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ORIGINAL ARTICLE

Optimizing spray drying conditions of sour cherry juice based on physicochemical properties, using response surface methodology (RSM)

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Abstract In this study, the effects of main spray drying conditions such as inlet air temperature (100–140 °C), maltodextrin concentration (MDC: 30–60%), and aspiration rate (AR) (30–50%) on the physicochemical properties of sour cherry powder such as moisture content (MC), hygroscopicity, water solubility index (WSI), and bulk density were investigated. This investigation was carried out by employing response surface methodology and the process conditions were optimized by using this technique. The MC of the powder was negatively related to the linear effect of the MDC and inlet air temperature (IT) and directly related to the AR. Hygroscopicity of the powder was significantly influenced by the MDC. By increasing MDC in the juice, the hygroscopicity of the powder was decreased. MDC and inlet temperature had a positive effect, but the AR had a negative effect on the WSI of powder. MDC and inlet temperature negatively affected the bulk density of powder. By increasing these two variables, the bulk density of powder was decreased. The optimization procedure revealed that the following conditions resulted in a powder with the maximum solubility and minimum hygroscopicity: MDC = 60%, IT = 134 °C, and AR = 30% with a desirability of 0.875.

Keywords Fruit juice powder · Sour cherry · Spray drying · Optimization · Response surface methodology (RSM) · Physicochemical properties

Introduction

Sour cherry is rich in bioactive compounds, including anthocyanin, polyphenols, and ascorbic acid. These compounds have many health benefits such as anti-neurodegeneration, anti-inflammation, anti-cancer, which inhibits the growth of tumor cells, and are useful in diabetic control (Bobe et al. 2006; Kang et al. 2003; Lachin and Reza 2012).

Spray drying is the process of converting liquid materials into the powder form. The liquid is sprayed into a chamber and mixes with hot air that evaporates the moisture from the sprayed liquid, and as a result, its solid components will leave the chamber in the form of dry particulates. Transforming fruit juice into the powder has lots of benefits such as increased shelf life, reduced weight, and convenient handling and transportation (Goula et al. 2004). However, this process is not easy because the major components of fruit juice including organic acids and low molecular weight sugars have a low glass transition temperature, low melting point, high hygroscopicity, and high water solubility which make them sticky. As a result, during spray drying, much of the powder sticks to the chamber wall (Adhikari et al. 2009a, b). Therefore, this kind of products cannot be dried without using any additive, so drying aids are used to produce fruit juice powder (Jayasundera et al. 2011).

According to the literature, the most studied factors in producing fruit juice powder by spray drying operation are the concentration of drying aid and drying temperature.

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Goula and Adamopoulos (2010) studied the effects of maltodextrin type and different juice/maltodextrin ratios on the physicochemical properties of orange juice powder. Mishra et al. (2014) studied the influence of inlet temperature and maltodextrin concentration on the physicochemical properties, free radical scavenging, and total phenolic content of amla powder. Caliskan and Dirim (2013), studied the feasibility of converting sumac extract into the powder form using spray drying process and also the effect of maltodextrin concentration and inlet/outlet temperature of drying air on the properties of the powder (Caliskan and Dirim 2013).

Since sour cherry is a perishable fruit with a short harvesting period and has many health promoting effects, the aim of this study was to produce sour cherry powder using spray drying process and also evaluate the effects of various maltodextrin concentrations and different inlet drying air temperatures on the physicochemical properties of the powder. Finally, the process parameters were optimized by applying the response surface methodology.

Materials and methods

Raw materials

Sour cherry concentrate with 65° Brix was obtained from Azarkam Co., Urmia, Iran. Its pH and acidity was 3.52 and 3.5 g malic acid/100 g concentrate, respectively.

Sample preparation and spray drying

The mixture of sour cherry and maltodextrin with 5°Brix was prepared for spray drying operation. The ratio of maltodextrin (DE = 17) to sour cherry juice was calculated based on the dry substance. For example, sample containing 45% maltodextrin means that 45% of the solid content of the sample comes from maltodextrin and 55% of its solid content comes from the dry substance of sour cherry juice. Then, the obtained sample was spray dried using a spray dryer (Mini Spray Dryer B-290, Buchi, Switzerland) with a flow rate of 2 ml/min. The pressure of atomizer was constant (4 bar) during the process.

Moisture content (MC)

Three g of spray dried powder was vacuum dried at 70 °C in a vacuum oven (TOWNSON & MERCER LTD) and it was weighed at 2 h interval until reaching 0.3% difference between two intervals. The moisture content of the sample was expressed in wet basis (Goula and Adamopoulos 2010).

Hygroscopicity

This experiment was conducted according to the Cai and Corke (2000) procedure with a slight modification. Two g of powder was placed in an airtight container containing saturated sodium chloride solution at 25 °C ($RH = 75 \pm 0.5\%$). After 1 week, samples were weighed and the hygroscopicity was measured by dividing the amount of absorbed moisture (g) by 100 g powder.

Water solubility index (WSI)

This physical property of powder was obtained according to the procedure described by Anderson et al. (1969). Two g of powder was vigorously mixed with 25 ml distilled water and was placed in a water bath at 37 °C for 30 min. Then, this solution was centrifuged at 10,000*g* for 20 min. The supernatant was oven dried at 105 °C and the WSI (%) was calculated by dividing the weight of the dried supernatant by the initial weight of the sour cherry powder.

Bulk density

Two gram of powder was placed in a 50 ml graduated cylinder and was shaken using a vortex vibrator for 1 min. For determining bulk density, the mass of the powder was divided by its occupied volume in the cylinder (Goula and Adamopoulos 2005).

Scanning electron microscopy (SEM)

In order to investigate the effects of drying conditions on the microstructure of the powder, scanning electron microscopy (SEM, KYKY-EM3200, China) operation was conducted at 26 kV. For better conductivity, surface of the powder was coated with a thin layer of gold.

Experimental design and statistical analysis

Response surface methodology (RSM) was applied in order to determine the effects of most important spray drying operational factors on the physicochemical properties of sour cherry powders. These factors were maltodextrin (carrier) concentrations (x_1: 30–60%), inlet temperature (x_2: 100–140 °C), and aspiration rate (x_3: 30–50%) each at three levels (Table 1). The studied physicochemical properties were moisture content (y_1), hygroscopicity (y_2), WSI (y_3), and bulk density (y_4). According to the central composite design (CCD), twenty treatments were conducted (Table 2).

In order to minimize the effects of unexplained variability in responses due to extraneous factors, the

Table 1 Independent variables and their level in central composite design (CCD)

Independent variables	Independent variable level		
	Low	Center	High
Maltodextrin concentration (% w/w)	30	45	60
Inlet temperature (°C)	100	120	140
Aspiration rate (%)	30	40	50

experiment was randomized. Using a second order polynomial function, the relationship between responses (y) and variables (x) were found:

$$y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \tag{1}$$

where y is the predicted value for response, and the coefficients of the function are represented by β_0 (constant term), β_i (linear effect), β_{ii} (quadratic effect), and β_{ij} (interaction effect). By analysis of variance (ANOVA) (Table 3), the significant terms in the model for each response were found. By using R^2 and adjusted-R^2, the adequacy of the model was examined.

Numerical optimization of the spray drying process was performed to optimize the studied process conditions in order to obtain powder with suitable physicochemical properties. Design Expert software (trial version 7.0.0, Stat-Ease Inc., Minneapolis, USA) was used to create the experimental design matrix, analyzing data, and optimizing procedure.

Data analysis

The matrix of central composite design and the experimental data for studied responses are presented in Table 2. The difference between experimental and predicted value for each response by fitting the experimental data to the second order polynomial model (Eq. 1) are shown in Table 4. ANOVA analysis was carried out in order to find out the significance of the independent factors and to check the adequacy of the model (Table 3). The *P* value was used for determining the significance of the coefficients. The lower the value of *P*, the more significant the term. When this value is lower than 0.05, the coefficient is considered as significant.

Results and discussion

Preliminary investigations

A preliminary investigation was carried out in order to find a suitable range of independent variables to be implemented in

Table 2 The matrix of central composite design and the experimental data of studied responses (y)

Run	Block	Independent variables			Response variables			
		MDC (%)	IT (°C)	AR (%)	MC (%)	Hygroscopicity (%)	WSI (%)	Bulk density (g/cm^3)
1	Block 1	45	120	40	5.1	25.0	76.2	0.45
2	Block 1	60	140	50	4.1	21.5	80.1	0.40
3	Block 1	60	100	50	5.7	19.8	74.0	0.49
4	Block 1	45	120	40	5.0	25.8	75.4	0.46
5	Block 1	45	120	40	5.1	25.9	76.3	0.47
6	Block 1	60	100	30	5.7	19.0	79.0	0.48
7	Block 1	60	140	30	3.8	22.0	82.0	0.39
8	Block 1	45	120	40	5.1	25.6	75.0	0.42
9	Block 1	30	100	50	6.1	28.0	70.7	0.52
10	Block 1	30	140	30	4.1	29.5	79.7	0.42
11	Block 1	30	100	30	5.9	28.7	71.9	0.50
12	Block 1	30	140	50	4.4	29.1	77.1	0.43
13	Block 2	45	120	40	5.1	24.8	75.7	0.49
14	Block 2	45	120	40	5.1	25.3	76.6	0.47
15	Block 2	45	120	50	5.2	25.6	74.1	0.44
16	Block 2	60	120	40	4.9	20.3	79.6	0.44
17	Block 2	30	120	40	5.2	30.1	73.2	0.48
18	Block 2	45	100	40	5.7	24.3	74.1	0.50
19	Block 2	45	120	30	4.9	26.0	77.1	0.45
20	Block 2	45	140	40	4.4	26.5	77.9	0.42

Table 3 Table of ANOVA for the effect of independent variable on the responses and the estimated regression coefficient of polynomial equation

Source	DF	Moisture Content (%)			Hygroscopicity (%)			WSI (%)			Bulk density (g/cm^3)		
		Coefficient	Sum of squares	*P* value	Coefficient	Sum of squares	*P* value	Coefficient	Sum of squares	*P* value	Coefficient	Sum of squares	*P* value
Model	9	5.0800	0.015	<0.0001	25.5200	194.3900	<0.0001	75.7900	149.3200	<0.0001	0.4600	0.0210	<0.0032
Linear													
b_1	1	−0.1500	0.230	<0.0013	−4.2800	183.1800	<0.0001	2.2100	48.8400	<0.0001	−0.0150	2.2500E−03	<0.0263
b_2	1	−0.8300	6.890	<0.0001	0.8800	7.7400	<0.0007	2.7100	73.4400	<0.0001	−0.0430	0.0180	<0.0001
b_3	1	0.1100	0.120	<0.0084	−0.1200	0.1400	0.5058	−1.3700	18.7700	<0.0017	0.0040	1.6000E−04	0.4969
Quadratic													
b_{11}	1	−0.0340	0.003018	0.6090	−0.5200	0.7200	0.1593	0.7300	1.4400	0.2515	0.004706	5.9440E−05	0.6762
b_{22}	1	−0.0340	0.003018	0.6090	−0.3200	0.2700	0.3715	0.3300	0.3000	0.5917	0.004706	5.9440E−05	0.6762
b_{33}	1	−0.0340	0.003018	0.6090	0.0830	0.0190	0.8101	−0.0680	0.0120	0.9124	−0.0100	2.8440E−04	0.3698
Interaction													
b_{12}	1	0.0000	0.000	1.0000	0.3500	0.9800	0.1063	−0.6400	3.2500	0.0987	−0.0013	1.2500E−05	0.8475
b_{13}	1	−0.0250	0.005	0.5123	0.1800	0.2500	0.3929	−0.3900	1.2000	0.2919	−0.0013	1.2500E−05	0.8475
b_{23}	1	0.0500	0.020	0.2056	−0.1200	0.1300	0.5376	0.2100	0.3600	0.5545	−0.0013	1.2500E−05	0.8475
Residual	9		0.097			2.7400			0.8630			2.8730E−03	
Lack of fit	5		0.090	0.0172		2.1300	0.172		7.0300	0.1225		1.2730E−03	0.6874
Pure error	4		6.2750E−03			0.6100			1.5900			0.0016	
Total	19		7.400			197.7900			158.7700			0.0240	
R^2		0.9869			0.9861			0.9454			0.8809		
Adj-R^2		0.9730			0.9722			0.8908			0.7617		
CV		2.0600			2.1900			1.2800			3.9200		

Indices 1, 2, and 3 refer to maltodextrin concentration, inlet air temperature, and aspiration rate, respectively

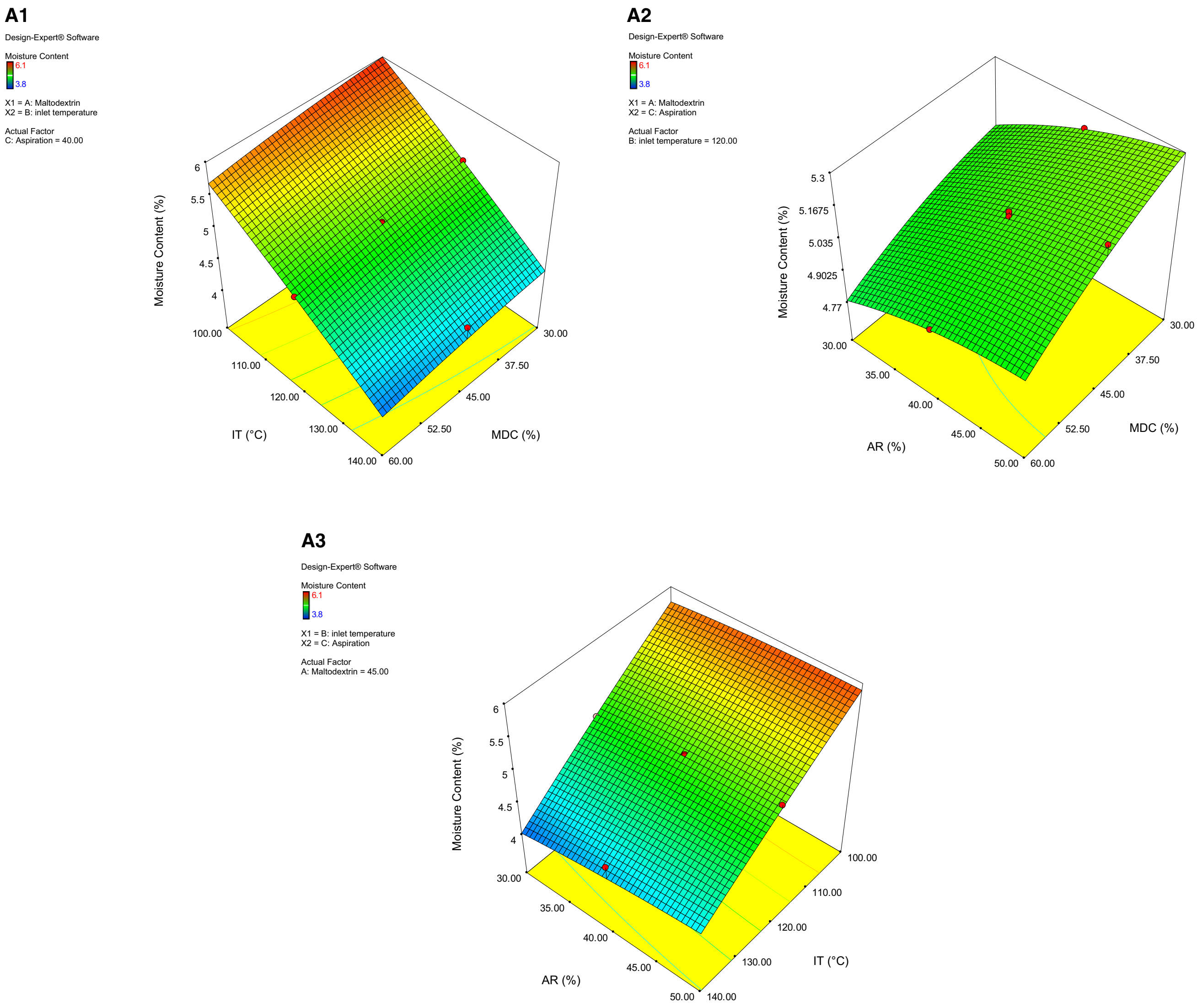

Fig. 1 The interaction effects of independent variables (MDC, IT, and AR) on the physicochemical properties of the sour cherry powder: **a** moisture content, **b** hygroscopicity, **c** WSI, and **d** bulk density

the experimental design. This procedure was performed based on trial and error and the criterion for this experiment was wall deposition. When the T_g of powder fall below the outlet temperature of spray dryer, it will stick to the cyclone wall (Bhandari et al. 2013). Therefore, according to this criterion in the preliminary investigation, the range of IT and MDC in the experimental design was set at 100–140 °C and 30–60%, respectively. The range of AR [30–50% (100% = 80 m^3/h)] was determined based on the powder loss through the cyclone outlet.

Moisture content

As seen in Table 3, Only the linear effect of MDC, IT, and AR had a significant effect on the moisture content of the powder ($P < 0.05$).

According to the estimated coefficients (Table 3), moisture content had a negative relationship with MDC and IT and a positive relationship with AR (Fig. 1). The interaction between factors and their quadratic effects were not statistically significant ($P > 0.05$). IT had a pronounced effect on the final moisture content of the powder than MDC and AR. The negative relationship between moisture content and IT can be attributed to the driving force of evaporation. The higher IT, the higher evaporation rate, thus the lower moisture content at the end of drying. Higher driving force due to the higher temperature gradient between the medium and particles results in a faster heat transfer into the particles thus removing more moisture from it. Our finding was in accordance with Ferrari et al. (2012) and Tonon et al. (2008), who conducted research on producing powder of

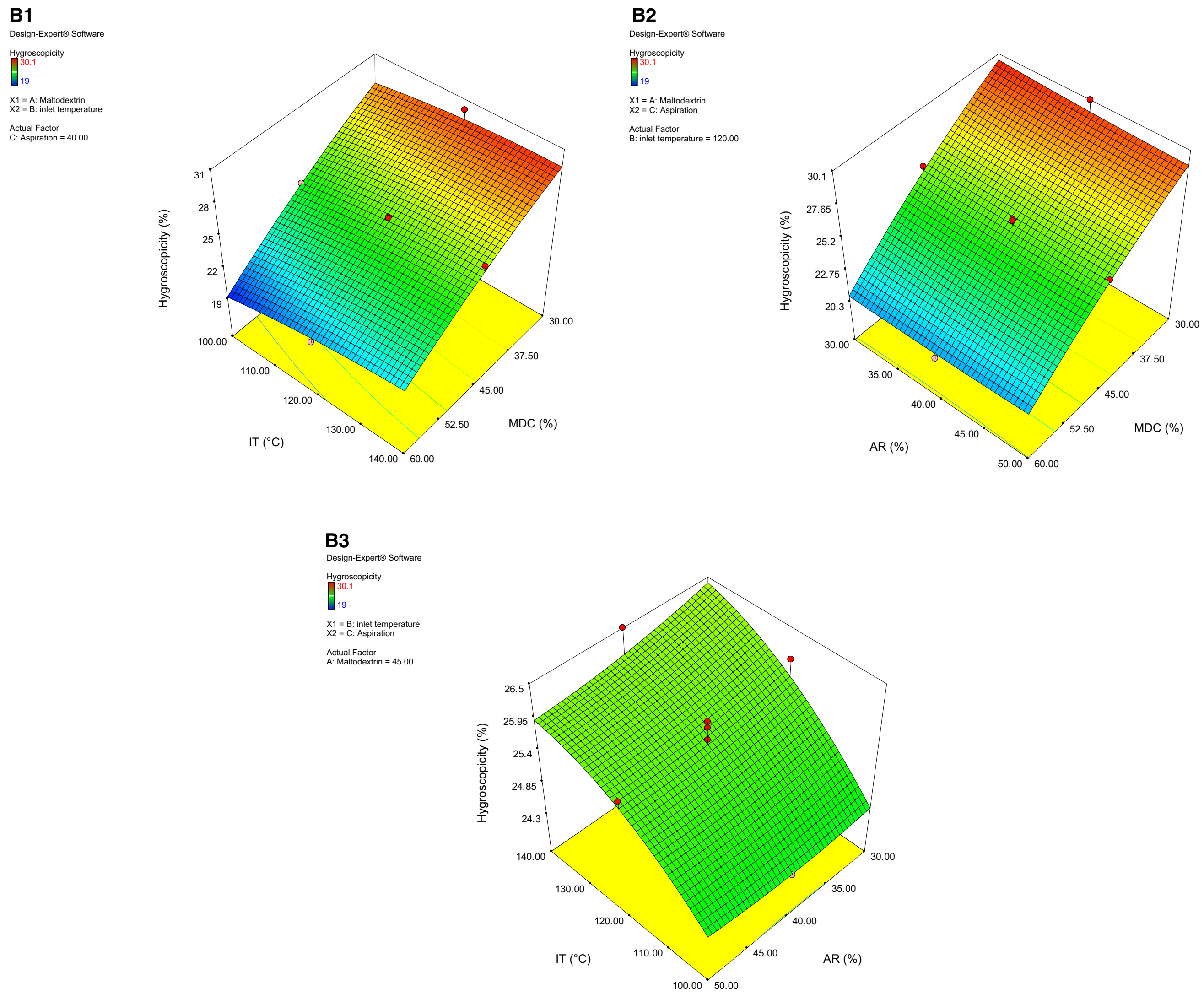

Fig. 1 continued

Blackberry and açaí, respectively. But Santhalakshmy et al. (2015), who spray dried jamun fruit juice at the temperature range of 140–160 °C reported an opposite result which may be due to the different inlet temperature of both studies.

Hygroscopicity

Among the second-order polynomial model terms, only the linear effects of MDC and IT were significant ($P < 0.05$). Between these two factors, the effect of MDC was remarkable (Table 3). In Fig. 1, the effects of different factors on the hygroscopicity are seen.

There was a negative relation between MDC and powder hygroscopicity. By increasing MDC, hygroscopicity decreased and this was due to the low hygroscopicity of maltodextrin. Tonon et al. (2008) reported the lowest hygroscopicity for açai powder when they used the maximum concentration of maltodextrin. Other researchers such as Ferrari et al. (2012) for blackberry powder and Mishra et al. (2014) for amla powder reported similar trend.

The relation between IT and hygroscopicity was positive (Table 3). By increasing IT, the powder hygroscopicity slightly increased (Fig. 1). This shows that the moisture content of powder affects the hygroscopicity. Due to the fast formation of particles at elevated temperature because of greater driving force, they will have an amorphous structure and thus will absorb more moisture from the surrounding air. This is in agreement with the findings of Muzaffar and Kumar (2015) and Santhalakshmy et al. (2015), but in opposite with the result of Mishra et al. (2014).

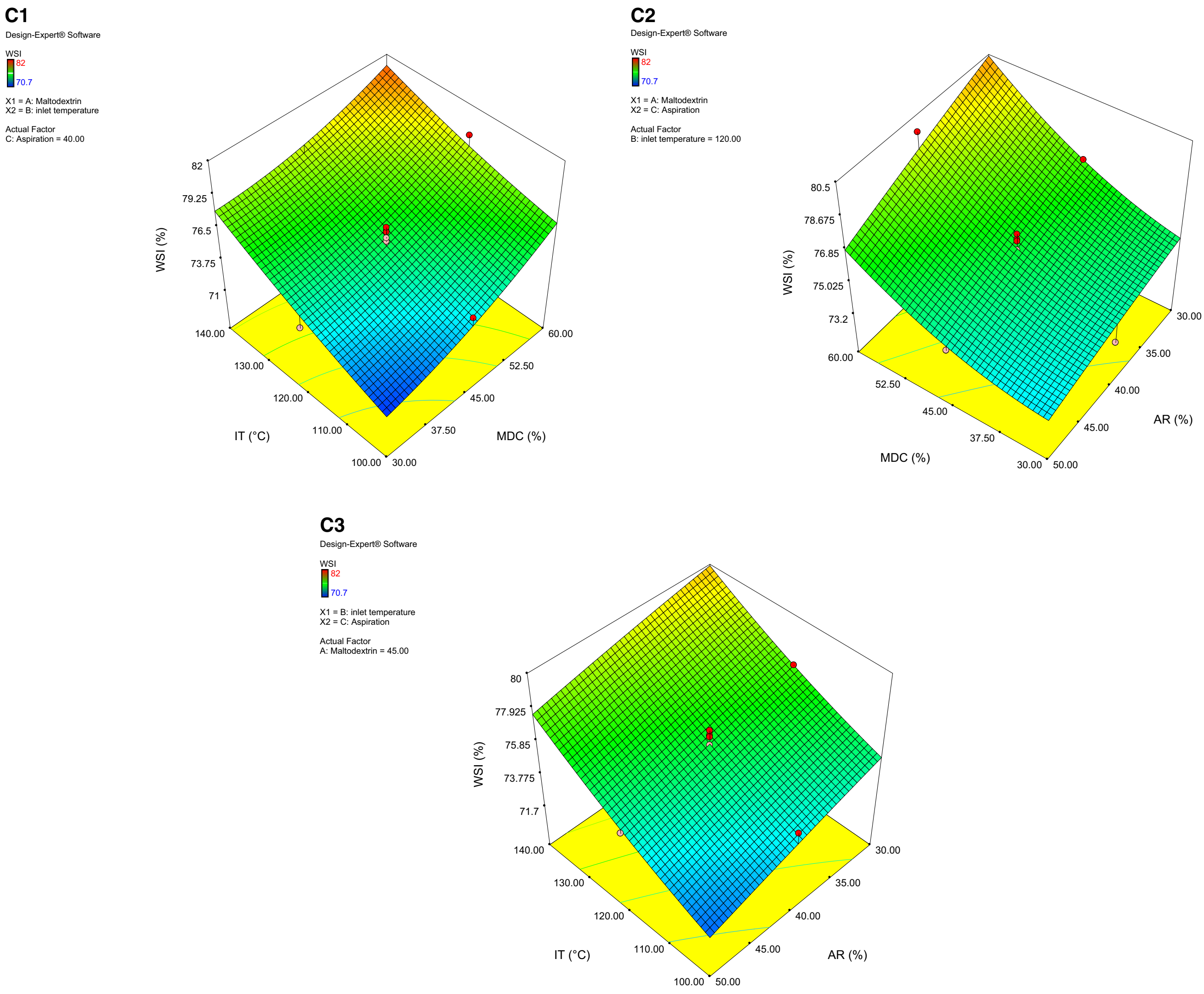

Fig. 1 continued

Water solubility index (WSI)

WSI ranged from 70.7 to 80.0%. According to the data presented in Table 3 only linear effects of independent variables were significant ($P < 0.05$). Other terms, including quadratic and interaction effects on WSI were not significant at the probability of 5%. By increasing MDC, the WSI increased (Fig. 1). This is due to the non-crystalline and amorphous nature of the maltodextrin (Santhalakshmy et al. 2015). Caliskan and Dirim (2013) reported that by increasing MDC, the solubility of sumac powder is increased. Because of the high solubility of maltodextrin in aqueous phase, it is one of the most used carrier agents in spray drying of plant extract (Cano-Chauca et al. 2005).

IT had a positive and significant effect on the WSI ($P < 0.0001$, Table 3). An increase in IT resulted in an increase in powder WSI (Fig. 1). This behavior can be attributed to the effect of IT on the moisture content of the powder. There is an inverse relation between moisture content of the powder and its solubility (Baldwin and Truong 2007; Goula and Adamopoulos 2005). Santhalakshmy et al. (2015), Muzaffar and Kumar (2015) observed the similar trend.

AR had a significant and negative effect on the WSI ($P < 0.05$, Table 3). By increasing AR, WSI decreased. This observation can be explained by the effect of AR on the moisture content of the powder. By reduction in the moisture content of the powder, it's solubility is increased, as stated in the section of the effect of IT on the solubility.

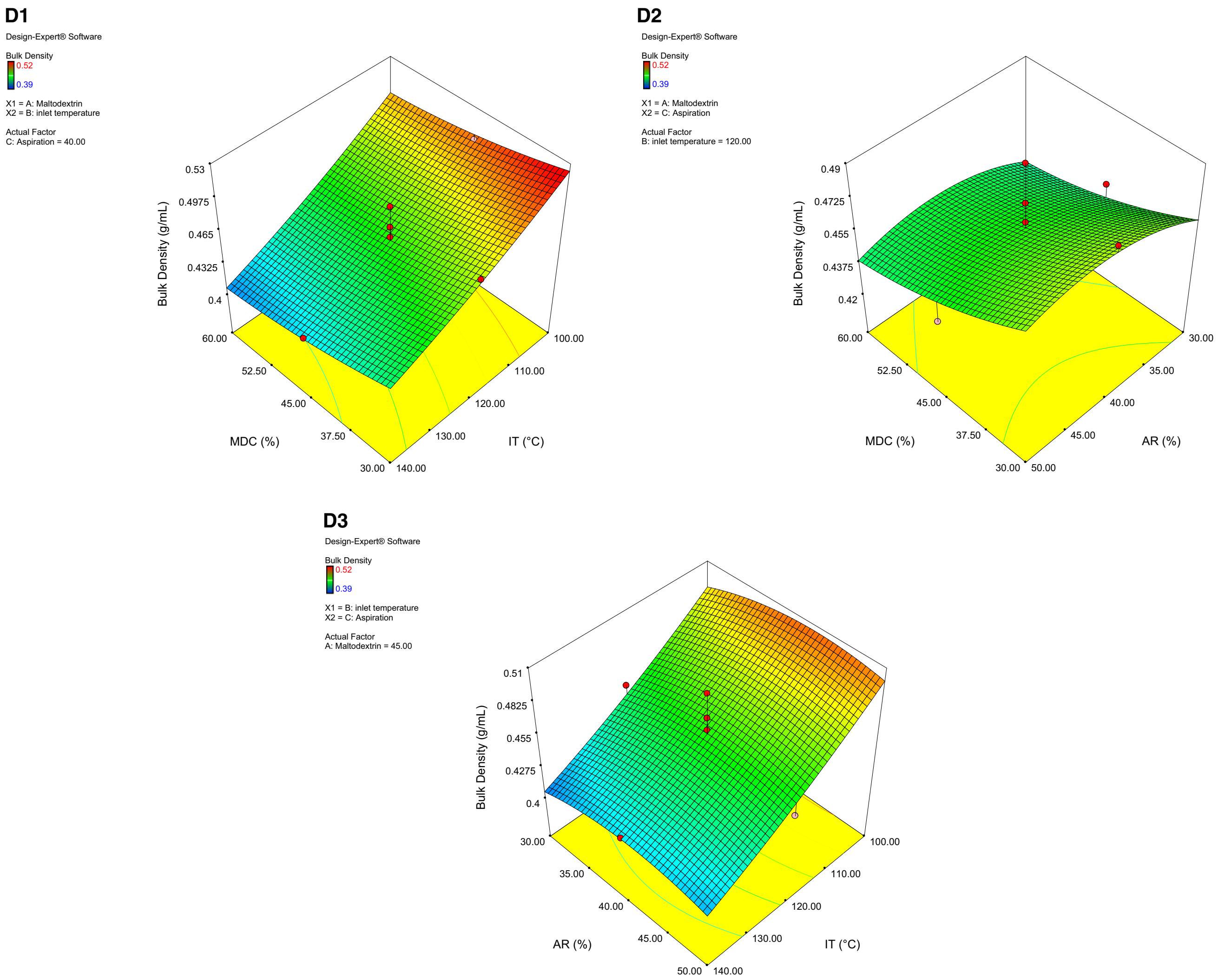

Fig. 1 continued

Our finding is in agreement with the finding of Mishra et al. (2014).

Bulk density

Powder bulk density was significantly affected by the linear effect of MDC and IT ($P < 0.05$, Table 3). At the 5% probability level, AR did not have a significant effect on the bulk density.

Linear effect of MDC, negatively affected the bulk density. By increasing MDC, powder bulk density decreased. This may be attributed to the effect of maltodextrin on the final moisture content of the obtained powder. The lower moisture content, the lower bulk density is. This observation is consistent with the findings of Caliskan and Dirim (2013), but Mishra et al. (2014) reported that the MDC did not have a significant effect on the bulk density of amla powder. This may be due to the low concentration of maltodextrin (5–9% w/v of juice with 40% dry substance) they used in their study.

IT was the most significant factor that affected bulk density of sour cherry powder. This independent factor inversely affected bulk density (Table 3). By increasing IT, bulk density decreased linearly (Fig. 1). By increasing drying air temperature, the evaporation rate increases, thus the resulting powder will have a more porous structure and lower shrinkage and as a result a lower bulk density (Chegini and Ghobadian 2005; Jumah et al. 2000; Walton 2000). Tuyen et al., and Mishra et al., reported the same result (Mishra et al. 2014; Tuyen et al. 2010).

Microstructure of sour cherry powder

Properties of food powder depend on the size, shape, and distribution of its particles. Thus, concerning powders properties should be accompanied by its microstructure (Dodds et al. 2013). Effect of inlet temperature on the

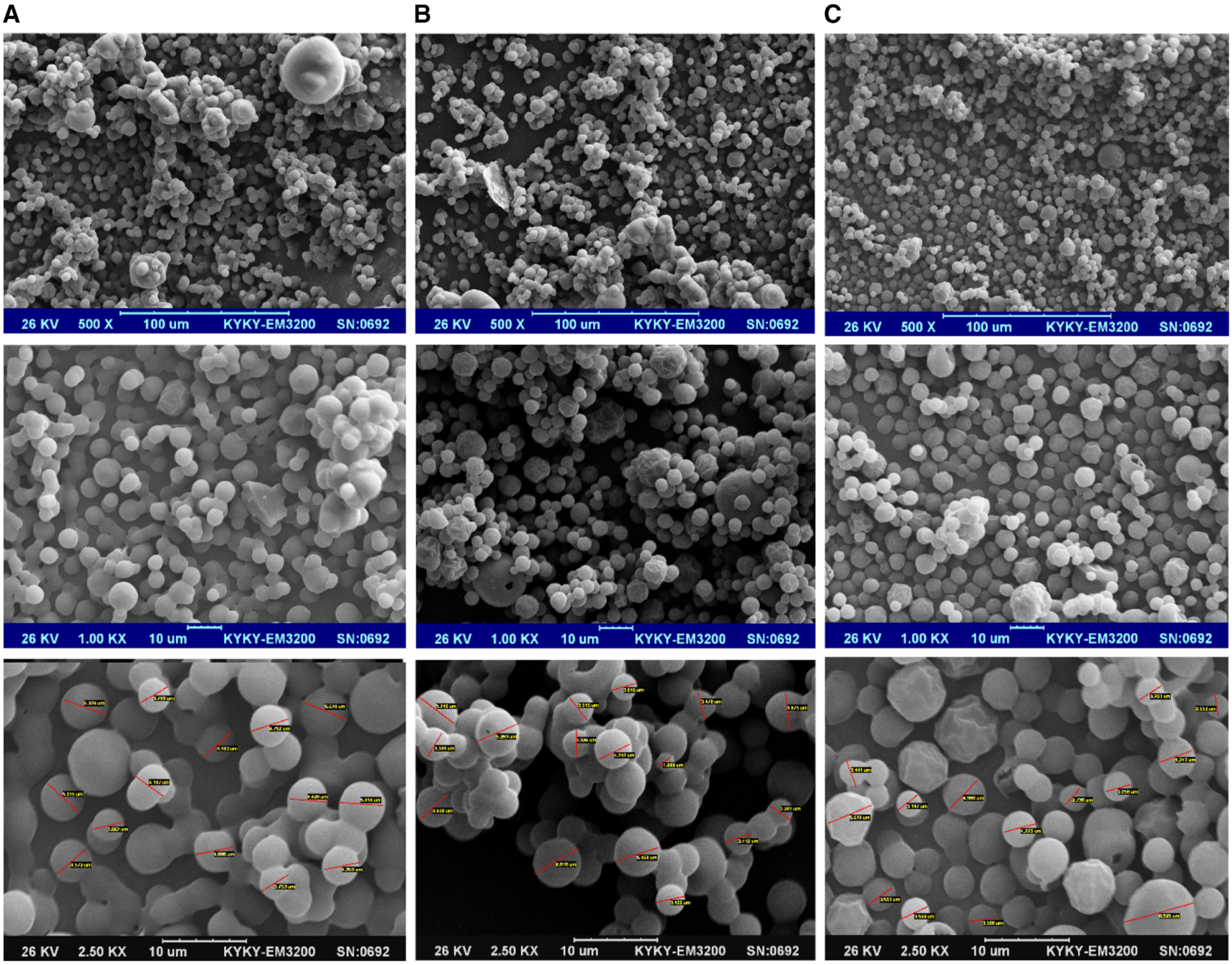

Fig. 2 Micrograph of particles. **a** IT = 140 °C, **b** IT = 100 °C, **c** IT = 120 °C. Micrograph of each sample was taken at three magnitudes of 500×, 1000×, and 2500×. The values of MDC and AR were kept constant at 45 and 40%, respectively

morphological properties of sour cherry powder is seen in Fig. 2. All samples showed spherical particle and smooth surface. At higher temperature (140 °C), the attraction between particles increased and cluster formation took place, but at 120 and 100 °C, there is not a strong attraction between particles. The range of studied temperature did not influence the surface smoothness of particles. As can be seen in Fig. 2, particle sizes are in the scale of μm and most of them fall in the range of 1–6 μm.

Optimization procedure

In order to find out conditions for producing optimum powder, numerical optimization procedure was carried out. Optimum powder was considered as a powder with minimum hygroscopicity and maximum WSI. Moisture content and bulk density were not considered as our goal. Therefore, in numerical optimization using Design Expert software, the goal for hygroscopicity and WSI was set at "minimize" and "maximize", respectively. The importance of both of these variables was set at "three stars (***)". For moisture content and bulk density, the goal was set at "in range". The selected solution provided by the software was MDC = 60%, IT = 134 °C, and AR = 30%. With this condition, we will produce a powder with these properties: Hygroscopicity = 21.5, WSI = 82%, moisture content = 4.1% and bulk density = 0.40 g/cm^3 with a desirability of 0.875.

Conclusion

Sour cherry powder was successfully produced using spray-drying technique. Effects of spray drying conditions on the physicochemical properties were evaluated using central composite design. All process conditions, namely IT, MDC, and AR had a significant effect on the

Table 4 Experimental and predicted values of response variables

MC (%)			Hygroscopicity			WSI (%)			Bulk density (g/cm3)		
Experimental	Predicted	Difference	Experimental	Predicted	Difference	Experimental	Predicted	Difference	Experimental	Predicted	Difference
5.1	5.1	0.0	25.0	28.5	3.5	76.2	75.8	0.4	0.45	0.46	0.01
4.1	4.1	0.0	21.5	26.0	4.5	80.1	79.5	0.6	0.40	0.40	0.00
5.7	5.7	0.0	19.8	22.6	2.8	74.0	75.0	1.0	0.49	0.49	0.00
5.0	5.1	0.1	25.8	28.5	2.7	75.4	75.8	0.4	0.46	0.46	0.00
5.1	5.1	0.0	25.9	28.5	2.6	76.3	75.8	0.5	0.47	0.46	0.01
5.7	5.6	0.1	19.0	21.0	2.0	79.0	78.9	0.1	0.48	0.48	0.00
3.8	3.9	0.1	22.0	24.4	2.4	82.0	82.6	0.6	0.39	0.40	0.01
5.1	5.1	0.0	25.6	28.5	2.9	75.0	75.8	0.8	0.42	0.46	0.04
6.1	6.0	0.1	28.0	31.5	3.5	70.7	70.0	0.7	0.52	0.52	0.00
4.1	4.1	0.0	29.5	32.6	3.1	79.7	78.7	1.0	0.42	0.43	0.01
5.9	5.9	0.0	28.7	30.6	1.9	71.9	72.4	0.5	0.50	0.51	0.01
4.4	4.5	0.1	29.1	33.5	4.4	77.1	77.2	0.1	0.43	0.43	0.00
5.1	5.1	0.0	24.8	28.5	3.7	75.7	75.8	0.1	0.49	0.46	0.03
5.1	5.1	0.0	25.3	28.5	3.2	76.6	75.8	0.8	0.47	0.46	0.01
5.2	5.2	0.0	25.6	29.2	3.6	74.1	74.4	0.3	0.44	0.45	0.01
4.9	4.9	0.0	20.3	23.7	3.4	79.6	78.7	0.9	0.44	0.45	0.01
5.2	5.2	0.0	30.1	32.3	2.2	73.2	74.3	1.1	0.48	0.48	0.00
5.7	5.9	0.2	24.3	26.8	2.5	74.1	73.4	0.7	0.50	0.50	0.00
4.9	4.9	0.0	26.0	28.0	2.0	77.1	77.1	0.0	0.45	0.44	0.01
4.4	4.2	0.2	26.5	29.6	3.1	77.9	78.8	0.9	0.42	0.42	0.00

physicochemical properties of powders. The optimization procedure revealed that the optimum powder, which have a minimum hygroscopicity and maximum WSI, could be produced by these conditions: IT = 134 °C, MDC = 60%, and AR = 30%. The experiment at this conditions was conducted and powder with the following properties was obtained: MC: 4.3 ± 0.1%, hygroscopicity: 20.3 ± 0.4%, WSI: 81.5 ± 0.3%, bulk density: 0.39 ± 0.02 g/cm^3.

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