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EFFECTS OF VARIABLES ON THE PRODUCTION OF RED-FLESHED PITAYA POWDER USING RESPONSE SURFACE METHODOLOGY

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Abstract. Central Composite Design technique from Response Surface Methodology (RSM) was used to investigate the effects of spray drying conditions on red-fleshed pitaya powder moisture content and bulk density. The spray drying independent variables and ranges are inlet air temperature (156-224°C), feed flow rate (16.6-33.4 ml/min) and maltodextrin concentration (31.6-48.4 %). Results showed that the data were adequately fitted to second order polynomial model. However, only linear terms proved to be significant for powder attributes. The best spray drying conditions within the experimental ranges for minimum powder moisture content of 3.88% would be inlet air temperature, feed flow rate and maltodextrin concentration of 224°C, 22.9 ml/min and 40% respectively. The maximum powder bulk density of 0.45 g/ml was obtained at inlet air temperature of 156°C, 16.6 ml/min feed flow rate and 48.4% maltodextrin concentration.

Keywords: Response surface methodology; spray-drying; red-fleshed pitaya

Abstrak. Teknik reka bentuk komposit pusat dalam kaedah gerak balas permukaan (RSM), telah dipilih untuk memeriksa pengaruh parameter-parameter pengeringan sembur terhadap kandungan lembapan dan ketumpatan pukal serbuk buah naga. Pemboleh ubah-pemboleh ubah tak bersandar dengan julat seperti yang disebutkan telah diuji kaji: suhu masukan (156-224°C), kadar aliran suapan (16.6-33.4 ml/min) dan kepekatan maltodextrin (31.6-48.4%). Keputusan menunjukkan bahawa data eksperimen dapat diwakili oleh bentuk polinomial tertib kedua. Bagaimanapun, hanya istilah linear mempunyai pengaruh mutlak terhadap keadaan serbuk. Parameter-parameter terbaik untuk mencapai nilai kandungan lembapan terendah adalah suhu masukan 224°C, kadar aliran suapan 22.9 ml/min dan kepekatan maltodextrin 40%, dengan jangkaan 3.88% kandungan lembapan. Nilai ketumpatan pukal yang maksimum iaitu 0.45 g/ml dicapai pada suhu masukan 156°C, kadar aliran suapan 16.6 ml/min dan kepekatan maltodextrin 48.4%.

Kata kunci: Kaedah gerak balas permukaan; sembur kering; buah naga

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1.0 INTRODUCTION

Red-fleshed pitaya *(Hylocereus polyrhizus)* belongs to order of Caryophyllales and family of Cactaceae. This fruit is becoming popular in Malaysia because of the delicate and juicy flesh. The peel and flesh of this species are red in colour. Red-fleshed pitaya has been recently proposed as a promising source of betalains (Stintzing and Carle, 2007). Since betalains possess high molar extinction coefficients, their colouring power is competitive to synthetic colourants (Stintzing *et al.*, 2003).

Nowadays, red beetroot (*Beta vulgaris*) extract in the concentrated and powder form is the main commercial source of betalains. However, application of red beetroot is restricted due to high nitrate level (Bednar *et al.*, 1991) and its earthy smell caused by geosmin and pyrazine derivatives (Moßhammer *et al.*, 2005). In contrast to red beetroot, red-fleshed pitaya fruits do not contain geosmin and pyrazine derivatives that are responsible for the unpleasant peatiness of the former. Alternatively, betacyanins from red-fleshed pitaya may be a potential source on top of red beetroots.

Spray drying is the transformation of feed from a slurry state into a dried particulate form by spraying the feed into a hot drying medium. It is the most commonly used method to produce fruit juice powder. Fruit juice powder such as pineapple, tomato, mango and watermelon have been produced using spray drying (Abadio *et al.*, 2004; Goula and Adamopoulos, 2005; Mani *et al.*, 2002; Quek *et al.*, 2007). Spray drying process produces powder with high storage stability, easier to handle for some applications and minimize the weight for transportation in comparison with liquid concentrates. In addition, this process may enhance colour, flavor, water binding capacity, and nutritional benefits of various fruit products. Furthermore, it is a highly appropriate process for heat sensitive components such as betacyanins.

The physical properties such as moisture content and bulk density of the resulting powder are influenced by some process variables, such as the characteristics of the liquid feed (viscosity, total solid content, feed temperature) and the drying conditions (feed flow rate, the inlet and outlet air temperature, compressed air pressure) as well as atomization technique and addition of drying adjuvant (Masters, 1985). Therefore, it is important to investigate the effect of process variables on powder properties and to find the best operating parameters in order to obtain powder with desired characteristics.

Response surface methodology (RSM) is one of the most commonly used techniques for determination and optimization of process variables. The process parameters have been merely investigated by conducting one-factor-at-a-time experiments. The result of one-factor-at-a-time experiments does not reflect actual changes in the environment as they ignore interactions between factors that are present simultaneously. An alternative is to use empirical modeling approach such as RSM. RSM is a collection of mathematical and statistical techniques for empirical model building that is an efficient tool for effect study and does not demand a lot of experimental data (Cornell, 1990). RSM were successfully used to link one or more responses to a set of variables when firm interaction is known. This method is an effective and successful technique used to obtain best value and most influencing variable to a few set of variables that affect the value of any response.

Due to strong consumer demand for more natural products, plant derived colourants such as fruit powder from red-fleshed pitaya (*Hylocereus polyrhizus*) have a large potential because of the public concern about possible or proven harmful effects of artificial colourants in food producing industries. However, there is little researches have been reported on investigation of the effect of process parameters on the production of red-fleshed pitaya powder from spray drying.

The objectives of this work were to investigate the effect of processing parameters in spray drying on the physical properties of powder and find out the best processing parameters for the production of high quality red-fleshed pitaya powder by using response surface methodology.

2.0 MATERIALS AND METHODS

2.1 Materials and Chemicals

Fresh red-fleshed pitayas were purchased from local market, Skudai, Johor Bahru, Malaysia. 15 red-fleshed pitayas with an average of 400 g and have no bruises on the skin were bought. Mature fruits were selected due to higher betacyanin content. The maltodextrin (DE 12) was supplied by Sainquip Supplies Sdn. Bhd.

2.2 Preparation of Fruit Juice and Spray Drying Sample

The red-fleshed pitayas were carefully washed with tape water to remove the dirt and foreign materials. The skins of the fruits were then peeled off before cut it into small pieces. The juice was extracted from the flesh by using fruit juice extractor (Philips, model HR 2826). The juice obtained was kept inside the refrigerator at -20°C until used.

The juice was thawed according to the quantity required for each run. The thawed juice was diluted with deionized water by 1:1 ratio on volume basis. The deionized water was heated to 50°C for dissolving the required quantity of maltodextrin until complete dissolution prior to pour it into the fruit juice. Maltodextrin was added to the juice with different weight/volume ratios. Electrical mixer (model IKA RW 20 digital) was used to promote and fasten the dissolution rate of maltodextrin into the heated deionized water and ensure a homogeneous phase of mixture after the mixing of maltodextrin solution into fruit juice. The homogeneous mixture was then filtered through a conventional filter.

2.3 Spray Drying

The sample was spray dried using a laboratory scale spray dryer (Buchi model SD-04, Switzerland). This spray dryer is operated in open-cycle, with co-current flow of hot air and sprayed materials. Atomization of sample was performed using two-fluid nozzle.

The sample was fed into the main chamber through a peristaltic pump and the feed flow rate was controlled by the pump rotation speed. The spray nozzle size, compressed air pressure and feed temperature were kept constant at 0.5 mm, 3 bar and 30°C respectively. Inlet air temperature varied from 156 to 224°C and feed flow rate varied from 16.6 to 33.4 ml/min, according to experimental design.

All the spray-dried powder was collected and kept in air tight container. The spray dryer was washed with water at the desired parameter setting for 10 minutes before and after the spray drying process.

2.4 Experimental Design

RSM was applied to investigate the effect of processing parameters on the physical properties of red-fleshed pitaya powder. A rotatable central composite design was used to design the tests for the spray drying of fruit juice. The inlet air temperature (X_1), feed flow rate (X_2) and maltodextrin concentration (X_3) were independent variables that affect the moisture content (Y_1) and bulk density (Y_2) of the spray-dried powder. The independent variables were transformed to range between -1 and +1 for the appraisals of factors. The variables were coded according to the following equation:

$$Z_i = (X_i - X_0) / \Delta X_i, \quad i = 1, 2, 3$$

where Z is the coded value of the independent variable; X_i is its real value; X_0 is its real value at the center point; and ΔX_i is the step change in the variable X_i. The specific codes are:

Z_1 = (inlet air temperature (°C) - 190) / 20

Z_2 = (feed flow rate (ml/min) - 25) / 5

Z_3 = (maltodextrin concentration (%) - 40) / 5

The coded and uncoded levels of the independent variables used in the RSM design are listed in Table 1.

Table 1 Uncoded and coded levels of independent variables used in RSM design

Coded variables levels (Z_i)	**Uncoded variables levels**		
	Inlet air temperature (°C), X_1	**Feed flow rate (ml/min), X_2**	**Maltodextrin concentration (%), X_3**
+1.682	224	33.4	48.4
+1	210	30	45
0	190	25	40
-1	170	20	35
-1.682	156	16.6	31.6
ΔX_i	20	5	5

The levels of the independent variables were based on preliminary experimental results. Five levels of each variable were chosen for the trials, including the central point and two axial points giving a total of 16 combinations as shown in Table 2.

Table 2 Experimental design for the spray drying tests

Trial Number (Run)	Independent variables		
	Inlet air temperature (°C)	**Feed flow rate (ml/min)**	**Maltodextrin concentration (%)**
1	170 (-1)	20 (-1)	35 (-1)
2	170 (-1)	20 (-1)	45 (+1)
3	170 (-1)	30 (+1)	35 (-1)
4	170 (-1)	30 (+1)	45 (+1)
5	210 (+1)	20 (-1)	35 (-1)
6	210 (+1)	20 (-1)	45 (+1)
7	210 (+1)	30 (+1)	35 (-1)
8	210 (+1)	30 (+1)	45 (+1)
9	156 (-1.682)	25 (0)	40 (0)
10	224 (+1.682)	25 (0)	40 (0)
11	190 (0)	16.6 (-1.682)	40 (0)
12	190 (0)	33.4 (+1.682)	40 (0)
13	190 (0)	25 (0)	31.6 (-1.682)
14	190 (0)	25 (0)	48.4 (+1.682)
15	190 (0)	25 (0)	40 (0)
16	190 (0)	25 (0)	40 (0)

2.5 Analysis of Spray Dried Powder

The spray-dried red-fleshed pitaya powder was analyzed for moisture content and bulk density.

2.5.1 Moisture Content

The moisture content of the spray-dried powder was determined by drying the powder sample at the temperature of 105°C in the oven until a constant weight was obtained (Kha *et al.*, 2010), and expressing the moisture loss in terms of percent wet basis (wb), 100 × kg water/kg wet material.

2.5.2 Bulk Density

Measurement of bulk density was carried out by gently adding approximately 2 g of spray-dried powder into an empty 10 ml graduated cylinder. The weight of the filled powder was determined by using an electronic weight. The bulk density of the powder was calculated by dividing the mass of the powder by the volume occupied in the cylinder (Kha *et al.*, 2010).

2.6 Model Fitting and Statistical Analysis

Second degree polynomial equation was chosen to link response behavior to change of independent variables level as follow:

$$Y = ß_0 + \sum ß_i X_i + \sum ß_{ii} X_i^2 + \sum\sum ß_{ij} X_i X_j$$

Where Y = predicted response, β_o = intercept coefficient, β_i = linear coefficient, β_{ii} = quadratic coefficient, β_{ij} = interactive coefficients and X_i and X_j are the levels of independent variables. Three independent variables involved in this study and hence k takes the value of 3. Thus, the above polynomial equation becomes:

$$Y = ß_0 + ß_1 X_1 + ß_2 X_2 + ß_3 X_3 + ß_{12} X_1 X_2 + ß_{13} X_1 X_3 + ß_{23} X_2 X_3 + ß_{11} X_1^2 + ß_{22} X_2^2 + ß_{33} X_3^2$$

Three-dimensional response surfaces and contour plots were used for facilitating a straightforward examination of the influence of experimental variables on the responses. The individual response surface and the contour plots were created by holding one of the three variables constant at their center points.

The regression coefficients ($ß_0$, $ß_1$, $ß_3$, $ß_{12}$ and so on) of the model for the two responses were estimated with multiple regression analysis. The fit quality of the models was checked from the coefficients of determination (R^2). The adequacy of each model was checked with the analysis of variance (ANOVA) using Fisher *F*-test.

3.0 RESULTS AND DISCUSSION

3.1 Effects of Spray Drying Conditions on Powder Moisture Content and Bulk Density

The values of powder moisture content and bulk density are presented in Table 3. The results of this experimental runs showed that production of red-fleshed pitaya powder is highly depend on the inlet air temperature, feed flow rate and maltodextrin concentration. The regression coefficients and *p* values of the coded second-order polynomial equation for powder moisture content and bulk density are presented in Table 4. For the powder moisture content, examination of *p* values of these coefficients indicated that linear terms of inlet air temperature and feed flow rate are significant at 95% confidence level ($p < 0.05$). On the other hand, linear terms of maltodextrin concentration and inlet air temperature are significant for the powder bulk density.

Table 3 Values of powder moisture content and bulk density

Run	Moisture content (%)	Bulk density (g/ml)
1	5.73	0.357
2	4.94	0.391
3	7.03	0.347
4	6.15	0.404
5	3.89	0.332
6	3.32	0.355
7	4.98	0.330
8	4.46	0.348
9	7.12	0.396
10	4.32	0.361
11	5.77	0.375
12	6.67	0.384
13	5.76	0.325
14	4.46	0.413
15	5.26	0.371
16	4.82	0.362

Table 4: Regression coefficients and *p* values of second-order polynomial model for response variables

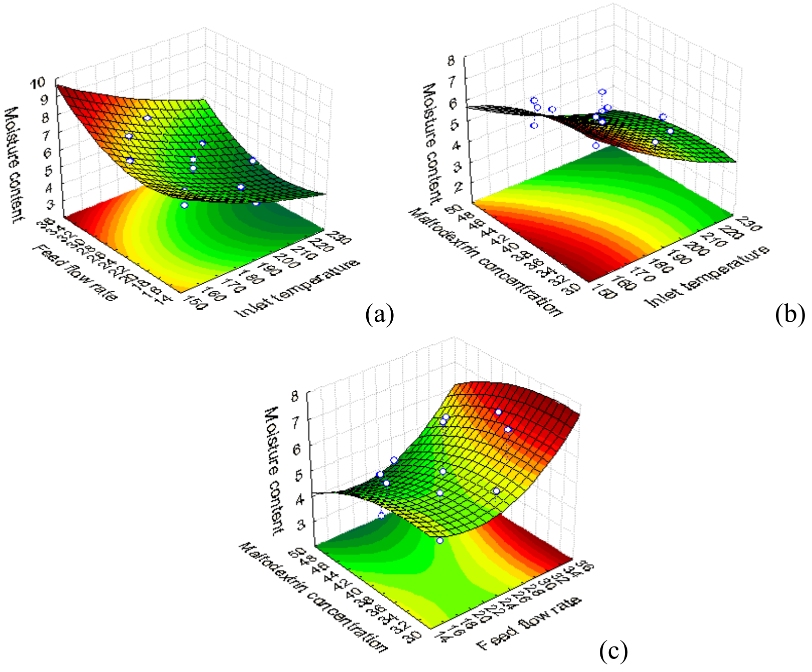
Variables	**Moisture content (Y_1)**		**Bulk density (Y_2)**	
	Coefficient	***p*-value**	**Coefficient**	***p*-value**
Intercept	5.1037	0.0000	0.3683	0.0000
X_1	-0.8720	0.0012	-0.0141	0.0205
X_1^2	0.0867	0.6518	-0.0000	0.9997
X_2	0.4579	0.0227	0.0007	0.8873
X_2^2	0.2635	0.1993	0.0004	0.9511
X_3	-0.3622	0.0528	0.0205	0.0040
X_3^2	-0.1290	0.5067	-0.0034	0.5630
X_1X_2	-0.0350	0.8646	-0.0015	0.8081
X_1X_3	0.0725	0.7250	-0.0062	0.3310
X_2X_3	-0.0050	0.9805	0.0022	0.7165

The coefficients of independent variables determined for the second-order polynomial model for the powder moisture content and bulk density are given as below:

$$Y_1 = 5.10367 - 0.87202X_1 + 0.45791X_2 - 0.36219X_3 + 0.08671X_1^2 + 0.26348X_2^2 - 0.12896X_3^2 - 0.03500X_1X_2 + 0.07250X_1X_3 - 0.00500X_2X_3 \quad (1)$$

$$Y_2 = 0.36826 - 0.01412X_1 + 0.00067X_2 + 0.02050X_3 - 0.00000X_1^2 + 0.00035X_2^2 - 0.00336X_3^2\ 0.00150X_1X_2 - 0.00625X_1X_3 + 0.00225X_2X_3 \quad (2)$$

The interpretation of equation (1) and (2) is based on the magnitude and sign of the regression coefficient. Coefficient of independent variables with higher value shows greater influence on the response. The sign indicates the direction of the proportionality coefficient in relation to the response. A positive sign implies direct proportional relation between the independent variable and the response. Meanwhile, a negative sign implies the relation between independent variable and the response is inversely proportional.



(a)

(b)

(c)

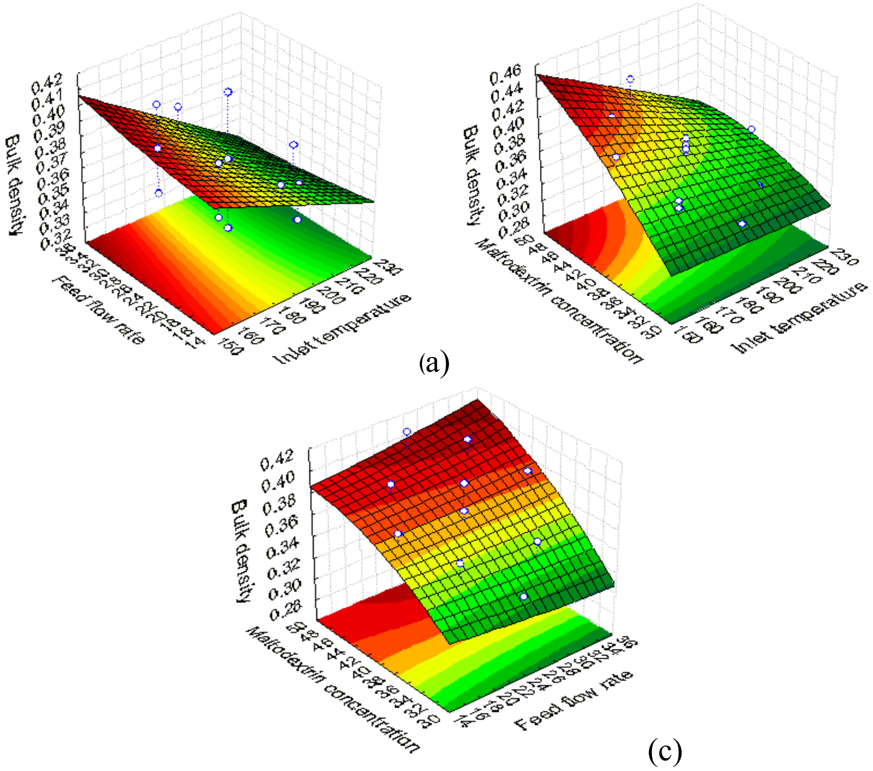
Figure 1 Response surface for powder moisture content, for (a) 40% maltodextrin concentration, (b) feed flow rate of 25 ml/min and (c) inlet air temperature of 190°C

Figure 1 showed the influence of inlet air temperature, feed flow rate and maltodextrin concentration on the powder moisture content. This response was influenced by all the independent variables. Powder moisture content decreases with the increases of inlet air temperature. At higher inlet air temperature, there is a greater temperature gradient between the atomized feed and the drying air, resulting in a greater driving force for water evaporation and thus producing powder with lower moisture content (Tonon *et al.*, 2008). Goula and Adamopoulos (2005), Kha *et al.* (2010), Leon-Martinez *et al.* (2010) and Quek *et al.* (2007) also observed a reduction of powder moisture content with increasing inlet air temperature, studying the spray drying of tomato pulp, Gac fruit juice, nopal mucilage and watermelon juice respectively.

The increase in feed flow rate increases the powder moisture content. Red-fleshed pitaya powder moisture content increases with the increase of feed flow rate. According to Masters (1985), larger droplets with smaller overall surface area are formed at higher feed flow rate. This may lead to lower drying rate. In addition, higher feed flow rate implies a reduction in the contact time between droplets and drying air, producing a less efficient heat transfer. This results in less water evaporation, and therefore higher moisture content (Leon-Martinez *et al.*, 2010).

The increase in maltodextrin concentration results in a decrease in the powder moisture content. Similarly, Kha *et al.* (2010) found that an increased concentration of maltodextrin 12 DE from 10 to 30% reduced the moisture content of the resultant Gac fruit aril powder. A similar result was also reported by Abadio *et al.* (2004) and Grabowski *et al.* (2006), studying the spray drying of pineapple juice and sweet potato puree. These results could be explained by the fact that addition of maltodextrin to the feed prior to spray drying increased the total solid content and reduced the amount of water for evaporation. Hence, the moisture content of the powder has been reduced (Quek *et al.*, 2007).

The best spray drying conditions are achieved if the powder moisture content reached minimum value. According to graphical optimization technique in figure 1, the best spray drying conditions within experimental ranges for minimum percentage powder moisture content of 3.88% were inlet air temperature of 224°C, 22.9 ml/min feed flow rate and 40% maltodextrin concentration.



(a)

(b)

(c)

Figure 2 Response surface for powder bulk density, for (a) 40% maltodextrin concentration, (b) feed flow rate of 25 ml/min and (c) inlet air temperature of 190°C

Figure 2 showed the influence of inlet air temperature, feed flow rate and maltodextrin concentration on the powder bulk density. This response was influenced by maltodextrin concentration and inlet air temperature.

Powder bulk density decreases with the increase of inlet air temperature. Similar trend for the powders bulk density was observed by Chegini and Ghobadian (2007), Goula and Adamopoulos (2005) and Leon-Martinez *et al.* (2010), studying on spray drying of orange juice, tomato pulp and nopal mucilage respectively. Increased inlet air temperature results in a rapid formation of dried layer at the droplet surface due to the skinning over or casehardening of the droplet at higher temperature. Case hardening is a phenomenon associated with many fruit and vegetables powders. This phenomenon occurs when evaporation rate from the surface of the particle is higher than the rate of moisture supply from the interior to the surface of the drying material. Therefore, the surface layer becomes substantially dried (Al-Asheh *et al.*, 2003). This leads to the formation of vapor-impermeable films on the droplet surface that encapsulates the moisture in the interior, followed by the formation of vapor bubbles and consequently droplet expansion. Therefore, the products are dried to a more porous and fragmented structure and there is a greater tendency for the particles to be hollow (Leon-Martinez *et al.*, 2010).

The increase in bulk density with decreasing inlet air temperature can also be attributed to the fact that a product of higher moisture content (dried at low inlet air temperature and high flow rate) would tend to have a higher bulk weight caused by the presence of water, which is considerably denser than the dry solid (Chegini and Ghobadian, 2007).

Powder bulk density showed an increase with an increase in maltodextrin concentration. The increase in maltodextrin concentration causes an increase in viscosity. According to Masters (1985), the droplets size of the atomized materials increases as feed concentration and viscosity increase, resulting in larger dried particles. Particle size has a tremendous impact on bulk density. Typically, as particle size increases, the bulk density will decrease. However, this is not apparent in spray-dried red-fleshed pitaya powder. The explanation for this behavior is if the dehydration conditions are such that the surface of the particle is not fully solidified or remains sticky and the particles collide with each other, then the particles may agglomerate (Grabowski *et al.*, 2006). The decrease in maltodextrin concentration decreases the glass transition temperature of the dried particles due to the lower the glass transition temperature, the stickiness behavior of the particles is stronger where, more particles tend to stick together, leaving more interspaces between them and consequently resulting in a larger bulk volume (Bhandari and Howes, 1999). As a result, the decrease in maltodextrin concentration leads to particles stickiness and decrease the powder bulk density.

The best spray drying conditions are achieved if the powder bulk density reached maximum value. According to graphical optimization technique, maximum powder bulk density of 0.45 g/ml was obtained at 156°C, 16.6 ml/min and 48.4% which was the best inlet air temperature, feed flow rate and maltodextrin concentration.

Table 5 ANOVA for powder moisture content and bulk density

Source	**Degree of freedom**	**Sum of squares**	**Mean squares**	F**-value**	R^2
Moisture content					
Regression	9	16.4102	1.8234	5.8972	0.8984
Residual	6	1.8551	0.30918	-	-
Total	15	18.2653	-	-	-
Bulk density					
Regression	9	0.00899	0.000999	3.5802	0.8428
Residual	6	0.001677	0.000279	-	-
Total	15	0.010667	-	-	-

The ANOVA for the powder moisture content and bulk density are shown in Table 5. The calculated values of F (F-calculated) were compared to the table value of $F_{(p\text{-}1,\, N\text{-}p,\, \alpha)}$ (F-tabulated) of one-tailed test. The null hypothesis which assumes that the observed and predicted values being compared are the same is accepted at α level of significance if the F-calculated is smaller than the F-tabulated. It means that the model is a good predictor of the experimental data.

The ANOVA showed that the F-calculated for powder moisture content and bulk density are 5.8972 and 3.5802 respectively. These F values are smaller than the tabulated- $F_{(9,\ 6,\ 0.01)}$ which is 7.98. The result validates that at 99% confidence level, the null hypothesis is true and the experiment response behavior could be represented by the second-order polynomial model.

The coefficient of determination (R^2) for powders moisture content and powders bulk density were 0.8984 and 0.8428 respectively. The value of R^2 is a measure of total variation of observed values about the mean explained by the fitted model. These values of R^2 are greater than 80% signifies a good agreement between experimental data and predicted values. Therefore, the fitted model obtained can be used to describe the effects of the factors within the experimental ranges.

4.0 CONCLUSION

Moisture content and bulk density are the most common physical properties of a powder product. The lower the moisture content and the higher the bulk density, the better the product is obtained. In this study, the effect of spray drying conditions, i.e. inlet air temperature, feed flow rate and maltodextrin concentration on red-fleshed pitaya powder moisture content and bulk density was studied. It was observed that:

(i) Powder moisture content decreases with an increase in inlet air temperature and maltodextrin concentration, and with a decrease in feed flow rate.
(ii) Powder bulk density increases with a decrease in inlet air temperature, and with an increase in maltodextrin concentration.

The result of statistical analysis showed that the second-order polynomial models were sufficient to describe and predict the response variable of the powder moisture content and bulk density to change in the process parameters for spray drying of red-fleshed pitaya juice within the experimental ranges. Linear terms of inlet air temperature and feed flow rate significantly affected powder moisture content. On the other hand, powder bulk density was affected by linear terms of maltodextrin concentration and inlet air temperature significantly. According to graphical optimization method, the best spray drying conditions within the experimental ranges for minimum powder moisture content would be: inlet air temperature of 223.6°C, feed flow rate of 22.9 ml/min and 40% maltodextrin concentration. The maximum powder bulk density of 0.45 g/ml was obtained at inlet air temperature of 156°C, 16.6 ml/min feed flow rate and 48.4% maltodextrin concentration.

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