Schutzurt, Germany). After that, the sample was inactivated at 100°C for 1 min, then filtered through a cheesecloth. The obtained solution was refrigerated at 10°C until use.

The peel was mixed with distilled water to the ratio of 1:1 (w/w) and crushed by a blender. Pectinex Ultra SP-L of 0.4% (weight/weight of peel) concentration was added into the peel solution. The mixture was hydrolyzed at pH natural (4.8-5.0), temperature 45°C for a period of 90 minutes. After that, the samples were inactivated at 100°C for 1 min, then centrifuged at 5000 rpm for 10 mins and filtered through a cheese cloth to get the peel extract. The obtained solution was refrigerated at 5-7 °C until use.

2.2.1. Preparation of spray drying infeed solution

The mixture includes flesh extract and peel extract with a ratio of 4:1 (w/w), its total soluble solid is 5.0-5.5%. The mixture was added to the different amounts of the maltodextrin DE 10 to reach the desired concentration (Table 1). The mixtures were mixed well by using a homogenizer at 5000 rpm for 5 mins (T18 digital Ultra Turrax homogenizer, IKA, Germany). All samples were prepared in a coated beaker aluminum to prevent light. The initial sample temperature was room temperature (29-31°C)

1.1.2. Spray drying condition

The mixtures were conducted using a laboratory-scale spray dryer (KD-400, the machine was invented by the Department of Chemical, at campus Ho Chi Minh city of Nong Lam University). For each inlet solution, 200 mL was sprayed through a nozzle tip. The samples were dried at several inlet temperatures (Table 1), and the feed flow rate was set at 500 mL/h which was controlled by the pump rotation speed at 2.5 rpm and compressor air pressure of 2.1 kgf/cm². Next, the encapsulated powders were collected and kept in a screw-capped amber bottle and stored at -18°C until further analysis.

All parameter of each step preparation were collected from the result of the preliminary test.

2.3. Experiment design

The RSM with central composite design (CCD) was used to find the spray drying condition optimal condition of the inlet air drying temperature X_1 (from 145°C to 155°C) and the concentration of maltodextrin X_2 (from 13% to 17%), in order to achieve the highest retention of betacyanin content and total phenolic. The experiment consisted of 11 runs with three central points. The levels for the independent variables were displayed in Table 1. The sample which was optimal was determining physical properties such as bulk density, water activity, color measurement, water solubility index, moisture content.

Table 1. Variable values of the parameters

Coded levels	X_1	X_2
-1	145	13
0	150	15
+1	155	17

X_1 : Inlet air drying temperature (°C) X_2 : Concentration of maltodextrin

2.4. Analytical method

2.4.1. Determination of moisture content

The moisture content of the powder was determined by using MX-50 moisture analyzer.

2.4.2. Determination of water activity

The water activity was measured using a water activity meter (Aqualab Series 3, Decagon, WA, USA).

2.4.3. Determination of betacyanin content

The betacyanin quantification was determined by using a spectrophotometer (Herbach et al., 2007). Absorption spectrum of betacyanin was determined at a wavelength of 538 nm. Betacyanin content (mg/g dry matter) was calculated by the following equation:

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

Where: A is the absorption value at the absorption maximum ($\lambda_{\max} = 538 \text{ nm}$). DF is the dilution factor. MW is the molecular weight of betanin (550g/mol). V is the pigment solution volume (mL). ϵ is the molar extinction coefficient of betanin ($\epsilon = 60,000 \text{ L mol}^{-1} \text{ cm}^{-1}$ in H_2O). L is the pathlength of the cuvette (L = 1 cm). W is the weight of dried material (g). 100: the coefficient converts from g to 100g

2.4.4. Determination of total phenolic content (TPC)

This method according to Lim et al. (2007). Aliquot 1 mL of each of these dilutions were mixed with 10 mL of distilled water and then with 0.5 ml of Folin-Ciocalteu reagent 10% and allowed to stand for 6 minutes. Then 1.5 mL of 20% sodium carbonate and 1 mL of distilled water were added to the reaction mixture. The absorbance was recorded after 2 hours in dark conditions at 760 nm spectrometrically. Total phenolic contents (TPC) were expressed in term of gallic acid equivalent mgGAE /100 g of dry matter (dm)

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

Where y: OD of 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 mg/100g.

2.4.5. Determination of bioactive compound's retention

Proportion of betacyanin or total phenolic retained = $\frac{Bc_1}{Bc_0}$, Where Bc_0 represents the betacyanin or total phenolic content before storage and Bc_1 represents

the betacyanin or total phenolic content after storage.

2.4.6. Color measurement

The color was determined using a Minolta Chroma Meter calibrated with a white standard tile. The results were expressed as Hunter colour values of L^* , a^* , and b^* , where L^* was used to denote lightness, a^* redness and greenness, and b^* yellowness and blueness.

2.4.7. Water solubility index (WSI)

Spray-dried pitaya powder (0.5) g was mixed with 6 ml of distilled water into a centrifuge tube. After continuously stirring for 30 min in water bath at 30°C, the suspension was centrifuged at 3000 rpm for 10 min. The supernatant was placed in a petri dish and dried at 105 °C for 4 hours to obtain the dry solids weight (Anderson et al., 1969). The WSI (%) was calculated as the percentage of dried supernatant with respect to the amount of the original 0.5 g pitaya powder:

$$\text{WSI (\%)} = \frac{\text{Weight of dry solids in supernatant}}{\text{Weight of dry sample}} \times 100$$

2.5. Statistical analysis

All data of this study were calculated by using Microsoft Excel software version 2016, and analysis of variance (ANOVA) was carried out by using JMP 13. The results were reported as mean \pm standard deviations and all experiments were carried out 2 times.

3. RESULTS AND DISCUSSION

3.1. Fitting the Response Surface Methodology (RSM) model to independent variables

The betacyanin retention of all samples was in the range of 62.56%–71.20%, with an average of 67.82%. On the other hand, the total phenolic retention for 11 runs was in the range of 69,54%-84,51%, with an average of 77,28% (Table 2). There was a clear positive interaction between the retention of betacyanin and total phenolic. This means that the powder obtained low betacyanin's retention with low total phenolic's retention (run 7), and high betacyanin's retention with high total phenolic's retention (run 9).

Table 2. The experimental values for the percentage retention of betacyanin and total phenolic (TPC) for the encapsulated red pitaya powder made from the flesh and peel extract mix obtained from the RSM-CCD design

Experiment number	Pattern	X ₁ Temperature (°C)	X ₂ Concentration % (w/w)	Betacyanin (%)		TPC (%)	
				Exp.	Pred.	Exp.	Pred.
1	—	145	13	68.58	68.75	79.18	79.39
2	a0	145	15	71.05	69.75	80.68	80.96
3	+—	145	17	65.82	66.95	73.46	72.96
4	0a	150	13	68.63	69.27	79.11	79.72
5	00	150	15	70.30	71.04	83.38	82.97
6	00	150	15	70.98	71.04	83.65	82.97
7	00	150	15	71.20	71.04	84.51	82.97
8	0A	150	17	70.32	69.03	74.64	76.66
9	+—	155	13	62.56	61.75	69.54	68.72
10	A0	155	15	63.66	64.31	71.32	73.67
11	++	155	17	62.91	63.07	70.57	69.04

X₁: The inlet air drying temperature; X₂: Maltodextrin concentration. (+) Upper limit value, (-) Lower limit value, (a) High axis value, (A) Low axis value, (0) Value at midpoint

Overall, the correlation between the inlet temperature level and the retention of betacyanin and TPC was negatively correlated at independent variable (X₁), quadratic variable (X₁₂), and reverse at interaction variable (X₁ X₂). Similar finding for the concentration of maltodextrin (Table 3).

As determined using the p-value, the coefficients of the retention of betacyanin and TPC for the inlet temperature were directly related and the maltodextrin concentration inversely related. However, the coefficients of the retention of betacyanin and TPC were all significant for the second - order quadratic effects, with both the inlet temperature and the maltodextrin concentration (Table 3). The final equations in terms of the two independent variables, inlet air drying temperature (X₁) and maltodextrin concentration (X₂) for retentions of betacyanin and TPC to obtain the predicted values in Table 3 were as follows:

$$Y_1 = - 3396.23 + 46.53 X_1 + 2.51 X_2 + 0.08 X_{12} - 0.16 X_1^2 - 0.47 X_2^2 \quad (1)$$

$$Y_2 = - 4778.68 - 64.65 X_1 + 9.74 X_2 + 0.17 X_{12} - 0.23 X_1^2 - 1.20 X_2^2 \quad (2)$$

In which, Y₁ and Y₂ are the retentions of betacyanin and total phenolic, respectively; X₁: The inlet air drying temperature, X₂: Maltodextrin concentration.

Table 3 also showed the coefficient of multiple determination for the retention of betacyanin (R² = 0.94), and TPC (R² = 0.95) were high. The R² value shows the closest relation between the experimental and prediction values, which confirm the accuracy

of the model and the existence of the optimal point. On the other hand, 94% and 95% of the change in the retention of betacyanin and TPC are influenced by independent factors such as drying temperature and maltodextrin concentration. According to Castillo (2007), the R²> 0.75 presents that the model is compatible with the experiment. It means the second-order polynomial models (equations 1 and 2) could be used for calculating the experimental data.

Table 3. The coded second-order regression coefficients for the retention of betacyanin and total phenolic (TPC) for the encapsulated flesh and peel pitaya powder obtained from the RSM CCD design

Independent Variables	Regression Coefficient Values	
	Betacyanin (%)	TPC (%)
Intercept	71.04**	82.97
Linear		
X ₂	-0.12	-1.53
X ₁	-2.72*	-3.65**
Quadratic		
X ₂ X ₂	-1.90*	-4.78**
X ₁ X ₁	-4.02*	-5.66**
Interaction		
X ₁ X ₂	0.78	1.69
R ²	0.94	0.95
p - value of lack of fit	0.096	0.063

X₁: The inlet temperature of the air drying; X₂: Maltodextrin concentration; *: p < 0.05; **: p < 0.01.

3.2. Effects of the air drying temperatures on maltodextrin concentrations on retention of betacyanin and total phenolic

In the present study, Table 2 and the 3D response surface (Figure 1) illustrated that the retention of betacyanin and TPC significantly increased under

the inlet temperature from 145°C to 150°C, and the maltodextrin concentration from 13% to 15%. However, the inlet temperature and the concentration of the encapsulating agents over this range gave a large loss of the bioactive compounds in spray-dried pitaya extract. That one similar trend was reported by Tze et al. (2012).

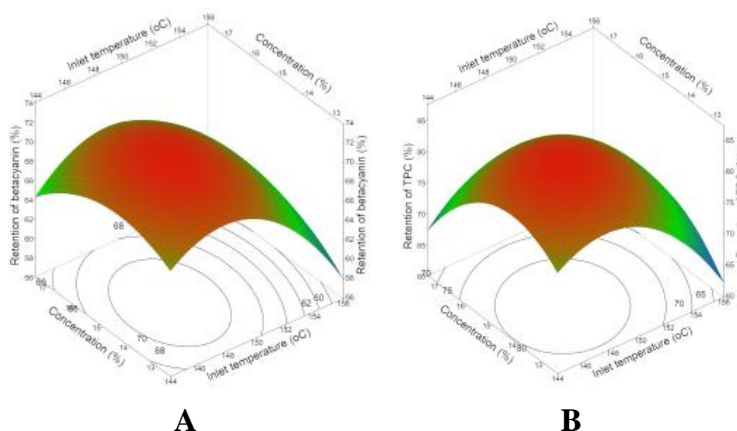


Figure 1. The 3D response surface of the retentions of (A) betacyanin and (B) total phenolic content (TPC) of powder (flesh and peel) in response to inlet temperature (X₁) and maltodextrin concentration (X₂)

These behaviors could be explained, at inlet temperature higher, the speed of heat transfer to the particle was faster, leading to moisture evaporation more, which helped the liquid convert powder greater. In addition, the agglomeration of maltodextrin with a certain concentration may reduce thermal degradation and oxidation and therefore, protecting the bioactive compounds in red pitaya from destruction. However, in another study, the authors found the betacyanin compound more deteriorated under high temperature (Wong & Siow, 2015; Liaotrakoon et al., 2013). Moreover, the high amount of maltodextrin could be increasing the viscous infeed solution, which causes the maltodextrin’s polymerization difficult, thereby reducing betacyanin and polyphenol content in powder.

3.3. The Optimal Formulation and Validation of the Models

The theoretical maximum value for the optimal formulation was predicted to be inlet temperature (X₁) of 148°C and maltodextrin concentration (X₂) of 14.62 % of the pitaya powder processing (Figure 2). These values were close to the predicted value by the quadratic model.

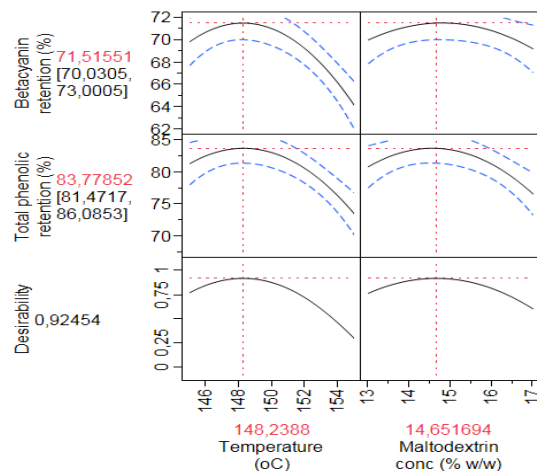


Figure 2. Prediction profiler plots for the retention of betacyanin, total phenolic content (TPC) of the encapsulated flesh and peel extract mix powder affected by inlet air drying temperature (X₁) and maltodextrin concentration (X₂)

The optimal drying conditions to obtain the different fruit powder slightly higher than the conditions were found in this study. For instance, the inlet temperature and maltodextrin concentration for blueberry were 140°C and 30%, respectively (Lingua et al., 2020), for bayberry were 50°C and 31%, respectively (Liu et al., 2017) and acerola and

seriguela juice mix powder were 140°C and 20% (Ribeiro et al., 2018). The difference could be explained by the different components of raw material, equipment, dextrose value of drying aid, etc.

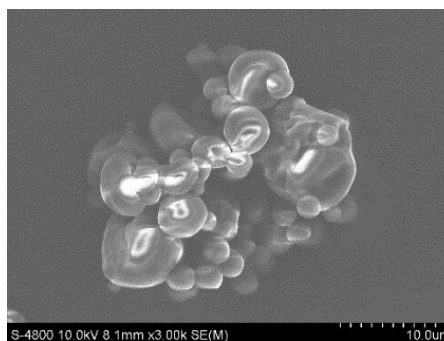
Table 4. Predicted and experimental values of the optimum experimental conditions for the retentions of the betacyanin and total phenolic (TPC) for the flesh and peel pitaya powder

Parameters	Predicted Experimental values	
	values	values
Inlet temperature (X ₁) (°C)	148.23	148
Maltodextrin concentration (%)	14.62	14.62
Retention of betacyanin (%)	71.51	68.04 ± 0.25
Retention of total phenolic (%)	83.79	84.17 ± 2.42

3.4. Physical properties of the Optimised Encapsulated Powder

The SEM of flesh and peel powder result showed the pitaya powder particles were spherical in shape, which had a little difference in particle size. As seen in Figure 3, its micrograph exhibited a continuous wall free of crack or pore on the surface. This meant its characteristic to avoid the bioactive compounds of powder being degraded by external factors (temperature, atmosphere...) during spray drying, storage, transportation. Most of the particles were wrinkled in morphology. The surface particle of powder in this study had consistently with the previous study, for instance, the morphology of the pitaya flesh powder with 20% maltodextrin had a smooth surface appearance (Tze et al., 2012);

The analysis results in Table 5 showed that the optimal pitaya powder in this work had a moisture content of 4.51% and water activity was 0.32. Under these conditions, the sample could be stable under the effect of microorganisms as well as the disadvantageous chemical reaction (Labuza, 1970, cited by Pittia & Antonello, 2016). These results were similar to those of other spray dried fruit powder such as tomato (Goula & Adamopoulos, 2008), bitter melon (Tan et al., 2015), or pitaya fruit powder (Tze et al., 2017).



The sample was sent to the nano laboratory of the Research and Development Center, placed in Hi-tech zone, Thu Duc city, Ho Chi Minh city.

Figure 3. SEM of the encapsulated flesh and peel powder prepared with the optimal formulation, 14.62% (w/w) maltodextrin and inlet temperature 148°C at magnifications of x 3000

The water solubility capacity of encapsulated powder was high (97.41%). Its value was higher than bitter melon powder 93.2% (Tan et al., 2015), mango powder 92-95% (Cano-Chauca et al., 2005). The powder component had maltodextrin, which had high water solubility due to pitaya powder easily dissolved in water (Cano-Chauca et al., 2005). That desirable attribute gave a pitaya powder distributed very well into a product, which improved the utilization ability of powder in the food processing such as beverage and consistency products.



Figure 4. The encapsulated flesh and peel pitaya powder under the optimal formulation: inlet temperature (148°C) and maltodextrin concentration (14.62%)

Figure 4 illustrated the optimal powder got bright pink color, which desirable attribute increased application possibilities of red pitaya powder into the food processing as a food colorant.

Table 5. Predicted and actual values of the optimum experimental conditions for the retentions of the betacyanin and total phenolic (TPC) for the flesh and peel powder

Physical Properties		
Moisture content (%)		4.51 ± 0.31
Water activity		0.32 ± 0.04
Water solubility index (%)		97.83 ± 0.29
Colour	Lightness	73.00 ± 0.41
	a*	27.23 ± 0.38
	b*	359.81 ± 0.02

4. CONCLUSION

The coefficient of the quadratic model for the retention of betacyanin and TPC was found. The

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- optimal formulation was predicted and confirmed to be inlet temperature 148°C and concentration of maltodextrin 14.62 % of spray-dried pitaya made from extract mixture of flesh and peel. Under these conditions, the encapsulated optimal of flesh and peel powder achieved the retention of betacyanin and total phenolic were 68.04% and 84.17%, respectively. The optimum of flesh and peel powder had a low moisture content and water activity value, high bulk density, and water solubility index.
- #### ACKNOWLEDGMENT
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