Muzaffar et al., *Cogent Food & Agriculture* (2016), 2: 1127583
http://dx.doi.org/10.1080/23311932.2015.1127583

cogent food & agriculture

FOOD SCIENCE & TECHNOLOGY | RESEARCH ARTICLE

Optimization of spray drying conditions for production of quality pomegranate juice powder

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Received: 18 November 2015
Accepted: 23 November 2015
First Published: 19 January 2016

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Reviewing editor:
Fatih Yildiz, Middle East Technical University, Turkey

Additional information is available at the end of the article

Abstract: The present study was aimed to optimize the spray drying operating parameters for the production of quality pomegranate juice powder using response surface methodology. The spray drying operating conditions including inlet air temperature (170–190°C), feed flow rate (18–30 mL/min), and blower speed varied (2,000–2,400 rpm) were used as independent variables. The responses evaluated were ascorbic acid content, anthocyanin content, moisture content, hygroscopicity, and water solubility index. Statistical analysis showed that among the independent variables, inlet air temperature showed greater effect on all the investigated responses. The derived optimum conditions were used for the powder production to check the validity of the quadratic model. Small deviations were observed between the experimental values and the predicted ones and the values were within the acceptable limits. The results showed that the optimum spray drying operating conditions for the production of pomegranate juice powder with optimum quality were 171°C inlet temperature, 30 mL/min feed flow rate, and 2,400 rpm blower speed. Under these optimum conditions, quality pomegranate juice powder with desirable properties of high content of vitamin C and anthocyanin, low moisture content, low hygroscopicity, and high solubility could be produced.

Subjects: Bioscience; Environment & Agriculture; Food Science & Technology

Keywords: pomegranate juice powder; optimize; spray drying; vitamin C; anthocyanin

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PUBLIC INTEREST STATEMENT

The process variables related to spray drying can be considered very important, since they directly are related with powder quality. Thus, in the present study, optimization of spray drying operating conditions for the production of quality spray-dried pomegranate juice powder was done. The results can be helpful for food companies looking for the production of quality pomegranate juice powder with high retention of anthocyanin and vitamin C content.

1. Introduction

Pomegranate (*Punica granatum* L.) is an important fruit cultivated in parts of Asia, North Africa, the Mediterranean, and the Middle East (Sarkhosh, Zamani, Fatahi, & Ebadi, 2006). Pomegranate fruit is round in shape with hard yellow-red outer skin. The small edible seeds and pulp are embedded in a clear membrane. The edible part of the fruit contains considerable amount of acids, sugars, vitamins, polysaccharides, polyphenols, and important minerals (Al-Maiman & Ahmad, 2002; Vardin & Fenercioglu, 2003). Consumption of pomegranate also claims medicinal uses including reduced risk of coronary heart disease, stroke, certain types of cancers, and aging (Sumner et al., 2005). Kulkarni and Aradhya (2005) reported that pomegranate juice extracted from pulp is an important source of anthocyanins (cyanidin, delphinidin, pelargonidin), phenolics and tannins (punicalin, pedunculagin, punicalagin, ellagic acid). Pomegranate juice contains almost three times the total antioxidant capacity compared with the same quantity of green tea or red wine (Gil, Tomás-Barberán, Hess-Pierce, Holcroft, & Kader, 2000).

Pomegranate fruit is seasonal and has a limited shelf life under ambient conditions. It creates heavy glut during production season and becomes scanty during off season. High nutritional value makes it desirable to have a pomegranate fruit/juice available throughout the year. Also, the demand for pomegranate juice powder as a base for formulation of new food products is increasing both in domestic and in international markets. So, this time-bound availability and high nutritional value of pomegranate fruit provide a driving force for the production of good quality pomegranate juice powder.

Spray drying technique is extensively used in industries for the transformation of wide range of products in powder form (Quek, Chok, & Swedlund, 2007). Spray drying results in powders with good quality, low water activity, longer shelf life, and ease of transport. Spray-dried fruit juice powders have some inherent problems such as stickiness and high hygroscopicity because of the presence of low-molecular weight sugars and acids which have low glass transition temperatures (Bhandari, Senoussi, Dumoulin, & Lebert, 1993; Shrestha, Howes, Adhikari, & Bhandari, 2007). These problems can be resolved by the addition of high-molecular weight carrier agents like maltodextrin.

The physicochemical properties of powders produced by spray drying technique depend upon some process variables like inlet air temperature, feed flow rate, feed characteristics, etc. (Bhusari, Muzaffar, & Kumar, 2014). Therefore, it is worthwhile to identify the ideal conditions for the production of quality pomegranate juice powder that exhibited high content of vitamin C and anthocyanin.

2. Materials and methods

2.1. Raw materials

Well-matured, fully ripened pomegranate (*Punica granatum*) fruit of Kandhari variety was procured from local market. Pectinase from Aspergillus *niger* (SRL, India) with activity 3.5–7 units/mg was used for clarification of pomegranate juice. Maltodextrin (DE 20) manufactured by Himedia, India, was used as a carrier.

2.2. Preparation of juice and spray drying

Fruits were washed with water, drained, and then the outer leathery skin which enclosed fleshy sacs was peeled off. The juice localized in the sacs was extracted with the help of a manually operated hydraulic press. The juice was treated with 0.0009% pectinase enzyme and incubated at 40°C for 90 min and then filtered to remove the suspended material from the juice. A blend of clarified pomegranate juice and maltodextrin (carrier agent) in the ratio of 75:25 (v/w) based on preliminary trials was prepared, which was used as a feed material for subsequent drying.

Pilot plant spray dryer (S.M. Scientech, India) with a cocurrent air flow was used for spray drying. The feed mixture was fed to the dryer by means of peristaltic pump. The independent variables affecting the powder quality were inlet air temperature, feed flow rate, and blower speed. Inlet air temperature, feed flow rate, and blower speed were varied from 170 to 190°C, 18 to 30 mL/min, and 2,000 to 2,400 rpm, respectively, according to an experimental design. After the completion of each experimental run, the powder was collected from the cyclone and the cylindrical parts of the dryer chamber by lightly sweeping the chamber wall as proposed by Bhandari et al. (1993). The powders were then packed in polyethylene bags and stored in desiccator for further analysis.

2.3. Analysis of pomegranate juice powder

The prepared pomegranate juice powder was analyzed for different physicochemical properties such as ascorbic acid content, anthocyanin content, moisture content, hygroscopicity, and water solubility index (WSI).

2.3.1. Total ascorbic acid

Total ascorbic acid content of the powder sample was measured according to AOAC microfluorometric method (AOAC, 2012).

2.3.2. Total anthocyanin

Anthocyanin content of pomegranate powder was determined according to the method followed by Ferrari, Germer, and de Aguirre (2012) using spectrophotometric pH differential technique, which is based on the anthocyanin structural transformation with a change in pH (colored at pH 1.0 and colorless at pH 4.5). Anthocyanins were extracted from the prepared powder sample using 70% acetone according to the method followed by Awika, Rooney, and Waniska (2004) with some modifications. Two dilutions of the sample were prepared with potassium chloride (0.025 M) and sodium acetate (0.4 M), which were used as buffer solutions at a pH of 1.0 and 4.5, respectively. Absorbance was measured in a spectrophotometer at 520 and 700 nm. Total anthocyanin content was calculated using the molar extinction coefficient of 26,900 L/cm mol for cyanidin-3-glucoside (cyd-3-glu). Results were expressed as milligrams of cyd-3-glu per 100 g of dry matter.

2.3.3. Moisture content

Moisture content of the powder sample was determined according to AOAC method (2012) Powder sample (2 g) was taken and dried in a vacuum oven at a temperature of 70°C until a constant weight was obtained. The samples were analyzed in triplicates and the mean was recorded.

2.3.4. Hygroscopicity

Hygroscopicity was determined according to the method followed by Tonon, Brabet, and Hubinger (2008) with some modifications. Samples of each powder sample (approximately 1 g) were placed at 25°C in a desiccator containing saturated NaCl solution (75.29% RH). After one week, samples were weighed and hygroscopicity was expressed as g of adsorbed moisture per 100 g dry solids.

2.3.5. Water solubility index

The WSI of the powders was determined as per the method described by Kha, Nguyen, and Roach (2010). Spray-dried pomegranate juice powder (2.5 g) and distilled water (30 mL) were vigorously mixed in a 100-mL centrifuge tube, incubated at 37°C in a water bath for 30 min, and then centrifuged for 20 min at 11,410 g. The supernatant was carefully collected in a pre-weighed beaker and oven-dried at a temperature of 103 ± 2°C. The WSI (%) was calculated as percentage of dried supernatant with respect to the amount of sample taken.

2.4. Experimental design

Response surface methodology (RSM) is an important tool in analyzing the experimental data, resulting in the optimization of conditions for product development. It is used for simultaneous optimization of multiple process variables to achieve desired response variables. RSM was used to derive the optimum process conditions using a three-parameter five-level central composite rotatable design giving 20 experimental runs including six replicates at the center point (Table 1). The independent variables affecting the quality of the end product were the inlet air temperature, feed

Table 1. Experimental design for spray drying runs with their corresponding response values

Experiment No.	Inlet air temperature (°C) (x_1)	Feed Flow rate (mL/min) (x_2)	Blower speed (rpm) (x_3)	Ascorbic acid (mg/100 g)	Anthocyanin (mg/100 gm)	Moisture content (%)	Hygroscopicity (g/100 g)	WSI (%)
1	170	18	2,000	28.91	144.08	6.7	13.71	91.01
2	190	18	2,000	15.56	137.49	5.21	15.24	98.98
3	170	30	2,000	30.01	149.99	7.02	12.34	92.10
4	190	30	2,000	22.81	136.13	5.86	14.94	96.53
5	170	18	2,400	29.86	145.04	6.83	13.84	91.32
6	190	18	2,400	16.02	137.86	5.18	15.42	99.12
7	170	30	2,400	31.56	151.12	6.98	12.38	92.19
8	190	30	2,400	22.48	138.42	5.701	14.98	96.57
9	163.2	24	2,200	28.19	155.17	7.54	10.84	87.96
10	196.8	24	2,200	11.52	137.39	5.17	15.08	98.95
11	180	14	2,200	23.95	138.33	5.47	15.21	95.32
12	180	34	2,200	26.87	145.28	6.19	14.3	93.81
13	180	24	1,864	24.16	139.1	5.88	14.81	93.88
14	180	24	2,536	24.85	145.09	5.82	14.98	94.67
15	180	24	2,200	23.97	142.1	5.98	14.74	94.02
16	180	24	2,200	21.05	141.05	5.67	14.94	94.01
17	180	24	2,200	22.97	140.77	5.82	15.01	93.89
18	180	24	2,200	23.65	141.98	5.06	14.35	93.11
19	180	24	2,200	23.12	142.71	5.88	14.72	94.33
20	180	24	2,200	23.09	140.35	5.25	14.09	93.95

Note: All the responses are mean values of three replicates.

flow rate, and blower speed. Response variables including vitamin C content, anthocyanin content, moisture content, hygroscopicity, and solubility were used as quality characteristics of pomegranate juice powder. A second-order polynomial equation was used to relate the response variable (Y_i) with independent variables (X_i).

$$Y_k = \beta_{k0} + \sum_{i=1}^{n} \beta_{ki} x_i + \sum_{i=1}^{n} \beta_{kii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{kij} x_i x_j$$

where Y_k is the response variable, Y_1 is the ascorbic acid (mg/100 g), Y_2 is the anthocyanin (mg/100 g), Y_3 is the moisture content (%), Y_4 is the hygroscopicity (g/100 g), and Y_5 is the water solubility index (%). x_i represents the coded independent variable (x_1 is the inlet air temperature, x_2 is the feed flow rate, x_3 is the blower speed); where β_{k0} was the value of the fitted response at the center point of the design, i.e. point (0,0,0). β_{ki}, β_{kii}, and β_{kij} were the linear, quadratic, and cross-product regression coefficients, respectively.

3. Results and discussion

The numerical values of the responses for each experimental run are listed in Table 1. ANOVA data for each response variable and its significance at 95% confidence level along with correlation coefficient are shown in Table 2. From the ANOVA data, it is confirmed that the fitted models were suitable, showing significant regression, low residual values, no lack of fit with satisfactory determination coefficients (R^2) of 0.9647, 0.9711, 0.9205, 0.9657, and 0.9848 for the responses including ascorbic acid content, anthocyanin content, moisture content, hygroscopicity, and WSI, respectively. The graphical representation for each response was developed as simultaneous function of the two independent variables according to their significance to the response (Figures 1 and 2).

Table 2. Significant levels of pomegranate juice powder responses using RSM

P > F	Ascorbic acid (mg/100 g)	Anthocyanin (mg/100g)	Moisture content (%)	Hygroscopicity (%)	WSI (%)
Model	<0.0001	<0.0001	0.0002	<0.0001	<0.0001
x_1 (Inlet air temperature)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
x_2 (Feed flow rate)	0.0013	0.0004	0.0189	0.0010	0.0101
x_3 (Blower speed)	0.4501	0.0069	0.8485	0.5581	0.3048
x_1^2	0.0397	0.0008	0.0023	<0.0001	0.8647
x_2^2	0.0074	0.7567	0.1637	0.7579	0.0147
x_3^2	0.0361	0.9917	0.1406	0.3698	0.0590
x_1x_2	0.0144	0.0034	0.3902	0.0344	0.0004
x_1x_3	0.5353	0.8681	0.7282	0.9545	0.8739
x_2x_3	0.9600	0.5461	0.7096	0.7931	0.8175
R^2	0.9647	0.9711	0.9205	0.9657	0.9848
Lack of fit	Not significant	Not significant	Not significant	Not significant	Not significant

Figure 1. Effect of inlet air temperature and feed flow rate on (a) ascorbic acid and (b) anthocyanin content of pomegranate juice powder.

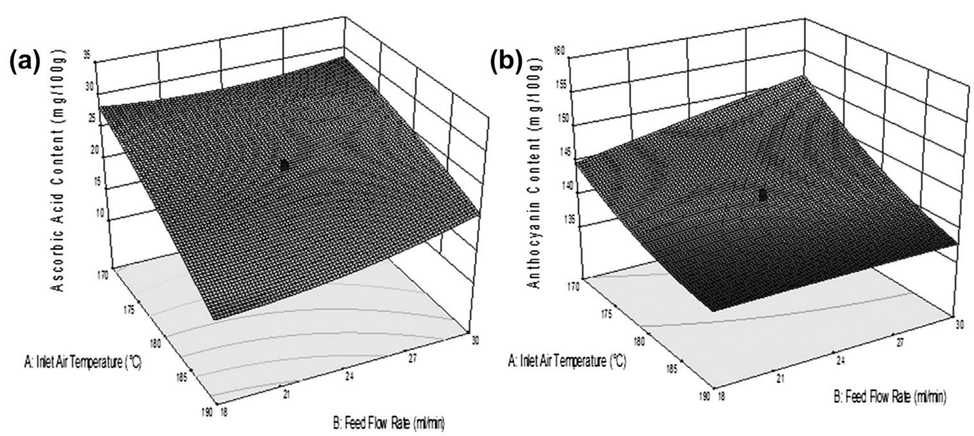
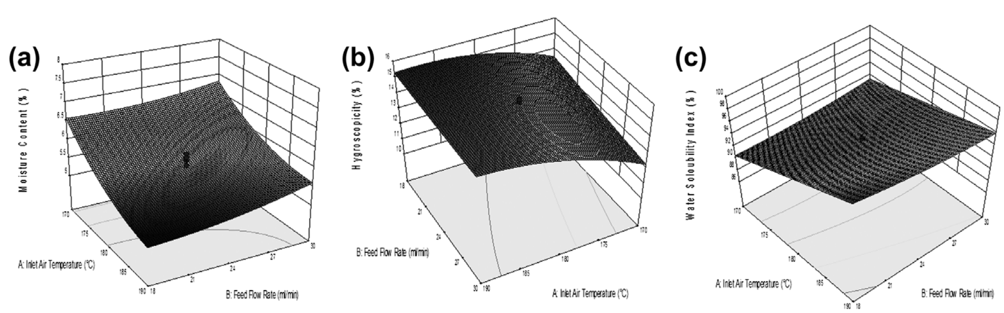


Figure 2. Effect of inlet air temperature and feed flow rate on (a) moisture content, (b) hygroscopicity, and (c) water solubility index of pomegranate juice powder.



3.1. Ascorbic acid

The coefficients of the first-order terms for variables (Equation 1) indicated that with increase in inlet air temperature, there was a decrease in ascorbic acid content, which can be attributed to the destruction of ascorbic acid at high inlet temperature. With increase in temperature, the rate of conversion of ascorbic acid to 2, 3-diketogulonic acid markedly increases, thereby reducing the vitamin C activity (Gregory, 1996). Similar reports of loss of vitamin C with increase in inlet temperature were reported by Patil, Chauhan, and Singh (2014) during the production of spray-dried guava powder. The feed flow rate showed a positive effect on ascorbic acid content, which may be related to the slower heat and the mass transfer occurring when the process was carried out with higher feed flow rates. Blower speed showed a little positive non-significant effect on ascorbic acid content (Table 2).

$$\begin{aligned}\text{Ascorbic Acid Content } &= +22.93 - 5.24^{*}x_1 + 1.57^{*}x_2 + 0.28^{*}x_3 - 0.81^{*}x_1^2 + 1.15^{*}x_2^2 \\ &\quad + 0.83^{*}x_3^2 + 1.36^{*}x_1x_2 - 0.30^{*}x_1x_3 - 0.24^{*}x_2x_3\end{aligned} \tag{1}$$

where x_1 is the inlet air temperature, $x_2$is the feed flow rate, and $x_3$is the blower speed.

3.2. Anthocyanin content

From Equation (2), it is clear that with increase in inlet air temperature, there was a decrease in anthocyanin content, and with increase in feed rate and blower speed, there was an increase in anthocyanin content.

$$\begin{aligned}\text{Anthocyanin Content } &= +141.53 - 5.14^{*}x_1 + 1.68^{*}x_2 + 1.09^{*}x_3 + 1.48^{*}x_1^2 - 0.099 \\ &\quad {}^{*}x_2^2 + 0.0033^{*}x_3^2 - 1.60^{*}x_1x_2 + 0.071^{*}x_1x_3 + 0.26^{*}x_2x_3\end{aligned} \tag{2}$$

Anthocyanin loss with increase in inlet temperature is due to the high sensitivity of these pigments to high temperature. These results are in accordance with the findings of Tonon et al. (2008) in spray drying of acai pulp. Besides this, anthocyanin degradation at higher temperature may also be related to the presence of sugars and proteins, which can result in Maillard reaction at higher temperatures. According to Von Elbe and Schwartz (1996), Maillard reaction products furfural and hydroxymethylfurfural condense together with the anthocyanins, leading to the formation of new compounds with brown coloration. Increase in anthocyanin content with increase in feed rate is related to the increase in moisture content, providing higher tendency for the powder to agglomerate, reducing the powder exposition to oxygen and protects the pigments against oxidation. Similar results were pointed out by Quek et al. (2007). Increase in blower speed showed a positive effect on anthocyanin content, which may be due to the increase in flow rate of air which consequently reduces the time elapsed by feed particles in the dryer chamber and thus minimizes the exposure time to higher temperature.

3.3. Moisture content

From Equation (3), it is clear that inlet air temperature shows maximum negative effect followed by feed rate having positive linear effect on the moisture content. The decrease in moisture content at higher inlet air temperatures is due to the greater temperature gradient between the atomized feed and the drying air, resulting in a greater driving force for water evaporation and thus produces powders with lower moisture content. Muzaffar and Kumar (2015) also observed similar results during spray drying of watermelon juice. Increase in feed flow rate offers shorter contact time between the feed and the drying air, making the heat transfer less efficient and resulting in lower water evaporation. The blower speed slightly decreased the moisture content of pomegranate juice powder which may be due to the increase in hot air flow rate at higher blower speed making efficient heat and mass transfer.

$$\begin{aligned}\text{Moisture Content} &= +5.61 - 0.70^{*}x_1 + 0.21^{*}x_2 - 0.015^{*}x_3 + 0.29^{*}x_1^2 + 0.11^{*}x_2^2 \\ &\quad + 0.12^{*}x_3^2 + 0.088^{*}x_1x_2 - 0.035^{*}x_1x_3 - 0.037^{*}x_2x_3\end{aligned} \tag{3}$$

3.4. Hygroscopicity

As given in Equation (4), it shows that inlet air temperature was the variable that has greater effect on powder hygroscopicity. The powders with lowest hygroscopicity values were obtained at higher feed flow rate and low inlet temperature, which were the variables that affected powder moisture content in an opposite way. This indicates that the lower the moisture content of the powder, greater is its tendency to absorb moisture, which is related to the greater water concentration gradient between the powder and the surrounding air. Similar findings were pointed out by Goula, Adamopoulos, and Kazakis (2004) during spray drying of tomato pulp. Blower speed showed a little positive non-significant effect on ascorbic acid content (Table 2).

$$\begin{aligned}\text{Hygroscopicity} = &+14.64 + 1.13^{*}x_1 - 0.37^{*}x_2 + 0.049^{*}x_3 - 0.61^{*}x_1^2 + 0.025^{*}x_2^2 \\ &+ 0.075^{*}x_3^2 + 0.26^{*}x_1x_2 + 0.006^{*}x_1x_3 - 0.029^{*}x_2x_3\end{aligned} \tag{4}$$

3.5. WSI

As given in Equation (5), the coefficients of the first-order terms for variables indicated that inlet air temperature has maximum positive effect and blower speed has a slight positive effect on WSI of pomegranate juice powders, while feed flow rate showed negative effect on WSI.

$$\begin{aligned}\text{WSI} = &+93.87 + 3.15^{*}x_1 - 0.41^{*}x_2 + 0.14^{*}x_3 - 0.022^{*}x_1^2 + 0.37^{*}x_2^2 + 0.27^{*}x_3^2 \\ &- 0.87^{*}x_1x_2 - 0.027^{*}x_1x_3 - 0.040^{*}x_2x_3\end{aligned} \tag{5}$$

Effect of the process variables on solubility is related to their effect on residual moisture content of powder samples. The lower the moisture content of the powder sample, the more soluble it is (Goula & Adamopoulos, 2005). Powders produced at higher temperature and lower feed rate resulted in powders with higher solubility due to their negative effect time on moisture content of the powder sample. Similar trend was reported by Muzaffar and Kumar (2015) during spray drying of tamarind pulp. Increase in water solubility with increase in blower speed is also related to its negative effect on powder moisture content.

3.6. Optimization

A numerical multi-response optimization technique of Design expert software 8.0.2 (Statease Inc., Minneapolis, USA) was used to determine the optimum conditions for the spray drying of pomegranate juice powder. The criterion for each independent variable and response was chosen, based on desired characteristics of pomegranate juice powder (Table 3). The optimization was applied to the selected ranges of inlet air temperature, feed flow rate, and blower speed, and the optimum level of the independent variables with predicted values of the responses was generated by Design Expert 8.0.2 software. By applying desirability function method, the solution was obtained for the optimum covering criteria with desirability value of 0.602. Inlet air temperature, feed flow rate, and blower speed of 171°C, 30 mL/min, and 2,400 rpm, respectively, were obtained as the most desirable solutions for the optimum spray drying operating conditions. Under this optimized condition, the predicted values for ascorbic acid content, anthocyanin content, moisture, hygroscopicity, and solubility were 29.84 mg/100 g, 151.68 mg/100 g, 6.81, 12.63, and 92.140%, respectively, close to the experimental values of 31.32 mg/100 g, 150.46 mg/100 g, 5.92, 13.95, and 93.57%, respectively, indicating the suitability of the model in optimizing the spray drying operating conditions for the production of quality pomegranate juice powder.

Table 3. Criteria and outputs of the numerical optimization of the responses for pomegranate juice powder

Variables	Goal	Lower limit	Upper limit	Importance
Inlet air temperature	Minimize	170	190	3
Feed flow rate	Is in range	18	30	3
Blower speed	Is in range	2,000	2,400	3
Ascorbic acid	Maximize	11.52	31.56	3
Anthocyanin	Maximize	136.13	155.17	3
Moisture content	Minimize	5.06	7.54	3
Hygroscopicity	Minimize	10.84	15.42	3
WSI	Maximize	87.96	99.12	3

4. Conclusion

Different tests were carried to study the effect of spray drying operating conditions on the quality attributes of spray-dried pomegranate juice powder. All the responses were affected by the process conditions. Inlet air temperature showed maximum effect on the process variables. Higher losses in ascorbic acid and anthocyanin content were observed at higher temperature, which is due to the thermal degradation of these compounds. It was concluded that RSM was effective in optimizing the process parameters for the production of spray-dried pomegranate juice powder with desirable values for all the responses studied. The best operating conditions for spray drying of pomegranate juice were 171°C inlet air temperature, 30 mL/min feed flow rate, and 2,400 rpm blower speed.

Acknowledgment
The author is thankful to Department of Food Engineering and Technology, SLIET, Longowal, India, for giving their support for smooth conduct of research.

Funding
The authors received no direct funding for this research.

Competing interests
The authors declare no competing interest.

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Citation information
Cite this article as: Optimization of spray drying conditions for production of quality pomegranate juice powder, Khalid Muzaffar, Bijamwar Vilas Dinkarrao & Pradyuman Kumar, *Cogent Food & Agriculture* (2016), 2: 1127583.

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