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Optimization of Spray Drying Conditions for Production of Ice Cream Mix Powder Flavored With Black Mulberry Juice

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ABSTRACT

The aim of this work was to optimize the spray drying conditions for the production of ice cream mix powder. A lab-scale spray dryer was employed for the spray drying process, the mix of salep and k-carrageenan was used as stabilizer and black mulberry juice added to ice creams as a natural flavor. Response Surface Methodology (RSM) was performed to examine the influence of inlet air temperature (120, 140, and 160°C), feed flow rate (5, 10, and 15%) and black mulberry concentration (15, 30, and 45%) on drying yield and total anthocyanin content of powders, overrun and melting rate of ice creams prepared from the reconstituted powders. Scanning electron microscope was used for monitoring the structure of the powders. The following optimum process conditions were determined: inlet air temperature of 138 °C, feed flow rate of 8% and juice concentration of 35%. These parameters led to the process yield, total anthocyanin content, overrun and melting rate of 76.14%, 54.11 mg L^{-1}, 74.50%, and 1.52 g min^{-1}, respectively.

Keywords: Natural flavor, Response surface methodology, Salep, SEM.

INTRODUCTION

Ice cream is a frozen dessert made from dairy product and is one of the most popular leisure foods around the world. The quality of ice cream is valued mostly by its flavor. Increasing preference of consumers towards healthier and more natural food products has increased the use of functional ingredients in the dairy industry. Recently, several functional ice cream formulations with natural antioxidants and phenolic compounds such as gooseberry [9], pomegranate peel phenolics [6], grape wine lees [14], and kiwi [27] have been successfully produced. One of the benefits of adding fruit juice to ice cream is the reduction of using commercially available flavoring and coloring agents [27]. Black mulberry (*Morus nigra*) is a popular edible fruit, which originates from Iran. *M. nigra* fruit is a good source of several phytonutrients and contains high amounts of total phenolics, flavonoids, and high antioxidant activity. These compounds play a potentially beneficial role in human health by reducing risks of cancer, cardiovascular disease, and other pathologies [10].

During storage of ice creams, depending on storage time and temperature, some textural defects can occur such as coarse or icy texture due to temperature fluctuation, recrystallization and increased storage time; sandy texture due to lactose crystallization; and shrinkage due to ambient pressure changes [11]. One of the most frequently

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used technique for increasing the shelf life of dairy products is spray drying. It is a well-established technique to turn dairy products such as cheese [8], yoghurt [17], skim milk [1, 16], cream and whole milk [5, 16], whey protein concentrates [16, 22], blend of juice and skim milk [24], yoghurt-ice cream [18], and ice cream [30, 31] into solid dry powder form. According to previous researchers, the shelf life of ice cream powders obtained by spray drying was longer than dry blended ice cream mixes, which refers to microencapsulation of fat by protein and lactose matrix during spray drying process. The presence of free fat on the powder surface is readily susceptible to oxidation and development of rancidity and it decreases wettability and dispersibility [16].

Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques that have been successfully used for developing, improving, and optimizing processes. RSM is an experimental design used to determine the relationship between a number of factors (independent variables) and one or more responses (dependent variables) [7, 20]. Most recently, RSM has been used in optimization of spray-drying conditions to reach the desired quality of products [1, 29]. The objective of the present study was to investigate how variation in the manufacturing parameters such as feed flow rate, inlet air temperature, and juice concentration affected the quality of ice cream powders (drying yield and total anthocyanin content) and the properties of ice creams made from the reconstituted powders (overrun and melting rate) using response surface methodology. Microstructure of spray-dried emulsions was also analyzed.

MATERIALS AND METHODS

Sample Preparation

Ice cream mixes were prepared by adjusting fat ratio of pasteurized milk to 6% with cream addition and other ingredients were in the following order: 5% 20DE maltodextrin (Tongaat Hulett starch, South Africa), 0.8% salep (provided locally, Kordestan, Iran) and 0.1% *k*-carrageenan (Sigma Co., St. Louis, MO), 10% skim milk powder (provided locally, Karaj, Iran), 10% sugar and black mulberry juice at three concentrations of 15, 30, and 45%. Total solid of ice cream mixes containing 15, 30, and 45% black mulberry juice was 35.45, 38.12, and 40.58%, respectively. In this study, the mix of salep and *k*-carrageenan was used as stabilizer. Salep is one of the stabilizers obtained by milling dried tubers of particular wild terrestrial orchids and used as an essential ingredient for the production of traditional ice cream in Iran. In addition to stabilizing properties, salep has health benefits. It primarily contains glucomannan (16-55%) and starch in its structure in addition to water and mineral matters [2].

Black mulberry juice was heated at 55°C for 1 minute, and then mixed with milk. The mix of juice and milk was sealed, covered with tin foil, and heated to 30–40°C in a water bath and then other ingredients such as sugar, skim milk powder, maltodextrin, salep and carrageenan were added to it with gentle stirring. The mix was then batch pasteurized at 76°C for 30 minutes using a water bath and homogenized at 2,000/500 psi (Avestin EmulsiFlex C3, Ottawa, Canada). A Büchi mini spray dryer (Model B-191, Büchi Laboratoriums-Technik, Flawil, Switzerland) was used and the effects of inlet air temperature (120, 140, and 160°C) and feed flow rate (5, 10 and 15%) on powder properties were studied. In all experiments, the aspirator rate, the feed temperature, the compressed air flow rate, and the atomizer pressure were kept at 50% or 925 N m^{-2}, room temperature or 20ºC, 800 L h^{-1}, and 4.5 bar, respectively. The highest level of compressed air flow rate was chosen because of its positive effect on drying yield. Yield is the amount of solid material obtained during spray drying divided by the total amount of solid

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ingredients delivered to the chamber of the spray dryer.

Powders were reconstituted to their original solids content, pasteurized at 76°C using a water bath, cooled to 4°C and remained at constant temperature for 24 hours to be aged. The aged mixes were then frozen and whipped in the household ice cream maker (Clatronic ice cream maker, Model ICM 3225, Kempen, Germany) at low speed for 30 minutes. The ice cream was collected at an exit temperature of −5.5°C and stored at −18°C throughout analyses.

Analytical Methods

Drying Yield

Process yield was determined by dividing the total solids content in the resulting powder by the total amount of solid mass in the feed mixture and expressed as a percentage.

Total Anthocyanin Content (TAC)

This method involves extraction of anthocyanin from ice cream samples, expression of their color at low pH and quantification based on their absorbance in the visible region of the light spectrum. Cyanidin-3-glucoside (cyd-3-glu) is the major anthocyanin in black mulberry juice, but it is not the only anthocyanin and the results are expressed in cyanidin-equivalents for comparative purposes only. Two mL of the melted ice cream sample was mixed with two mL methanol (containing 100 g kg^{-1} HCl) and then centrifuged at 3,000×g for 10 minutes. The supernatant (1 mL) was added to 9 mL 0.1M HCl and the absorbance was measured at 520 nm [14]. The total anthocyanin content of the samples (cyd-3-glu equivalents, mg L^{-1}) was calculated by the following equation:

$$\text{TAC}=\frac{\text{A}\times\text{MW}\times\text{DF}\times 1000}{\text{MA}\times L} \quad (1)$$

Where, *A*= Absorbance; *MW* (Molecular Weight)= 449.2 g mol^{-1} for cyd-3-glu; *DF* (Dilution Factor)= 10; *MA* (Molar Absorptivity)= 26,900 L mol^{-1} cm^{-1} for cyd-3-glu; *L*= Path length in cm, and 1,000= Factor for conversion from g to mg.

Determination of Overrun

A known volume of ice cream mix and frozen ice cream was weighed and overrun was calculated according to the Equation (2):

$$\text{Overrun }(\%)=\frac{x-y}{z}\times 100 \quad (2)$$

Where x =Weight of a known volume of ice cream mix, y =Weight of known volume of ice cream and z =Weight of a known volume of ice cream mix

Melting Characteristics

Melting behavior was assessed by withdrawing 150 g of ice cream from the freezer at −20°C and putting it on a 0.2 cm wire mesh screen and allowing melting at ambient temperature (22±1°C). The weight of the material passed through the screen was recorded at 5 minutes time intervals. The melting rate was determined as the slope of the graphs of the dripped portion as function of the time, and expressed in g min^{-1}.

Scanning Electron Microscopy (SEM)

The microstructure of the ice cream powders was examined using a scanning electron microscope (XL-30, Philips, Amsterdam, The Netherlands). To obtain SEM images, a small amount of powders was taken from well mixed powder samples and coated with very thin layer of gold under high vacuum conditions to provide a reflective surface for the electron beam. Gold coating was carried out in a sputter coater BIO-RAD E-5200 (Bio-Rad Laboratories Ltd., London, UK) under a low

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vacuum in the presence of inert argon gas. The gold-coated samples were subsequently viewed under the microscope.

Experimental Design

RSM was performed to study the effects of spray drying parameters including inlet air temperature (X_1), feed flow rate (X_2), and black mulberry juice concentration (X_3) to maximize the drying yield, total anthocyanin content, and overrun as well as to minimize the melting rate of ice cream samples. Twenty experimental runs including 6 replicates at the center point were generated based on the face-centered experimental design, with three independent variables at three levels for each variable (Table 1).

The relationship of the independent variables and the response was calculated by the second-order polynomial Equation (3). Four models of the following form were developed to relate the four responses (Y) to the three process variables (X):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} X_i X_j \quad (3)$$

Where, Y is the predicted response; β_0 is a constant; β_i is the linear coefficient; β_{ii} is the squared coefficient; β_{ij} is the cross product coefficient; and k is the number of factors [3].

The Analysis of Variance (ANOVA), determination of the regression coefficients and the generation of three-dimensional graphs were carried out using the Design Expert software. Significance of the model and model terms were judged by determining the probability level that the F-statistic calculated from the data was less than 5%. The model goodness was checked by R^2, adjusted R^2, Adequate precision, PRESS and Coefficient of Variation (CV) [20]. Statistical analysis to examine significant difference between experimental and predicted values was performed by one sample T-test at the 95% confidence level using the SPSS version 16.0 for Windows

Table 1. Independent and response variables for the 20 trials of the experimental design.

Run no	Independent variables			Responses			
	Inlet air temperature (°C)	Feed flow rate (%)	Black mulberry concentration (%)	Drying yield (%)	Total anthocyanin content (mg L^{-1})	Overrun (%)	Melting rate (g min^{-1})
1	140 (0)	10 (0)	30 (0)	75.45	58.05	71.26	1.75
2	140 (0)	10 (0)	15 (-1)	78.33	47.12	76.31	1.94
3	140 (0)	10 (0)	30 (0)	74.12	59.52	72.85	1.73
4	160 (+1)	5 (-1)	45 (+1)	66.29	54.46	63.27	1.45
5	160 (+1)	15 (+1)	45 (+1)	55.51	70.85	53.81	1.78
6	160 (+1)	5 (-1)	15 (-1)	75.55	32.09	77.09	2.08
7	140 (0)	10 (0)	45 (+1)	68.34	69.64	62.24	1.38
8	120 (-1)	15 (+1)	15 (-1)	57.70	59.27	75.33	2.05
9	120 (-1)	5 (-1)	45 (+1)	62.52	60.07	70.61	1.02
10	160 (+1)	15 (+1)	15 (-1)	61.47	50.74	66.27	2.30
11	140 (0)	10 (0)	30 (0)	72.38	60.91	73.04	1.72
12	140 (0)	5 (-1)	30 (0)	78.73	47.52	75.29	1.65
13	140 (0)	10 (0)	30 (0)	76.32	57.81	70.78	1.69
14	120 (-1)	10 (0)	30 (0)	62.47	59.12	74.43	1.49
15	120 (-1)	15 (+1)	45 (+1)	47.84	78.38	60.52	1.50
16	140 (0)	10 (0)	30 (0)	71.16	61.09	72.52	1.72
17	120 (-1)	5 (-1)	15 (-1)	70.22	39.41	85.45	1.66
18	140 (0)	15 (+1)	30 (0)	69.35	70.21	66.58	2.02
19	140 (0)	10 (0)	30 (0)	73.17	60.45	73.29	1.75
20	160 (+1)	10 (0)	30 (0)	66.25	53.21	66.32	1.88

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(SPSS Inc., Chicago, IL, USA).

RESULTS AND DISCUSSION

Fitting Models

The results of ANOVA indicated that the contribution of the quadratic models was very significant (P< 0.0001) (Table 2). The mathematical models generated from the experimental data using Design-Expert software, respectively, for the drying yield (Y_1), total anthocyanin content (Y_2), overrun (Y_3), and melting rate (Y_4) are expressed by the following:

$$Y_1= 74.07+2.43\ X_1 - 6.14\ X_2 - 4.28\ X_3 - 10.16\ X_1^2 \quad (4)$$

$$Y_2= 59.42 - 3.49\ X_1 +9.59\ X_2 +10.48\ X_3 - 2.92\ X_1^2 \quad (5)$$

$$Y_3= 71.85 - 3.96\ X_1 - 4.92\ X_2 - 7\ X_3 - 1.91\ X_3^2 \quad (6)$$

$$Y_4= 1.73 + 0.18\ X_1 +0.18\ X_2 - 0.29\ X_3 - 0.04\ X_1^2 +0.11\ X_2^2 - 0.065\ X_3^2 - 0.04\ X_1X_2+0.025\ X_2X_3 \quad (7)$$

The high R^2 (0.976–0.998) and adjusted R^2 (0.954–0.995) values indicate that a high proportion of variability in the response models can be explained successfully by the models [16]. The *CV*, which indicates the relative dispersion of the experimental points from the predictions of the Second Order Polynomial (SOP) models, were found to be 2.61, 2.66, 1.66, and 1.17% for process yield, total anthocyanin content, overrun and melting rate, respectively. Generally, *CV* value should not be greater than 10%. The suitable PRESS values also suggest the adequacy of the fitted quadratic models for predictive applications. The adequate precision measures the signal-to-noise ratio with a ratio greater than 4 being desirable. The high adequate precision values (24.546–94.503) indicated that the fitted models could be used to navigate the design space (Table 2). ANOVA also showed that the lack of fit was not significant for any response models at a 5% significance level and that model adequacies were appropriate. The RMSE-prediction for the studied response variables ranged from 0.014 to 1.254. This factor was used to assess the model performance in forecasting data.

It is important to check the adequacy of the fitted models in order to ascertain their validity. Figure 1a shows that the normal plot of residuals for responses was normally distributed, as they lie approximately on a straight line and shows no deviation of the variance. The results of all these plots (Figure 1-a) indicated that developed models are adequate to describe the responses. The Cook's distance values are in the determined range (Figure 1-b); there is strong evidence for influential observations in these data.

Drying Yield

Table 2 shows that the linear effects of independent variables were significant on the drying yield. Quadratic effect of inlet air temperature was also significant at *P*< 0.0001 in the experimental domain studied. The mutual interaction between all the independent variables was found to be insignificant. Figure 2-a shows the influence of inlet air temperature, feed flow rate, and black mulberry concentration on the spray drying process yield. Increase in inlet air temperature from 120 to 140°C induced an increase in yield, which can be attributed to the greater efficiency of heat and mass transfer processes and decreasing the probability of hitting the inadequate drying particles to the drying chamber wall. Similar results were reported by Tonon *et al*. [29] and Goula and Adamopoulos [12]. But, increasing inlet air temperature to 160°C showed a negative effect on drying yield especially for samples containing higher amount of black mulberry juice.

Papadakis *et al*. [21] also reported that product stickiness in spray drying is increased if the surface temperature of the particles exceeds glass transition temperatures (Tg)+20°C, because at temperatures equal to Tg+10°C, the product

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Table 2. ANOVA results for the linear, quadratic, and interaction terms of each variable and coefficients for the predicted model.

Source	*df*	Drying yield (%)			Total anthocyanin content (mg L^{-1})			Overrun (%)			Melting rate (g min^{-1})		
		Coef	Sum of squares	*P*-Value	Coef	Sum of squares	P-Value	Coef.	Sum of squares	*P*-Value	Coef	Sum of squares	*P*-Value
Model	9	74.07	1249.60	< 0.0001**	59.42	2205.49	< 0.0001**	71.85	925.58	< 0.0001**	1. 73	1.53	< 0.0001**
X_1	1	2.43	59.15	0.0015**	-3.49	121.80	< 0.0001**	-3.96	156.66	< 0.0001**	0.18	0.31	< 0.0001**
X_2	1	-6.14	377.49	< 0.0001**	9.59	919.68	< 0.0001**	-4.92	242.06	< 0.0001**	0.18	0.32	< 0.0001**
X_3	1	-4.28	182.93	< 0.0001**	10.48	1097.68	< 0.0001**	-7.00	490.00	< 0.0001**	-0.29	0.84	< 0.0001**
X_1^2	1	-10.16	283.72	< 0.0001**	-2.92	23.43	0.0078*	-	1.80	0.2160^{ns}	-0.040	0.0035	0.0060**
X_2^2	1	-	0.63	0.6649^{ns}	-	0.13	0.8088^{ns}	-	0.17	0.6922^{ns}	0.11	0.033	< 0.0001**
X_3^2	1	-	3.84	0.2948^{ns}	-	1.36	0.4425^{ns}	-1.91	10.03	0.0110*	-0.065	0.012	0.0002**
X_1X_2	1	-	0.68	0.6508^{ns}	-	1.22	0.4657^{ns}	-	0.12	0.9811^{ns}	-0.040	0.0013	0.0002**
X_1X_3	1	-	0.68	0.6508^{ns}	-	0.92	0.5262^{ns}	-	1.42	0.2686^{ns}	-	0.0012	0.4813^{ns}
X_2X_3	1	-	0.16	0.8248^{ns}	-	1.81	0.3776^{ns}	-	0.24	0.6394^{ns}	0.025	0.0015	0.0044**
Residual	10		31.45			21.29			10.35			0.0037	
Lack of Fit	5		12.89	0.6503^{ns}		11.03	0.4696^{ns}		5.08	0.5160^{ns}		0.0012	0.7830^{ns}
Pure error	5		18.55			10.26			5.27			0.0025	
Total	19		1281.04			2226.78			935.92			1.53	
R^2		0.976			0.990			0.989			0.998		
Adj R^2		0.954			0.982			0.979			0.995		
PRESS		175.47			96.13			35.17			0.013		
CV (%)		2.61			2.54			1.45			1.12		
Adequate Precision		24.546			45.665			44.154			94.503		
RMSE-prediction		1.254			1.032			0.719			0.014		

**: Highly significant (P< 0.01); * Significant (P< 0.05) and ^{ns}: For not significant.

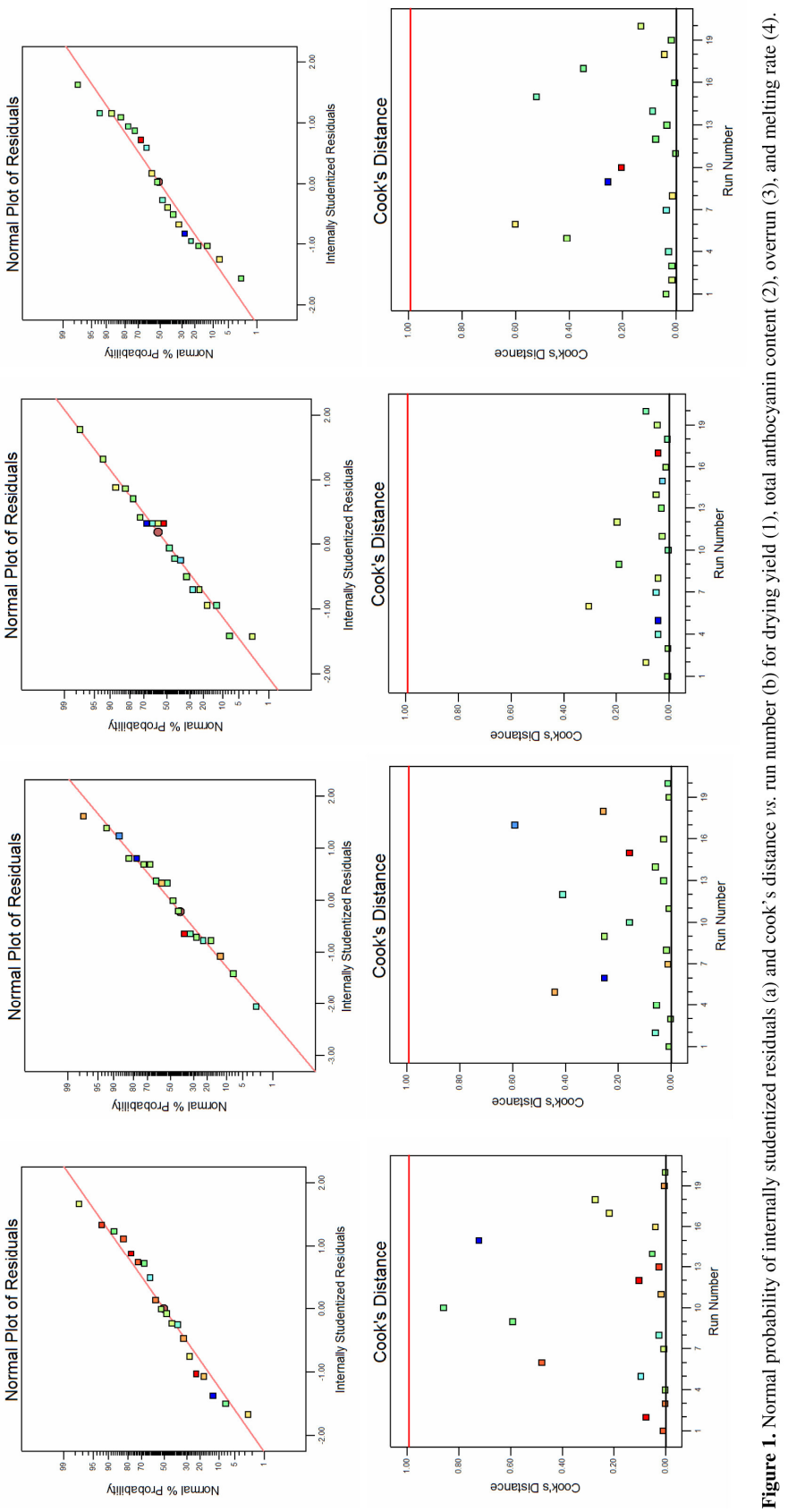

Figure 1. Normal probability of internally studentized residuals (a) and cook's distance *vs.* run number (b) for drying yield (1), total anthocyanin content (2), overrun (3), and melting rate (4).

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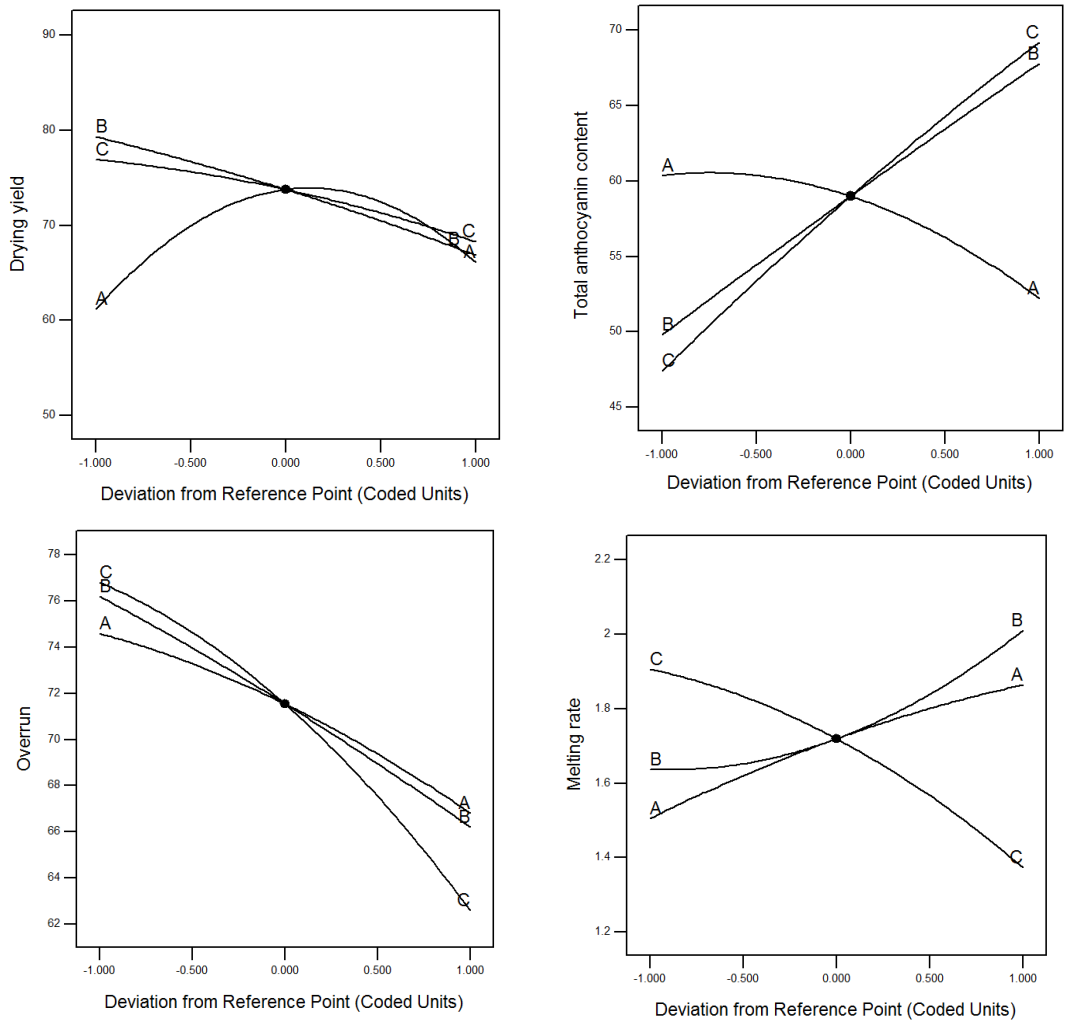

Figure 2. Perturbation graph showing the effect of independent variables on the yield (a), total anthocyanin content (b), Overrun (c) and melting rate (d) (Actual factors: A: Inlet air temperature= 140°C, B: Feed flow rate= 10%, C: Black mulberry concentration= 30%).

begins to show adhesion, and at Tg+20°C it shows stickiness. Increasing feed flow rate led to lower process yield because higher feed rate reduced the contact time between feed and the drying air and caused a decrease in the rate of heat and mass transfer. This is in agreement with the results published by Toneli *et al*. [28] and Tonon *et al*. [29] working with spray drying of tomato pulp and açai, respectively. Açai (Euterpe oleraceae Mart.) is a typical fruit from Amazonia. It has been recognized for its functional properties for use in nutraceutical products, due to its high antioxidant activity, which is related to its high anthocyanin and phenolic content [29].

The process yield decreased by increasing of juice concentration in ice cream mixes (Figure 2a). According to Bhandari *et al*. [4] the sticky behavior of juices in spray dryer is attributed to a high concentration of low molecular weight sugars and organic acids, which have low Tg, being rubbery and thermoplastic at the temperatures of the chamber. Rao and Gupta [24] also reported that, as the proportion of juice increased in the blends of orange juice and skim milk, the drying yield decreased due to the high viscosity of the blends. They concluded high viscosity had a negative effect on the spray drying process, such as clogging of the nozzle [24].

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Total Anthocyanin Content

The results indicated that the linear effects of all independent variables were significant (P< 0.0001) on the total anthocyanin content of ice cream powders containing black mulberry juice (Table 2). Furthermore, Quadratic effect of inlet air temperature was significant. The mutual interaction between independent variables was found to be insignificant (Table 2). Powders anthocyanin content varied from 33.09 to 84.38 mg L^{-1}. Figure 2-b shows the influence of inlet air temperature, feed flow rate, and black mulberry concentration on the TAC. Anthocyanin degradation of ice cream powders increased with an increase in inlet air temperature due to the high sensitivity of these pigments. Previous researchers also verified that an increase in inlet drying temperature resulted in a greater loss of phytochemicals such as lycopene of tomato powders, *β*-carotene of water melon, and carotenoid of Gac fruit powder [13, 15, 23].

Besides inlet air temperature, the outlet air temperature has also an important effect on the powder properties. By increasing feed flow rate, the outlet temperature is decreased and, consequently, the anthocyanin degradation of ice cream powders is decreased.

The positive effect of adding black mulberry juice in the range of 15-45% on the total anthocyanin of ice cream powders is shown in Figure 2b. Similar results were reported by Cam *et al*. [6], Hwang *et al.* [14], Sun-Waterhouse *et al.* [27], and Sagdic *et al.* [25] working with ice cream samples which were fortified by pomegranate peel phenolic, grape wine lee, juice of kiwifruit, and phenolic substances, respectively.

Overrun

Overrun is due to incorporation of air during the freezing process. During the freezing step of ice cream production, the fat globules are mechanically damaged by the shear forces and the ice crystallization process, which leads to agglomeration and partial coalescence of the fat globules. Many quality properties of ice cream are related to partially coalesced fat, like slow meltdown, good shape retention, resistance to shrinking, strong whipping properties, and high overrun [26]. The linear effects of three independent variables were significant on the overrun values (P< 0.0001). In quadratic terms, black mulberry juice concentration had significant effects on this factor (Table 2). The mutual interaction between variables was found to be insignificant. Higher inlet air temperature and feed flow rate caused a decrease in overrun of ice cream samples, which may be related to the low content of free fat or lower degree of partial coalescence during dynamic freezing (Figure 2-c). According to previous researches, increasing inlet air temperatures led to a smaller free fat content in the powder. It is due to the formation of a fast dry crust during drying, which would prevent the disruption and coalescence of fat droplets [32]. On the other hand, it is concluded by other researchers that the free fat increased linearly with air outlet temperature due to the formation of cracks on powder particle surface, which verify the decrease of overrun by increasing feed flow rate [32]. Black mulberry juice decreased the overrun of ice cream (Figure 2-c). Since the viscosity of ice cream mixes increased with juice, probably less air was incorporated in the ice cream mix during freezing, which resulted in lower overrun. The decrease of overrun values with fruit juice was in agreement with the results indicated in the literature [9, 14].

Melting Rate

Table 2 shows that the linear and quadratic effects of all independent variables were significant on the melting rate. The mutual interaction between inlet air temperature and feed flow rate was also significant. Ice cream powders that were produced at higher inlet air temperature and feed flow rate showed faster melting rates (Figure 2-d). It may be related to lower levels of

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destabilized fat. A greater extent of destabilized fat increased the resistance to flow of the serum phase as ice melted, which led to slower melt down [19]. Increasing black mulberry juice concentration slowed down the melting rate or provoked longer melting time (Figure 2-d). It might be originated from the components of black mulberry juice such as polysaccharides having the ability to absorb water and reduce the free movement of water molecules.

Optimization and Model Verification

The second order polynomial models obtained in this study were utilized for each response in order to determine optimum conditions. Regarding drying yield, total anthocyanin content, overrun, and melting rate the following optimum conditions were established for producing spray-dried ice cream: inlet air temperature of 138°C, feed flow rate of 8% and black mulberry concentration of 35%. At this optimum point drying yield, total anthocyanin content, overrun, and melting rate were determined to be 76.14%, 54.11 mg L^{-1}, 74.50%, and 1.52 g min^{-1}, respectively. The complex desirability value for the optimum solution was 0.89. After determination of optimum conditions for ice cream powder production, experiments based on the optimum spray-drying parameters were performed and repeated three times. The results of one sample *T*-test to examine significant difference between experimental and predicted values are shown in Table 3. The column labeled "Sig. (2-tailed)" gives the two-tailed *P*-value associated with the test. As reported in Table 3, all the *P*-values are larger than α level of 0.05, which implies that there is not a statistically reliable difference between predicted and measured values of the four responses.

Microstructure

In encapsulation, the absence of cracks, pores and breakage is critically important to wall functionality in limiting core deterioration and/or losses during storage. Microcapsules should be spherical and non-agglomerating, which indicated satisfactory flow properties. Smooth surface of microcapsules is preferred to shriveled and dented surface. During spray drying, rapid cooling generated dents due to the wall material shrinkage, especially at high drying rates [33].

The morphology of the microcapsules which were spray dried at different inlet air temperature and feed flow rates and contained various black mulberry concentrations is shown in Figure 3. Structural analysis revealed there were many more wrinkles in the surface of particles, which were produced at lower inlet air temperature (Figure 3-a). Decreasing feed flow rate and drying temperature had a negative effect on particle size, which is related to slower drying rates and shrinkage of particles. According to Tonon *et al*. [29], at high inlet air temperature, because of faster drying rates, the skin becomes dry and hard quickly, so that the hollow particle cannot deflate when vapor condenses within the vacuole as the particle moves into cooler regions of the dryer. However, when the drying temperature is lower, the skin remains moist and supple for longer, so that

Table 3. Comparison between the measured and predicted results.

Responses	Predicted	Observed± SD	Standard error mean	Mean differences	Sig (2-tailed)
Drying yield	76.14	75.35 ± 0.70	0.41	0.79	0.191^{ns}
Total anthocyanin content	54.11	53.69 ± 0.42	0.24	0.41	0.231^{ns}
Overrun	74.50	73.19 ±0.74	0.43	1.31	0.092^{ns}
Melting rate	1.52	1.46 ±0.05	0.03	0.06	0.188^{ns}

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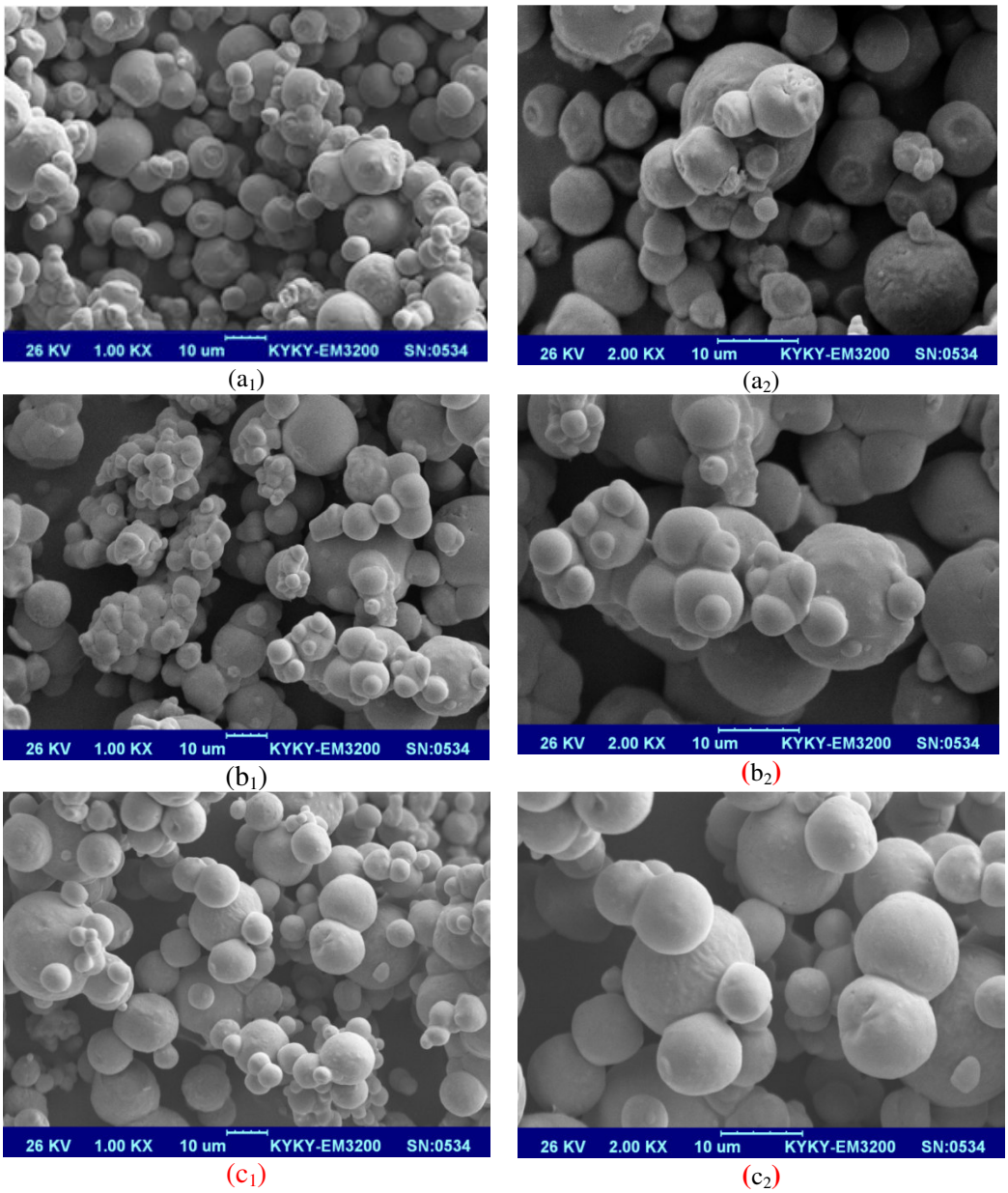

Figure 3. Micrographs of ice cream powders produced at: (a) The lowest inlet air temperature (feed rate of 10% and juice concentration of 30%); (b) The highest juice concentration (temperature of 140°C, feed rate of 10%), and (c) Optimized conditions. (1) 1,000X, and (2) 2,000X.

the hollow particle can deflate and shrivel as it cools.

More adherences of particles were observed in wall systems of particles, which contained high concentration of black mulberry juice (Figure 3-b). It can be due to amorphous surfaces of particles. The particulate structure of the product generated under the optimum spray-drying conditions had well-separated microparticles with hardly any surface cracks in the wall systems (Figure 3-c). The spherical and smooth surface shows the suitability of the spray drying conditions for encapsulation.

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CONCLUSIONS

In this study, the effects of inlet air temperature, feed flow rate, and juice concentration on ice cream powder properties such as drying yield and total anthocyanin content, and reconstituted mix properties such as overrun and melting rate were studied. The results obtained in the present work indicate that, due to black mulberry's nutritive value, high anthocyanin content, attractive flavor and color, it may be used as a suitable source of natural additive to produce value-added ice cream. The optimization results indicated that an inlet air temperature of 138°C, feed flow rate of 8%, and black mulberry concentration of 35% will produce samples with the best properties. At these optimum conditions drying yield, total anthocyanin content, overrun, and melting rate were found to be 76.14%, 54.11 mg L^{-1}, 74.50% and 1.52 g min^{-1}, respectively. The predictive capability of RSM was satisfactory and T-test failed to reveal a statistically significant difference between the predicted and measured values. In addition, the microstructure of powders produced at the optimized conditions verified good encapsulation with smooth and spherical surface.

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بهینه سازی شرایط خشک کردن پاششی جهت تولید پودر بستنی حاوی آب شاتوت

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چکیده

هدف از این مطالعه بهینه‌یابی شرایط خشک کردن پاششی به منظور تولید پودر بستنی می‌باشد. جهت انجام فرایند از یک خشک‌کن پاششی آزمایشگاهی استفاده شد. از ترکیب ثعلب و کاپا کاراگینان به عنوان پایدارکننده و از آب شاتوت به عنوان طعم دهنده طبیعی استفاده شد. به منظور بررسی تأثیر دمای هوای ورودی (۱۲۰، ۱۴۰ و ۱۶۰°C)، سرعت جریان ماده ورودی (۵، ۱۰ و ۱۵٪) و میزان آب شاتوت (۱۵، ۳۰ و ۴۵٪) بر راندمان خشک کردن و میزان آنتوسیانین کل پودرهای تولیدی، میزان افزایش حجم و سرعت ذوب بستنی‌های حاصل از بازسازی پودرها، از روش سطح پاسخ و جهت بررسی ریزساختار پودرها از میکروسکوپ الکترونی استفاده گردید. شرایط بهینه بدست آمده عبارتند از: دمای هوای ورودی°C ۱۳۸، سرعت جریان ماده ورودی ۸٪ و میزان آب شاتوت ۳۵٪. در این شرایط راندمان خشک کردن، میزان آنتوسیانین کل، افزایش حجم و سرعت ذوب نمونه تولیدی به ترتیب ۷۶/۱۴٪، ۵۴/۱۱ mg/L، ۷۴/۵۰٪ و ۱/۵۲ g/min بدست آمد.

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