

Optimization of Spray Drying Parameters for Pink Guava Powder Using RSM

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Abstract The optimization of pink guava was executed using central composite face-centred design to optimize the spray drying parameters of inlet temperature, maltodextrin concentration (MDC) and feed flow (FF). The experimental results were significantly ($p < 0.01$) fitted into second-order polynomial models to describe and predict the response quality in terms of the final moisture, particle size and lycopene with R^2 of 0.9749, 0.9616, and 0.9505, respectively. The final moisture content significantly ($p < 0.01$) decreased with increasing inlet temperature and MDC, whereas the particle size increased. In contrast, the lycopene content significantly ($p < 0.01$) decreased with the higher temperature and increased with increasing MDC. However, according to multiple response optimization, the optimum conditions of 150°C inlet temperature, 17.12% (w/v) MDC and 350 mL/h FF-predicted 3.10% moisture content, 11.23 μm particle size and 58.71 mg/100 g lycopene content. The experimental observation satisfied the predicted model within the acceptable range of the responses.

Keywords: pink guava, maltodextrin, spray drying, optimization, lycopene

Introduction

Pink guava is a popular fruit, commonly known as the “apple of tropics” reflecting the excellent color, medicinal value and industrial application of this fruit (1). Guava fruits are aromatic and slightly acidic with a strong flavor. Pink guavas contain higher antioxidant activities than white guavas (2) and possess vitamin C at approximately-260 mg/100 g of pulp (3) or 300 mg/100 g of fresh guava (4), which is 3 to 6 times higher than that in oranges. Pink guava is one of the most abundant sources of lycopene, which encompasses more than 80% of the total carotenoids in this fruit. Pink guava contains (4-7 mg/100 g of pink flesh) twice the amount of lycopene of green tomatoes and red-fleshed papayas and greater than red water-melon (5). Guavas are reported to have high anti-inflammatory, hepatoprotective, anticancer and antioxidant activity (6).

However, the lifespan of pink guava (approximately 1 week after harvest) is short (7), and these fruits are easily perishable when ripe and readily undergo physical damage due to improper handling and unsafe transportation; resulting in wastage. Although, a few pink guava products have recently been produced and marketed, the popularity and consumer satisfaction of these products have not been well documented.

Recently, fruit powders have received much attention in the food

industry due to numerous benefits and economic potentials over liquid products. Fruit powders have low moisture content with a longer shelf life, reduced volume and weight, which leads to reduced packaging, easier handling and transportation, and reduced expenditures. Fruit powders can also be used in different food formulations and pharmaceutical products as a natural flavor additive or coloring ingredient (8). Hence, the production of pink guava powder shows the potential to reduce waste, fulfill the market demand and enrich the commercial prospect of pink guava.

Spray drying is a recognized technique, which has been effectively used in mango, gac fruit, raspberry and amla (9-12). However, the most common problem in spray drying of fruit juices is powder sticking to the drying chamber due to the high sugar content naturally present in fruits. As a drying agent, maltodextrin has proved most effective for the reduction of stickiness (13). The quality of the final product depends on different processing conditions (10). For example, moisture content represents the stability ensuring property of the powder product, which is directly influenced by the spray inlet temperature and maltodextrin concentration along with the particle size, bulk density and hygroscopicity studied in gac fruit, amla and acai powder (10,12,14). In addition, an increased feed flow rate might increase the final moisture content due to insufficient drying time and low water evaporation (15).

Furthermore, particle size is important in the analysis of handling, processing, and packaging of food powders. In acai fruit powder, the particle size was affected through inlet temperature and maltodextrin concentration (14). The increased size and irregular-shape particles are responsible for poor bulk density and oxidative degradation (16). Similarly, the sensory compounds and pigments are influenced by higher inlet temperature and higher drying aid concentration (10,14). The lycopene loss was approximately 24% in watermelon powder, when the spray drying inlet temperature changed from 145 to 175°C (17), whereas Goula and Adamopoulos (18) showed the lycopene loss was approximately 8.07 to 20.93% in the spray drying of tomato pulp under different conditions.

However, there are very few studies concerning the powder properties of guava fruits. Patil *et al.* (19) generated white guava powder through spray drying and observed few quality properties. In contrast, Kong *et al.* (20) studied a Beaumont variety of pink guava puree by-product and showed that the lycopene content of freeze-dried powder was 17.21 mg/100 g and that of oven-dried powder ranged from 8.18 to 13.62 mg/100 g. Similarly, according to Nora *et al.* (21), the red guava (*P. cattleyanum Sabine*) powder produced through freeze drying and hot air drying showed a total carotenoid content of 2.44 and 0.89 mg/100 g, respectively (excluding lycopene). However, this process has limitations in how the spray conditions affect the powder properties and lycopene contents of pink guava. The exploration of the optimum spray drying conditions for pink guava might ensure the desired quality of powder.

Response surface methodology (RSM) is a collection of statistical and mathematical practices, which has vast application in the design, development and formulation of new products, even in the improvement of existing products in food engineering. Many researches have been performed to optimize biochemical process conditions, drying conditions, extraction process conditions for desired output (22-24). There are 2 most common and popular designs involved in RSM such as, Central Composite Design (CCD) and Box-Behnken Design (BBD). The CCD is considered as most effective for uniform precision with lower runs required, chronological investigation and reasonable information for lack of fit test. This design is very flexible and it involves three classes of design such as rotatable, spherical and face-centered. The application of this design depends on region of interest and region of operability (25,26).

Thus, the aim of the present study was to optimize the spray drying conditions to generate the desired powder quality with maximum lycopene retention using a central composite design (CCD) with response surface methodology (RSM).

Materials and Methods

Raw materials and chemicals Pink guava juice was obtained from Sime Darby Beverages Pvt. Ltd., Perak, Malaysia and maltodextrin DE 10 from Bronson & Jacobs Pvt. Ltd., Sydney, Australia. The lycopene

analytical standard (85% purity) was purchased from Sigma-Aldrich (St. Louis, MO, USA), and n-hexane (95% purity), acetone (99.5% purity) and dichloromethane (99.8% purity) from Friendemann Schmidt (Parkwood, Australia), and ethanol (99.9% purity, Merck KGaA, Darmstadt, Germany) were used HPLC-grade solvents. HPLC-grade mobile phases, such as acetonitrile (99.9% purity), methanol (99.8% purity) and 2-propanol (99.8% purity) from Friendemann Schmidt were collected.

Sample preparation and spray drying The pink guava juice was diluted with distilled water at a ratio of 1:1 based on preliminary experiments (data not shown). The TSS content was maintained at $5.5 \pm 0.1^\circ\text{Bx}$ and subsequently sieved through a 250 μm sieve, followed by the addition of maltodextrin to the juice sample at concentrations of 10% to 20% (w/v) and homogenized at 5,000 rpm for 8 min prior to adequate mixing (27) using homogenizer (Wise Mix HG-15A; Daihan Scientific, Co. Ltd., Wonju, Korea). In every case, a 300 mL sample was subjected to spray drying using a spray dryer (Lab plant SD-05; Lab plant UK Ltd., Filey, UK). The inlet air temperatures of 150 to 170°C and feed flow rate of 350 to 500 mL/h were set under controlled conditions of outlet temperature, air flow rate and compressor air pressure at $90 \pm 2^\circ\text{C}$, $47 \pm 2 \text{ m}^3/\text{h}$ and $2.1 \pm 1 \text{ bar}$, respectively. The spray drying conditions were selected based on preliminary experiments (data not shown) followed by previous study (10,19,17).

Moisture content The moisture content analysis was conducted using the AOAC method (28). One gram of sample was carefully measured and dried in a vacuum oven (Binder Vacuum Oven, VDL 53; Binder GmbH, Tuttlingen, Germany) at 70°C until constant weight was obtained and the analysis was performed in triplicate. The final moisture content was calculated as the ratio of the total weight of moisture loss to the total weight of the powder sample.

Particle size distribution The particle size distribution was measured using a particle size analyzer (Mastersizer 2000; Malvern Instruments Ltd., Malvern, UK) (29). The particle size was expressed as D [4,3], the mean diameter over the volume distribution which is generally used to characterize a particle (14). The analysis was performed in triplicate.

Lycopene extraction and HPLC analysis The lycopene content was extracted using a slightly modified version of the procedure of Sommano *et al.* (30). First, the 0.5 g powder sample was reconstituted in 10 mL of distilled water in a 50 mL conical flask. Then, the extraction solvents of 10 mL of hexane-acetone-ethanol (2:1:1) and 5 mL of water were added and shaken at 200 rpm for 10 min using the WiseCube Shaking Incubator (WIS-20R; Daihan Scientific Co. Ltd.). Then, the solution was transferred into a 50 mL centrifuge tube and centrifuged at $12,000 \times g$ for 10 min using a 5804 R centrifuge with a F-34-6-38 rotor (Eppendorf AG, Hamburg, Germany). Two layers were observed in the tube and the upper layer-hexane

(lycopene) was collected. The extraction solvent was evaporated using a rotary evaporator (Laborata 4001 efficient; Heidolph Instruments GmbH & Co. KG, Schwabach, Germany) under vacuum pressure at 35°C. The residue was re-dissolved in 2 mL dichloromethane and subsequently filtered through a 0.45 µm membrane filter and stored at -20°C for HPLC analysis.

The lycopene content was determined using the HPLC procedure of Heredia *et al.* (31) with some modifications. The HPLC equipment comprised a pump, injector (Waters 2695 mod. Alliance; Waters Ltd., Milford, MA, USA) and a photodiode array detector (Waters 2996; Waters Ltd.). The stationary phase was loaded onto a 5 µm column (X-select HSS T3; Waters Ltd.). An isocratic mobile phase system of acetonitrile: methanol: 2-propanol (44:54:2 by vol.) was used followed by Anguelova and Warthesen (32). The column temperature was set at 25°C, with a flow rate at 1.5 mL/min and wavelength at 472 nm. The injection volume was 20 µL. The lycopene content was calculated using a standard calibration curve prepared at concentrations of 0.108 to 1.733 mg/mL.

Experimental design and statistical analysis A central composite face-centered design was used for optimization where two processes and one formulation condition were investigated such as inlet air temperature, feed flow rate and maltodextrin concentration. The measured responses were moisture content, mean particle size and lycopene content. According to the central composite design (CCD), twenty treatments were performed, and the centre points were repeated six times. The experimental design acquired from CCD is

shown in Table 1. The experimental design and data analysis were performed using the design of expert software (33). The experimental data were adjusted to a quadratic model to express the response variables as a function of the independent variables using the following equation.

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} x_{ij} \quad (1)$$

where Y is the desired value of response, β_0 is the constant; and β_i , β_{ii} and β_{ij} are linear coefficient, quadratic coefficient and cross-product coefficients, respectively. x_i and x_{ij} are the levels of the independent variables. The model adequacies were assessed after evaluating the lack of fit, R^2 , Adjusted R^2 , Predicted R^2 , Adequate Precision, prediction error sum of squares (PRESS) and coefficient of variation (CV) were obtained from the analysis of variance (ANOVA).

Results and Discussion

Model fitting The experimental data of pink guava powder in terms of the moisture content, particle size and total lycopene content are listed in Table 1. These experimental values were used as a raw data in response surface methodology (RSM) program to generate the best predicted model and its statistical analysis. However, the quadratic model was observed most significant ($p < 0.01$) and suitable considering the lowest standard deviation, the highest R^2 -squared values (R^2 , adjusted R^2 and predicted R^2) and the lowest PRESS. The

Table 1. Central composite face-centered design obtained from RSM and experimental values of the responses for the required conditions

Run	Type	Parameters			Responses		
		Tempt. (°C)	MDC (%)	Feed flow (FF) (mL/h)	Moisture content (%)	Particle size (µm)	Lycopene content (mg/100 g)
1	Center	160	15	425	2.95±0.12	11.79±1.10	44.14
2	Axial	160	15	500	3.41±0.17	12.67±0.53	52.84
3	Fact	170	10	350	2.55±0.09	13.07±0.76	23.59
4	Center	160	15	425	2.78±0.23	11.43±0.68	45.78
5	Axial	160	20	425	2.65±0.08	13.55±0.82	51.95
6	Fact	170	20	350	2.22±0.13	14.48±0.67	44.05
7	Axial	170	15	425	2.38±0.11	12.97±0.86	38.47
8	Fact	150	10	500	4.46±0.15	12.6± 1.13	40.36
9	Axial	160	15	350	2.74±0.10	11.35±0.81	50.51
10	Center	160	15	425	2.99±0.13	11.34±0.93	46.77
11	Fact	150	20	500	4.18±0.14	13.23±1.10	58.37
12	Fact	150	20	350	3.18±0.21	12.4±1.10	55.16
13	Center	160	15	425	2.86±0.13	11.24±0.44	46.93
14	Axial	150	15	425	3.35±0.25	11.33±0.36	60.15
15	Axial	160	10	425	3.36±0.14	11.64±1.89	34.67
16	Center	160	15	425	2.90±0.15	11.2±0.73	49.53
17	Fact	170	20	500	3.08±0.09	14.72±0.80	47.19
18	Fact	170	10	500	3.38±0.13	13.79±1.20	28.18
19	Fact	150	10	350	3.41±0.19	10.97±1.56	43.98
20	Center	160	15	425	2.80±0.12	11.88±0.71	46.81

N.B.: Values are mean±standard deviation

responses were associated with the independent variables and the best-fit model, in terms of coded factors, is shown as follows using a polynomial equation, Eq. (1):

$$MC=2.86-0.50A-0.18B+0.44C-0.015AB-0.045AC-2.500E-0.03BC+0.030A^2+0.17B^2+0.24C^2 \quad (2)$$

$$PS=11.57+0.85A+0.63B+0.47C+0.035AB-0.19AC-0.16BC+0.44A^2+0.88B^2+0.30C^2 \quad (3)$$

$$LC=47.85-7.65A+8.59B+1.01C+1.28AB+1.02AC-0.67BC-0.32A^2-6.32B^2+1.85C^2 \quad (4)$$

where, MC=moisture content (%), PS=particle size (μm), LC=lycopene content (mg/100 g), A=inlet temperature, B=maltodextrin concentration and C=feed flow rate.

The accuracy and fitness of the final predicted model was considered based on the results of analysis of variance (ANOVA). According to the findings presented in Table 2, the probability value (p -value) of the response models was less than 0.01, suggesting that the models for the responses are statistically sound. The coefficient of variation (CV) is a measure of deviation from the mean values, which shows the reliability of the experiment. In general, $CV < 10\%$ indicates better reliability (25). From Table 2, the moisture content, particle size and lycopene content showed low CV values (< 4), indicating that the models of the responses are highly reliable. Adequate precision (AP) is a comparative measure between the predicted values and the

mean prediction error. $AP > 4$ indicates the consistency of the predicted model to describe the process (34). The responses showed $AP > 4$, which ensures that the predicted models are consistent with the spray drying process. The lack of fit value was greater than 0.05, indicating that this value was not significant relative to the pure error, which was mostly preferred. The PRESS values were considered to be minimum to obtain a well-accorded model. The model suitability was also investigated based on the results of fitted-line plots of predicted versus experimental (Fig. 1). The diagnostic line plots signified the intimate closeness between the predicted and experimental values for these responses, suggesting adequate consistency between the experimental and predicted data. Additionally, high R^2 value (> 0.96) was obtained, which suggests that the model is highly compatible.

Effect of process parameters The effect of process conditions, such as temperature (Temp.), feed flow (FF) and Maltodextrin concentration (MDC), was observed on the three responses, such as the final moisture content, particle size and total lycopene content of pink guava powder. The three-dimensional (3D) contour plots of the quadratic model, with one parameter maintained constant at midpoint and the other parameters changed within the experimental limits are presented in Fig. 2A-2F.

Parameter's effect on moisture content Moisture content is the

Table 2. ANOVA evaluation of linear, quadratic, and interaction terms and coefficients for the prediction model of spray-dried pink guava powder

Variance source	df	Moisture content		Particle size		Lycopene content	
		Sum of squares	p -value	Sum of squares	p -value	Sum of squares	p -value
Model (Const.)	9	5.53	<0.0001	23.94	<0.0001	1515.39	<0.0001
Linear							
A	1	2.47	<0.0001	7.22	<0.0001	585.84	<0.0001
B	1	0.34	0.0006	3.98	<0.0001	738.57	<0.0001
C	1	1.94	<0.0001	2.25	0.0007	10.10	0.2841
Interactions							
AB	1	1.8E-003	0.7294	9.8E-003	0.7554	13.21	0.2246
AC	1	0.016	0.3110	0.28	0.1170	8.28	0.3295
BC	1	5.0E-005	0.9539	0.20	0.1739	3.62	0.5135
Square							
A ²	1	2.4E-003	0.6899	0.53	0.0406	0.28	0.8543
B ²	1	0.079	0.0401	2.15	0.0008	109.81	0.0039
C ²	1	0.16	0.0076	0.24	0.1408	9.37	0.3012
Residual	10	0.14		0.96		78.84	
Lack of fit	5	0.11	0.1162	0.54	0.3881	63.37	0.0739
Pure error	5	0.034		0.41		15.47	
Total	19	5.67		24.90		1594.23	
R^2		0.9749		0.9616		0.9505	
Adj- R^2		0.9523		0.9271		0.9060	
Pre- R^2		0.8462		0.8273		0.6605	
Adeq. precision		26.632		17.888		17.38	
PRESS		0.87		4.30		541.24	
C.V. %		3.87		2.50		6.18	

N.B., p value < 0.05 is significant at $\alpha = 0.05$; Lack of fit is not significant at p value > 0.05 .

A, Inlet temperature; B, Maltodextrin concentration; C, Feed flow rate

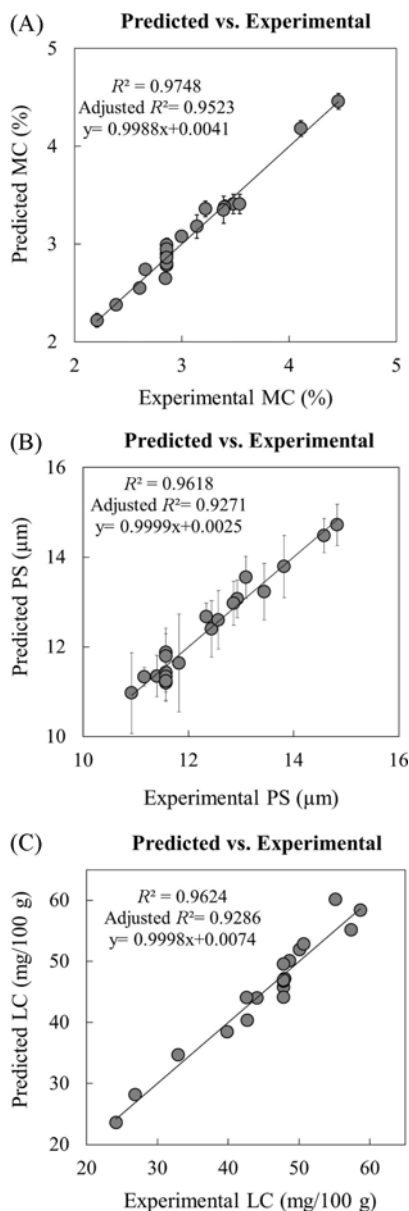


Fig. 1. The fitted line plot signifying the closeness between predicted values and experimental values for (A) moisture content (MC), (B) particle size (PS) and (C) lycopene content (LC)

most important property and is intimately associated with the entire quality and long shelf life of the powder product. The moisture content of pink guava powder was observed in the range of 2.22 to 4.46% (Table 1). The effects of processing conditions on moisture content are shown in Fig. 2A-2C. The results revealed that the higher temperature at low FF (Fig. 2A) and higher MDC at low FF produced a lower moisture powder product (Fig. 2C). A similar observation was reported on kefir powder (23). According to Table 2, the temperature, MDC and FF considerably affected the moisture content of pink guava powder. To obtain a better understanding of the parameters effect, a diagnostic plot of deviation from reference point is presented in Fig. 3A, where two variables were held constant at the

midpoint and one variable was changed within the experimental ranges. The moisture content decreased with the increasing temperature from 150 to 170°C and increasing MDC of 10 to 15% and sharply increased with the higher FF. Supporting results were reported in the spray drying of Amla juice and Gac fruit according to (10,12). This result reflects the higher temperature gradient resulting from rapid water evaporation with a higher rate of heat transfer, ultimately leading to less moisture product and higher MDC, affecting to the total soluble solids of the feed and reduced water evaporation and resulting in reduced moisture product. According to Atalar and Dervisoglu (23), a higher FF is responsible for increased moisture content, reflecting the short drying time between the feed droplets and drying air inside the drying chamber.

Parameter's effect on particle size In the quality control sector, the particle size of the powder products is considered to be a crucial property emphasizing handling, processing, packaging and storage (35). The parameters were individually significant on the particle size of pink guava powder (Table 2). The 3D surface plot of particle size versus inlet temperature, MDC and FF have shown in Fig. 2D-2F. The results showed that the lower temperature with lower MDC (Fig. 2D) and lower FF at lower temperature (Fig. 2E) resulted to the lower particle size of the final product. To obtain a clearer understanding, we observed from Fig. 3B that the particle size increased with increasing drying temperature and increasing FF. Tonon *et al.* (14) and Nijdam and Langrish (36) showed that the particle was smaller in size at a lower temperature. This effect might reflect the significant relationship between water evaporation and particle shrinkage at that drying temperature. The particle size increased with increasing MDC. However, a reduction from 10 to 15% MDC was observed, reflecting an effective relationship between particle shrinkage rate and water evaporation rate at that concentration of MDC. Additionally, the results shown in Fig. 2D-2F revealed clearer curvatures and the optimum condition for minimum particle size.

Parameter's effect on lycopene content Table 1 showed that the lycopene content ranges from 23.59 to 60.15 mg/100 g, indicating that the variation between the maximum and lowest value of the lycopene content was approximately 36.56 mg/100 g during spray drying at different conditions. The temperature and MDC significantly affected the lycopene content (Table 2). Fig. 2G-2I revealed that, higher temperature reduced the lycopene content and higher MDC increased the lycopene retention effectively (17,18). Even though, the interaction parameters (AB, AC and BC) were not significant on lycopene content (Table 2), it can be observed from Fig. 2G and 2I, the interaction between inlet temperature and MDC and interaction between FF and MDC produced distinct curvatures showing the optimum conditions for maximum lycopene content. However, Fig. 3C showed that the lycopene content decreased with increasing temperature. The lowest point of A signifies that the lycopene content decreased below 40 mg/100 g at 170°C where the other

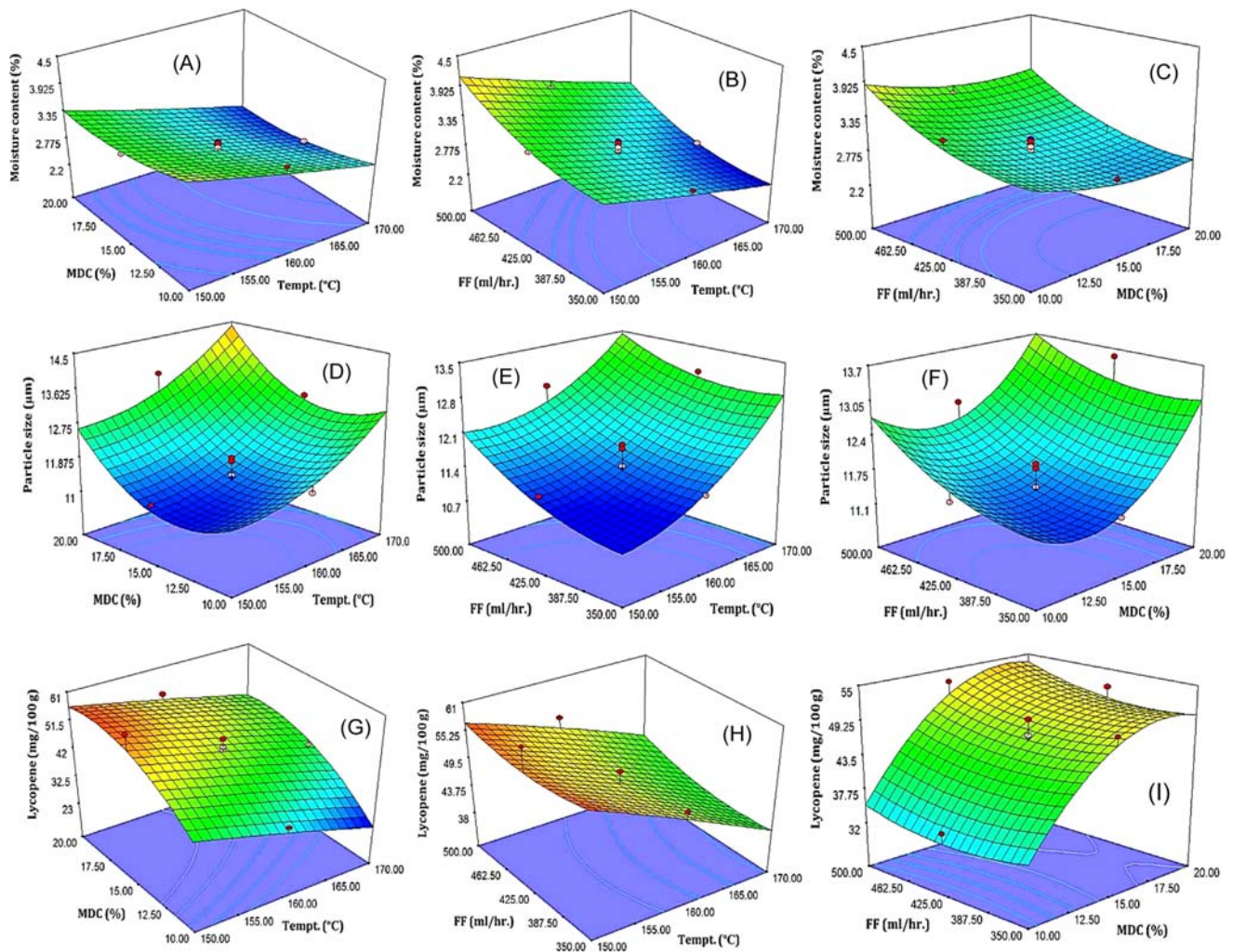


Fig. 2. Response surface and contour plot for moisture content (A,B and C), particle size (D, E and F) and lycopene content (G, H and I)

parameters were constant at 15% MDC and at 425 mL/h FF. At this point, the moisture content was approximately 2.38% (Fig. 2A) suggesting that the increased temperature enhanced the water evaporation rate by increasing the spray droplet temperature, leading to lycopene degradation. A similar relationship was verified for spray-dried tomato pulp (18). In contrast, lycopene content increased with increasing MDC, whereas there was a slight reduction at 20% MDC. Although, higher MDC increased the lycopene retention, MDC decreased the total lycopene content due to the excessive portion of MDC present in the powder (Fig. 2C). Similar observations for the total carotenoid content of spray-dried gac fruit powder and the pigment of water melon powder were reported (10,17).

Optimization of the spray drying process The aim of this investigation was to discover the optimum process parameters for maximum lycopene content with low moisture content and small particle size. For optimization, the desirability function was generated after limiting the preferred goal of parameters and responses, such as minimizing the spray drying temperature, maximizing the MDC

and minimizing the FF during spray drying to obtain the desired quality of powder. Similarly, responses, such as the final moisture content and particle size of the spray-dried powder were minimized and the lycopene content was maximized. According to the desirability function, the predicted optimum condition was obtained as 150°C temperature, 17.12% MDC and 350 mL/h feed flow rate. Table 3 shows the experimental and predicted values of the responses at optimum condition according to the quadratic model for the response variables.

Verification of the model In terms of model verification, three experiments were carried out under the recommended optimum condition with a slight modification in maltodextrin concentration by 17% in exchange of 17.12%. The experimental values and predicted values are tabulated in Table 3. The obtained experimental values were adequate with the predicted values of the response surface model, because the experimental values were very close to the predicted values, which satisfy the predicted model.

In conclusion, the optimization of the spray process conditions for

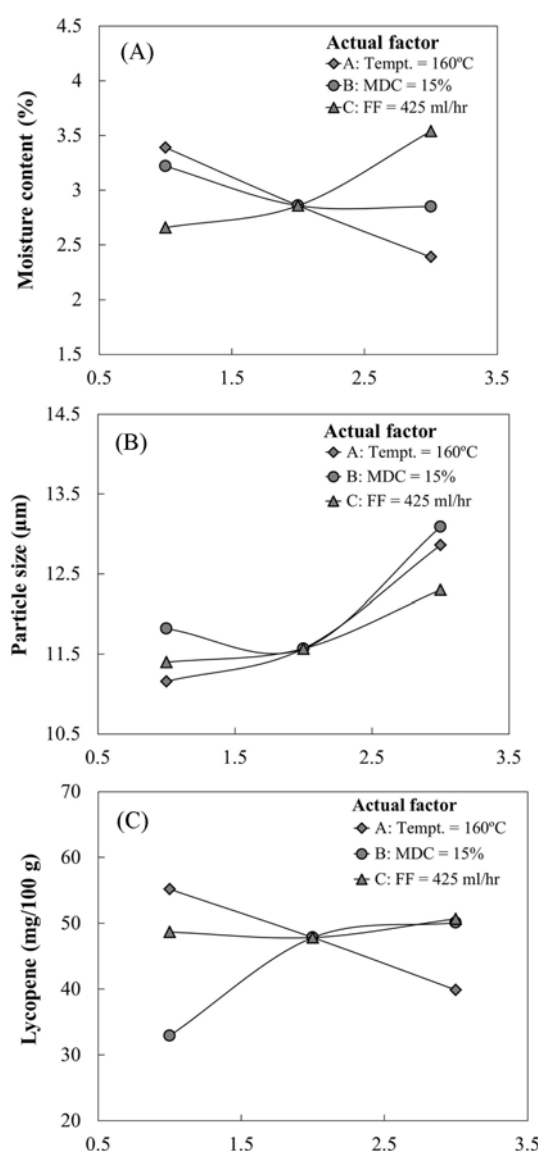


Fig. 3. Deviation from reference point (coded units)-(A) for moisture content (%); (B) for particle size (μm) and (C) for lycopene content (mg/100 g)

pink guava was effectively implemented using a response surface methodology. To describe and predict the variation of response variables, significant empirical equations ($R^2 > 0.96$) were generated. All of the parameters were highly significant ($p < 0.01$) to the responses apart from the feed flow which was insignificant to the lycopene content. With the increased inlet temperature and MDC, the final moisture content reduced; and the particle size increased. However, the lycopene content decreased with increasing temperature and increased with increasing MDC. In addition, higher pump flow increased the final moisture content and particle size. The multiple response optimization revealed the optimum conditions to maximize the lycopene content in the powder with low moisture content and low particle size. The experimental results verified the optimized model in terms of 54.70 ± 0.10 mg/100 g of lycopene, 3.08 ± 0.13 of

Table 3. Predicted and experimental values of the responses at optimum conditions for spray-dried pink guava powder

Responses	Values	
	Experimental	Predicted
Moisture content (%)	3.08 ± 0.13	3.10
Particle size (μm)	11.78 ± 0.65	11.27
Lycopene content (mg/100)	54.70 ± 0.10	58.71

N.B.: Values are mean \pm standard error

moisture content and 11.78 ± 0.65 μm of particle size at the optimum conditions of inlet temperature of 150°C , MDC of 17% (modified) and feed flow of 350 mL/h. Overall, the results of the present study suggest that the obtained model is acceptable for the improved retention of lycopene content with longer shelf-life which can subsequently be applied in large scale production for the fortified food formulations or products enriched in lycopene.

Disclosure The authors declare no conflict of interest.

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