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Modeling of physicochemical and functional parameters of pumpkin *(Cucurbita pepo)* powder using response surface methodology

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Abstract

Pumpkin (*Curcurbita pepo)* is a rich source of carotene, vitamins, minerals, pectin and dietary fiber however it is perishable in nature. Spray drying was used for the preparation of pumpkin powder to enhance the shelf life and process parameters were modelled. Response surface methodology was used to investigate the effect of maltodextrin concentrations (10-30%), inlet air temperature (140-210˚C) and feed flow rate (8-12 rpm) at constant 2400 rpm blower speed during spray drying on different physico-chemical, functional and anti oxidants properties to obtain optimum quality of spray dried pumpkin juice powder. The optimized conditions for dehydration were found as maltodextrin concentration of 15%, inlet air temperature of 177˚C and feed flow rate of 10 rpm. Experimental values of the responses under the optimized conditions were as water activity: 0.14, L^* value: 96.54, a^* value: 4.91, b^* value: 21.71, water solubility index: 82.58%, water absorption index: 10.09 g/g, hygroscopicity: 13.25 g/100g, bulk density: 0.29 g/ml, process yield: 64.53%, beta carotene: 3.06 mg/100g, moisture content: 3.25% and DPPH (2,2diphenyl 1- pecrylhydrazyl) free radical scavenging activity: 40.95%.

Introduction

Pumpkin *(Cucurbita pepo)* is a rich source of carotene, vitamins, minerals, pectin and dietary fibre. Pumpkin is a valuable source of carotenoids and ascorbic acid, which have major roles as the functional constituents and antioxidants. In pumpkins, carotenoid is a natural plant pigment, which is responsible to render orange colour. Tee and Lim (1991) investigated three major carotenoids in pumpkin *(Cucurbita maxima)*. The carotenoids were lutein (0.94 μg/g), α-carotene (0.756 μg/g), and ß-carotene (0.578μg/g). Murkovic *et al*. (2002) also reported beta carotene (0.06 -7.4 mg / 100 g), α-carotene (0-7.5 mg / 100 g) and lutein (0.17 mg / 100g) in three species of pumpkins (*Cucurbita pepo, Cucurbita maxima* and *Cucurbita moschata*) respectively. Recent research indicates that beta-carotene rich diet may reduce the risk of developing certain types of cancer and offers protection against heart disease. Hence, supplementation of pumpkin flour may improve the nutritional quality of the food products.

Drying or dehydration is being used to enhance the shelf-life of perishable food for further use in other value added products (Kumar *et al*., 2011). The spray drying is a continuous operation in which almost any pumpable liquid can be converted into a free flowing powder. Spray drying is an essential unit operation for the manufacture of many products with specific properties. The physical and chemical properties of powders produced by spray drying technique depend upon process variables, like characteristics of the liquid feed (viscosity, particle size, flow rate) and of the drying air (temperature, pressure), as well as the type of atomizer and also the carrier agents (polymer or gum) used. Therefore, it is mandatory to monitor and formulate a crucial state of process variables to optimize the drying process and to obtain finished products with better sensory characteristics, nutritional quality and higher yield. The optimization of the spray-drying process involves the evaluation of parameters concerning both the spray-dryer and the feed (Wendel and Celik, 1997). Response surface methodology is a popular tool to optimize the process parameters and has been successfully used for the optimization of hot air drying of infused apple cubes, recovery of dried dark fig, β-galactosidase production and fenugreek enriched extruded product by Diamante and Yamaguchi (2012), Bachir-bey *et al.* (2014), Al-jazairi *et al*. (2015) and Wani and Kumar (2016) respectively. Spray dried fruit juice powder

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have some inherent problems such as stickiness, hygroscopicity and solubility due to presence of low molecular weight sugars and acids having low glass transition temperature (Bhandari *et al.*, 1993). Thus powder can stick on the wall of chamber during drying process, which leads to lower yield and certain operational problems. These problems can be resolved by addition of some carrier agents, like polymers and gums. Along with reduction in substantial hygroscopicity of powder, polymers and gums can also be used for microencapsulation imparting protection to sensitive food components against unfavorable ambient conditions.

Pumpkin can be processed into flour, which has longer shelf-life. The flour can be useful in different food products because of its highly desirable flavour, sweetness, functional constituents and deep yellow-orange colour. It has been used to supplement cereal flours in bakery products, sauces, instant noodle, soups, spice as well as a natural colouring agent in pasta and flour mixes (Saeleaw and Schleining, 2011). Effect of particle size and temperature on rheological, thermal and structural properties of pumpkin flour dispersion was studied by Ahmed *et al*. (2014). Effect of enzymatic liquefaction, concentration of career agent and spray drying inlet temperature on pumpkin powder characteristics was also investigated by Shavakhi *et al*. (2012). But, there is a need to develop technology to achieve pumpkin powder without the application of enzymes because of economy of the process. The response surface methodology is a collection of statistical and mathematical techniques, useful for developing, improving, and optimizing processes (Bas and Boyaci, 2007; Khuri and Muhopadhyay, 2010; Kumar *et al*., 2015). Therefore, the study was under taken to optimize the process parameters (air inlet temperature, feed flow rate and maltodextrin concentration) to prepare pumpkin juice powder on the basis of various responses using Response Surface Methodology (RSM).

Materials and Methods

Materials

Well matured, fully ripened, completely yellow and orange in color pumpkin fruit free from blemishes and mechanical injuries were procured from local market, Longowal, Sangrur, India. Maltodextrin (DE20) (Himedia laboratories Pvt. Ltd., Mumbai, India) was used as carrier agent.

Methods

Following unit operations were involved in the preparation of spray dried pumpkin juice powder.

Extraction of juice

The fruits were washed in cold tap water followed by manual peeling using stainless steel knife. Fruits were cut in small pieces (2 X 3 X 2 mm^3) after removal of seeds. The pieces so prepared, were ground by using mixer grinder with the addition of water in double the quantity, which was set by conducting preliminary experiments (fruit to water ratio:: 1:1, 1:2, 1:3) on the basis of total soluble solid (TSS) and juice yield. The mix was filtered through muslin cloth followed by addition of maltodextrin.

Drying treatments

The juice was dried using spray drying techniques. The range of process parameters viz. maltodextrin concentration (10-30%), inlet air temperature (140-210˚C) and feed flow rate (8-12 rpm) were selected on the basis of available scientific data for different fruit juices. Maltodextrin concentration with range of 10-30% was under taken for the spray drying of gac fruit (Kha, 2010), while inlet air temperature and feed flow rate with range of 140-210˚C and 8-12 rpm respectively were under taken for the spray drying of acai fruit (Tonon *et al*., 2008).

Experimental design

For optimization of spray drying process conditions, the experiments were conducted according to Central Composite Rotatable Design (CCRD), Response Surface Methodology with three variables at five levels each. The independent variables were air inlet temperature, feed flow rate and maltodextrin concentration for spray drying process. The designed experiments are shown in Table 1.

Spray drying

The different proportions of maltodextrin as per CCRD (Table 1) were dissolved in minimum amount of warm water (50°C) and then the solution was mixed with the pumpkin juice. A mixture of 1000 ml of pumpkin juice, consisting of different proportions of maltodextrin as per designed experiments was prepared. Pilot plant Spray-dryer (S.M. Scientech, India) with a co-current air flow was used. The speed of blower was kept 2400 rpm. Distilled water was pumped into the dryer at a constant flow rate (10 rpm = 30 ml/min) to achieve the desired inlet and outlet temperatures. The spray dryer was run at the above conditions for about 10 min before the feed was introduced. The product was collected in a pre-weighed, insulated glass bottle connected at the end of cyclone collector.

Table 1. Experimental design for spray drying of pumpkin juice powder under different designed conditions

Sample No.	M.D. Concentration (%)		Inlet Air Temperature (°C)		Feed Flow Rate (RPM)	
	Coded	Uncoded	Coded	Uncoded	Coded	Uncoded
1	-1	10	-1	140	-1	8
2	1	30	-1	140	-1	8
3	-1	10	1	210	-1	8
4	1	30	1	210	-1	8
5	-1	10	-1	140	1	12
6	1	30	-1	140	1	12
7	-1	10	1	210	1	12
8	1	30	1	210	1	12
9	-1.682	3.18	0	175	0	10
10	1.682	36.82	0	175	0	10
11	0	20	-1.682	116.14 (116*)	0	10
12	0	20	1.682	233.86 (234*)	0	10
13	0	20	0	175	-1.682	6.64 (7*)
14	0	20	0	175	1.682	13.36 (13*)
15	0	20	0	175	0	10
16	0	20	0	175	0	10
17	0	20	0	175	0	10
18	0	20	0	175	0	10
19	0	20	0	175	0	10
20	0	20	0	175	0	10

* targeted experimental value as the actual designed values were difficult to maintain during experiment.

Water activity (a_w)

Water activity meter (Cole-parmer) was used to measure a_w of the spray dried powders.

Colour measurement

Colour measurements of the powder samples were carried out using a Hunter Lab Colour spectrophotometer (I-5 Model, Greath Mackbeth). The instrument was standardized each time with a black and a white tile. The L^* value gives a measure of lightness of the product colour from 100 for perfect white and 0 for black. The redness/greenness and yellowness/blueness are denoted by a^* and b^* values respectively.

Water solubility index (WSI)

The WSI of the pumpkin juice powder was determined using the method described by Anderson *et al.* (1969).

$$Water\ solubility\ index(\%) = \frac{Weight\ of\ dry\ soild\ in\ supernatant}{Weight\ of\ sample} \times 100 \quad (1)$$

Water absorption index (WAI)

WAI of the pumpkin juice powder was determined using the method described by Yamazaki (1953).

$$Water\ absorption\ index(g/g) = \frac{Weight\ of\ residual\ sample}{Weight\ of\ sample} \quad (2)$$

Hygroscopicity

Hygroscopicity of pumpkin juice powder was determined according to the method proposed by Cai and Corke (2000), with some modifications. Samples, powder (approximately 1 g) were placed at 25°C in a container with NaCl saturated solution (75.29%RH). After one week, samples were weighed and hygroscopicity was expressed as g of adsorbed moisture per 100 g dry solids (g/100 g).

Bulk density

Bulk density (g/ml) was determined by gently adding 2 g of pumpkin juice powder into an empty 10 ml graduated cylinder and holding the cylinder on a vortex vibrator for 1 min. The bulk density was determined by estimating the ratio of mass and the volume occupied by the powder in the cylinder.

Process yields

One of the main indices of a spray-dryer performance is the product recovery. Total recovery of each spray dry run was calculated by adding cyclone and sweep recoveries together, which can be estimated by addition of the mass of the powder collected in cyclone collector (cyclone recovery) and the mass of powder obtained following manual sweeping of the wall of spray-dryer glass chamber (sweep recovery). The relationship between total solids content in the resulting powder and total solids content in the feed mixture was expressed as process yield (Tonon *et al.*, 2008).

$$\%\ Process\ yield = \frac{Total\ solid\ content\ in\ resulting\ powder}{Total\ solid\ content\ in\ feed\ mixture} \times 100 \quad (3)$$

Beta carotene

The estimation of the β-carotene was carried out as per the method adopted by Sharma *et al*. (2009) with some modifications. Powder, 5 g was mixed with 10-15 ml acetone. The filtrate was transferred to separating funnel followed by addition of 10 ml of 3% acetone in petroleum ether in the separating funnel. The petroleum ether containing pigments was collected in a glass beaker. The extraction was then repeated until no more colour was extractable. The pigments were transferred into petroleum ether phase by diluting the acetone with water. The adsorbent column of magnesium oxide and supercel, having 10 cm length was added with Na_2SO_4 (1 cm) at the top of the column. The column was continuously washed with the eluent (3% acetone in petroleum ether) and the wash was continued till eluent was colourless. The contents of the flask were transferred to volumetric flask and diluted to volume with the eluent. The intensity of the colour was measured at 452 nm using 3% acetone in petroleum ether as blank and the beta carotene was estimated using the following expression:

$$\mu g\ of\ carotene\ per\ 100\ g = \frac{Concentraion\ of\ carotene\ in\ solution\ from\ std.curve\ (\mu g/ml) \times final\ vol. \times dilution}{Weight\ of\ sample} \times 100 \quad (4)$$

Moisture content

The moisture contents of pumpkin juice powder samples were measured using standard method (AOAC, 1990).

Free radical scavenging activity

Antioxidant activity of the pumpkin juice powder was evaluated by the DPPH (2,2diphenyl 1-pecrylhydrazyl) radical scavenging activity method. DPPH solution (200µg/ml), was prepared by adding 10 mg of DPPH to 50 ml volumetric flask and diluting to volume with 70% (v/v) aqueous methanol. The sample of spray dried pumpkin juice powder was prepared in 2 ml of 70% (v/v) methanol ranging from 0.10-0.50 mg per ml to produce DPPH radical scavenging level ranging approximately 10-85%. All samples and DPPH solution were placed in water bath for 20 min to ensure that all soluble components were dissolved.

An aliquot, 0.5 ml of the sample solution was added to 2 ml of DPPH in a 20 ml test tube. The control sample was prepared by adding 0.5 ml 70% (v/v) methanol to 2 ml of DPPH solution. The sample was vortexed on level 5 for 10-15 sec and held at room temperature (20-25°C) in the dark for 15 min. The absorbance of sample and control sample was determined at 517 nm and the DPPH free radical scavenging activity was calculated by following formula:

$$\%DPPH\ radical\ scavenging\ activity = \frac{OD\ at\ 517\ nm\ of\ control - OD\ at\ 517\ nm\ of\ sample}{OD\ at\ 517\ nm\ of\ control} \times 100 \quad (5)$$

Modelling

A complete second order quadratic model was employed to correlate the independent process variables. For each term of the second order polynomial equation was determined through multiple regression analysis using Design Expert software. The responses for different experimental combinations were related to the coded variables (x_i, i=1, 2 and 3) by a second degree polynomial equation as given below:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1.x_2 + \beta_{13} x_1.x_3 + \beta_{23} x_2.x_3 + \varepsilon \quad (6)$$

The coefficients of the polynomial were represented by β_0 (constant), β_1, β_2, β_3 (linear effects); β_{12}, β_{13}, β_{23} (interaction effects); β_{11}, β_{22}, β_{33} (quadratic effects); and ε (random error). The experiments were designed using the software, Design Expert Version 6.0.10 (State-Ease, Minneapolis, MN). The same software was used for statistical analysis of experimental data.

Experimental data were fitted to the selected models to derive the regression coefficients. Statistical significance of the terms in the regression equation was examined by analysis of variance (ANOVA) for each response. ANOVA is important in determining the adequacy and significance of the quadratic model. The p-values were used to check the significance of each of the coefficients, which is necessary to understand the pattern of the mutual interactions between the test variables. For more significant coefficient, the magnitude of p value should be low. The significances of all terms in the polynomial were judged statistically by computing the F-value at a probability (P) of 0.05.

A complete second order quadratic model employed to fit the data and adequacy of the model was tested considering R^2 (the coefficient of determination, a measure of the amount of variation around the mean explained by the model) *Adj* R^2, *Pred* R^2, Adequate Precision, *LoF* (lack of fit) and Fisher's F-test (Montgomery, 2001). The model is reliable with an R^2 value closer to 1 (The closer the R^2 value is to 1, the better is the model fit to the experimental data). Coefficient of determination, R^2 is defined as the ratio of the explained variation to the total variation and is measure of the degree of fit. It is also the proportion of the variability in the response variables, which is accounted for the regression

Table 2. Effect of different process conditions on the various responses

Sample No.	Water activity (a_w)	Colour values L*	a*	b*	WSI (%)	WAI (g/g)	Hygroscopicity (g/100g)	Bulk density (g/ml)	Process yield (%)	Beta carotene (mg/100g)	Moisture content (%)	DPPH Scavenging (%)
1	0.24	95.34	5.2	23.60	85.20	14.79	15.45	0.38	71.74	3.65	3.60	51.66
2	0.15	97.87	2.62	11.33	97.60	4.8	12.53	0.42	45.54	2.80	2.65	40.67
3	0.18	94.96	4.85	24.45	87.30	12.85	16.67	0.28	73.66	2.47	2.5	44.86
4	0.10	97.45	2.72	14.61	96.50	2.62	13.45	0.27	48.94	2.2	1.55	35.13
5	0.25	95.48	4.44	23.09	82.85	13.8	13.23	0.40	65.37	3.65	3.55	51.64
6	0.13	97.01	4.36	17.35	96.82	4.15	10.36	0.45	43.39	2.79	2.75	40.66
7	0.15	94.31	5.08	21.58	87.55	12.03	14.31	0.34	69.15	2.46	2.97	44.85
8	0.09	96.22	4.64	20.24	99.41	2.39	11.21	0.38	46.00	2.19	2.25	35.13
9	0.27	93.5	8.2	33.70	76.40	17.85	16.29	0.38	78.75	3.42	3.39	55.84
10	0.13	97.14	4.46	17.64	99.69	1.35	10.90	0.47	37.47	2.43	1.65	37.27
11	0.22	96.97	3.63	13.97	90.17	8.6	12.85	0.38	61.68	3.26	2.86	45.93
12	0.13	96.24	3.51	17.15	93.24	6.75	14.55	0.27	66.52	1.85	1.80	34.58
13	0.12	96.14	3.14	16.91	95.25	7.76	14.56	0.26	53.56	2.99	3.20	40.83
14	0.12	95.79	3.92	16.45	92.85	7.75	11.49	0.39	48.81	3.04	3.89	40.82
15	0.12	95.61	3.67	17.65	92.20	8.35	13.35	0.26	60.33	3.12	2.62	39.52
16	0.12	95.5	3.59	17.45	92.34	8.46	13.45	0.28	60.34	3.14	2.64	39.51
17	0.13	95.67	3.75	17.59	92.54	8.65	13.47	0.25	60.24	3.15	2.60	39.54
18	0.11	95.62	3.95	17.15	92.17	8.43	13.48	0.27	60.38	3.13	2.55	39.53
19	0.13	95.56	3.97	17.05	91.45	8.55	13.49	0.29	60.05	3.14	2.66	39.5
20	0.13	96.05	4.7	18.93	93.31	8.95	13.49	0.29	61.87	3.18	2.91	40.43

analysis (Mclaren *et al*., 1977). In a multiple linear regression model, adjusted R^2 measures the proportion of the variation in the dependent variable accounted for by the explanatory variables. The adjusted R^2 denotes the percentage of variation explained by only the independent variables that actually affect the dependent variable. Adequate precision compares the range of the predicted values, at the design points to the average variance, of the prediction (a function of model parameters, the number of points, and the variance, estimated by the root mean square residual from ANOVA). The adequate precision value should be greater than 4 for fitness of model for prediction purposes (Montgomery, 2001). Lack of fit value was also taken into consideration for judging the fitness of the model.

Numerical multi-response optimization technique was adopted to determine the optimum conditions for the spray drying of the pumpkin juice powder using software, Design Expert. The constraints were selected depending upon their acceptance or unacceptance. The regression coefficients were then used to make statistical calculation to generate three-dimensional plots from the regression model. Design Expert 6.0 was used for this purpose and contour plots were developed for selected parameters. Design Expert (version 6.0, by STAT-EASE Inc., USA) was used for the analysis of the data.

Results and Discussion

Pumpkin powder prepared with maltodextrin concentrations (10-30%), inlet air temperature (140-210°C) and feed flow rate (8-12 rpm) at constant blower speed, 2400 rpm in spray drying were analyzed for different physico-chemical, functional and antioxidants properties. Effect of maltodextrin concentration, feed flow rate and inlet air temperature on water activity, colour – L*, a* and b*, water solubility index, water absorption index, hygroscopicity, bulk density, process yield, beta carotene, moisture content, DPPH radical scavenging activity is shown in Table 2. A second order polynomial model for the dependent variables was established to fit the experimental data (Table 3).

Water activity

Water activity (a_w) is an important index because it can greatly affect the shelf life of the powder. It varied from 0.09 to 0.27, indicating that pumpkin powders under the different designed conditions were microbiologically stable (Table 2). The change in water activity may be mainly because of change in composition and processing conditions. The coefficient of determination, R^2, was 0.98 (Table 3) for the polynomial regression model. Adjusted R^2 was 0.97. The Model F-value of 60.28 whereas lack of fit was not significant. It implied that the model was significant for the simulation of the response. It was observed from Table 3 that linear, square terms of maltodextrin concentration (x_1) and temperature(x_2) and their interaction (x_1x_2) were significant (P<0.05).

Figure 1(a) shows the interaction effect of maltodextrin concentration (x_1) and inlet air

Table 3. Regression coefficients of the second-order polynomial model and their significance

Coefficients	Water activity	Colour L*	Colour a*	Colour b*	WSI	WAI
β_0	$+0.12^{***}$	$+95.66^{***}$	$+3.96^{***}$	$+17.62^{***}$	$+92.31^{***}$	8.55^{***}
β_1	-0.04^{***}	$+1.07^{***}$	-0.84^{***}	-4.12^{***}	$+6.34^{***}$	-4.93^{***}
β_2	-0.03^{***}	-0.29^{**}	+0.03	$+0.79^{**}$	$+0.98^{**}$	-0.79^{***}
β_3	−0.004	-0.25^{**}	$+0.34^{**}$	+0.61	−0.29	-0.22
β_{11}	$+0.03^{***}$	0.11	$+0.80^{***}$	$+2.88^{***}$	-1.46^{***}	0.40^{**}
β_{22}	$+0.02^{***}$	$+0.34^{***}$	−0.18	-0.69^{**}	−0.16	-0.28^{**}
β_{33}	−0.003	$+0.16^{**}$	-0.26	−0.36	$+0.85^{**}$	-0.31^{**}
β_{12}	$+0.009^{**}$	+0.04	+0.01	$+0.85^{**}$	−0.66	-0.03
β_{13}	−0.001	-0.20^{**}	$+0.52^{**}$	$+1.88^{**}$	+0.53	0.12
β_{23}	−0.004	−0.15	+0.15	−0.34	$+0.79^{**}$	0.07
R^2	0.98	0.98	0.93	0.98	0.99	0.996
Adjusted R^2	0.97	0.96	0.87	0.95	0.97	0.993
F	60.28^{***}	54.76^{***}	14.86^{***}	40.63^{***}	92.03^{***}	320.57^{***}
Adequate Precision	25.99	28.69	16.88	28.97	35.66	67.72
Lack of Fit	1.82	1.14	1.27	3.96^{*}	3.07	4.17^{*}
Coefficients	**Hygroscopicity**	**Bulk density**	**Process yield**	**ß carotene**	**Moisture content**	**DPPH radical scavenging**
β_0	+13.46	+0.27	+60.45	+3.15	$+2.68^{***}$	39.65^{***}
β_1	-1.55^{***}	$+0.02^{***}$	-12.12^{***}	-0.29^{***}	-0.47^{***}	-5.32^{***}
β_2	$+0.51^{***}$	-0.04^{***}	$+1.45^{***}$	-0.44^{***}	-0.37^{***}	-3.20^{***}
β_3	-1.09^{***}	$+0.03^{***}$	-1.85^{***}	+0.003	0.18^{**}	-0.004
β_{11}	$+0.05^{**}$	$+0.05^{***}$	-0.62^{**}	-0.082^{***}	-0.08	2.49^{***}
β_{22}	$+0.09^{**}$	$+0.02^{***}$	$+1.50^{***}$	-0.21^{***}	-0.15^{**}	0.26^{**}
β_{33}	-0.19^{***}	$+0.02^{***}$	-3.78^{***}	-0.07^{***}	0.34^{***}	0.59^{**}
β_{12}	-0.07^{**}	-0.01	+0.04	0.15^{***}	0.01	0.32^{**}
β_{13}	+0.02	+0.01	$+0.72^{**}$	-0.001	0.05	0.003
β_{23}	-0.03	0.02^{**}	+0.13	-0.001	0.14^{**}	0.003
R^2	0.998	0.96	0.997	0.998	0.969	0.998
Adjusted R^2	0.997	0.93	0.993	0.996	0.941	0.995
F	819.09^{***}	29.35^{***}	320.23^{***}	609.82^{***}	34.63^{***}	449.61^{***}
Adequate Precision	106.20	15.68	64.89	90.16	19.89	75.38
Lack of Fit	3.89^{*}	1.61	2.57	2.94	1.84	1.21

*Significant at $P < 0.1$, **Significant at $P < 0.05$, ***Significant at $P < 0.001$ df: degrees of freedom

temperature (x_2) on water activity. It was observed that water activity decreased with the increase in inlet air temperature and maltodextrin concentration. This may be due to decrease in available moisture at increased inlet air temperature and higher concentration of maltodextrin. The negative correlations of air inlet temperature and MD with water activity are in agreement with the results reported for watermelon powder by Quek *et al.* (2007). The reduction in water activity of pumpkin powder from 0.30 to 0.17 with the increase in air temperature from 140-190ºC was also reported by Shavakhi *et al.* (2012).

Colour L^* Value

The color, L^* value of powder varied from 93.50 to 97.87, which showed variation of darkness/whiteness of pumpkin powder under the different designed conditions (Table 2). The changes in L value may be due to change in composition of ingredients and processing conditions. The coefficient of determination, R^2 and Adjusted R^2 for the model were found 0.98 and 0.96 respectively (Table 3) whereas the model F-value was 54.76. Adequate Precision (28.69) showed that that the model can be used to predict L value under the set of designed conditions. All the process variables at linear level (Table 3), temperature (x_2) and feed flow rate (x_3) at quadratice level and maltodextrin concentration and feed flow rate at interactive level had the significant effect ($P<0.05$). It was observed that an increase in maltodextrin concentration caused a significant increase in lightness (L^* value) of the powder. This may be due to white colour of maltodextrin, resulting in higher lightness of powders, represented by a higher L^* value. Similar results have been reported for spray dried pineapple juice powders (Abadio *et al.*, 2004), gac fruit powder (Kha, 2010) and sweet potato powders (Grabowski *et al.*, 2006). The lightness was also decreased slightly with increase in feed flow rate.

Colour a^* and b^* value

The color, a^* value of powder varied from 2.62 to 8.20 (Table 2). The changes may be due to change in composition and processing conditions. Color, a^* value was predominantly affected by maltodextrin concentration and feed flow rate at linear level, maltodextrin concentration at quadratic level and maltodextrin concentration and feed flow rate at interactive level at inetractive level ($P<0.05$). It was observed that the color, a^* value decreased with the increase in maltodextrin concentration. The increase in flow rate caused an increase in redness of the product, which may be due to the change in processing time. The combined effect of both maltodextrin and feed rate was prominent,

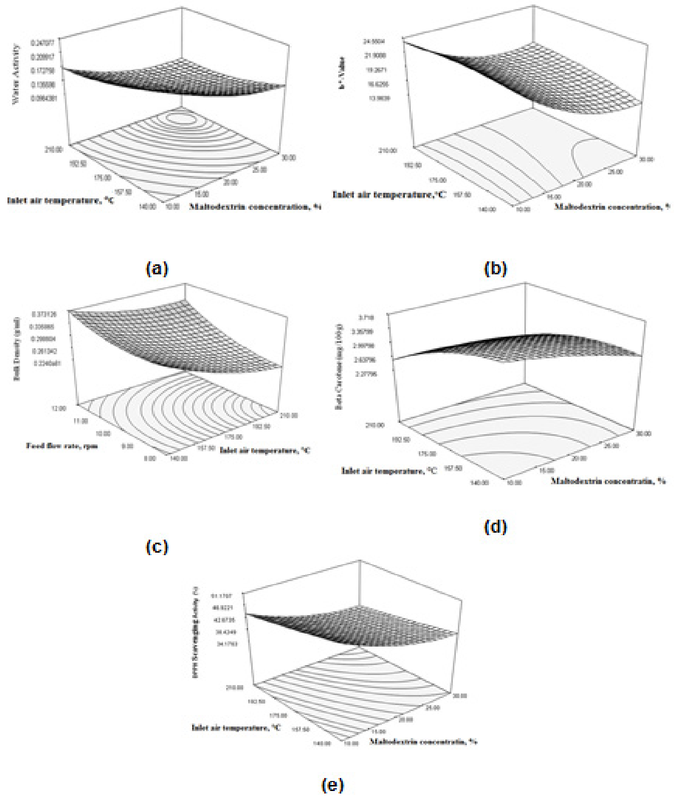

Figure 1. Response Surface plots of (a) water activity (b) colour b* (c) bulk density (d) beta carotene and (e) antioxidant activity as a function of independent variables

which may be due to the variation in maltodextrin concentration during processing and solubility with the juice which created an appropriate mix for the feeding into the spray drying. The color b^* value of powder varied from 11.33 to 33.70 (Table 2) and was prominently affected by the linear and square terms of maltodextrin concentration, temperature; and interactions of maltrodextrin concentration with air inlet temperature (x_1x_2) and feed flow rate (x_1x_3) separately ($P<0.05$) (Table 3). An increase in maltodextrin concentration decreased color b^* value (Figure 1(b)) (yellowness), which may be due to decrease in proportion of pumpkin juice with increase in maltodextrin concentration. The pumpkin juice was mainly responsible for the yellow color due to substantial amount of carotenoids pigments. The increase in yellowness was observed with the increase in air temperature, which may possibly be due to application of heat during spray drying which may enhance nonenzymatic reactions, resulting in the generation of more yellow color. The combined effect of both maltodextrin and feed rate was prominent, which may be due to the interaction effect of pumpkin juice and maltodextrin concentration during the process.

Water solubility index and water absorption index

Water solubility index is generally indicated the degradation of molecular components (Tangirala *et al.*, 2012) and measures the degree of starch conversion during heat processing, which is the amount of soluble polysaccharide released from the starch (Yagci and Gogus, 2008). WSI of powder varied from 76.40 to 99.69%. The data showed variation in dispersing characteristics of pumpkin powder under the different designed conditions (Table 2). The change may be due to change in composition and processing conditions. The linear terms of maltodextrin concentration and temperature; and square terms of maltodextrin concentration and feed flow rate whereas inlet air temperature and feed flow rate (x_2x_3) at intercative level were the significant parameters ($P<0.05$) (Table 3). An increase in maltodextrin concentration and temperature increased the solubility index, which may be due to addition of maltodextrin and then dextrinization of the same at higher temperatures. Similar increase in dextrinization of starch like materials in extrudates has been reported by Kumar *et al.* (2010a, 2010b). WAI is determined by measuring the volume occupied by the starch after swelling in excess water, which maintains the integrity of starch in aqueous dispersion (Mason and Hoseney, 1986). WAI of powder varied from 1.35 to 17.85 g/g. (Table 2). Table 3 showed the all the process variables affected WAI at quadratic level but maltodextrin concentration and temperature were the significant parameters at linear level (P<0.05).

Hygroscopicity

Hygroscopicity is the measure of adherence of a substance to attract and hold water molecules from the surrounding environment through either absorption or adsorption with the material by an increase in stickiness, volume, or other physical characteristic of the material. The hygroscopicity of powder varied from 10.36 to 16.67g/100g. All linear and square terms; and interaction term of maltodextrin and temperature (Table 3) were found significant (P<0.05). An increased maltodextrin concentration decreased hygroscopicity. This may be due to the fact that maltodextrin is a material with low hygroscopicity and validate its efficiency as a carrier agent. Decrease in hygroscopicity with increasing concentrations of maltodextrin concentration has also been reported by Tonon *et al.* (2008) and Moreira *et al.* (2009). With increase in inlet air temperature, hygroscopicity was also increased. This is in agreement with the results for pumpkin juice (Bas and Boyaci, 2007). Similar positive correlation of hygroscopcity with temperature for acai powder was also observed by Tonon *et al*. (2008).

Bulk density

The bulk density of powder varied from 0.25 to 0.47 g/ml, under the different designed conditions (Table 2). The variation may be due to change in ingredient composition and processing conditions. Figure 1(c) shows the interaction effect of inlet air temperature and feed flow rate (x_2x_3) on bulk density. Bulk density increased with the increase in feed flow rate. The higher feed flow rate may cause lesser retention time of material in contact to heat, which may lead to comparatively higher moisture content and higher density. The decrease in bulk density with the increase in temperature was also observed. The results are in agreement with the finding of a number of studies (Cai and Croke, 2000; Kha, 2010). At higher temperature, higher drying rates are generally achieved, implying shrinkage of the droplet consequently lower density of the powder (Chegini and Ghobadian, 2005).

Process yield

The process yield of powder varied from 37.47 to 78.75%, (Table 2) under the different set of designed conditions. The coefficient of determination, R^2 and Adjusted R^2 were 0.997 and 0.993 respectively (Table 3) for the polynomial regression model for process yield, whereas lack of fit was non-significant. The model F-value, 320.23 and Adequate Precision, 64.89 showed that the model can be used to predict process yield. All the process variables at linear and quadratic level and temperature and feed rates at the interactive level had significant effect (P<0.05) (Table 3). It was also observed that process yield was slightly increased with an increase in feed flow rate up to 10 rpm and decreased further. A decrease in process yield was observed with increase in maltodextrin concentration, which may probably be due to increased viscosity of the mixture. The higher viscosity may result in pasting of mixture on chamber wall and low process yield of powder. Similar negative influence of MD concentration on process yield has been reported by Tonon *et al.* (2008). The use of MD to obtain fruit powders is unavoidable but it is important to maximize the proportion of fruit juice relatively to maltodextrin concentration (Bhandari *et al*., 1993). The drying index which is an indicator of the spray-drying ability of a given component is 0.27, 0.51 and 0.85 for fructose, glucose, and sucrose, respectively (Bhandari *et al*., 1997). This indicates that fructose is the most difficult component for spray drying followed by glucose and sucrose.

Beta carotene

The β-carotene content of powder varied from 1.85 to 3.65 mg/100g (Table 2). The coefficient of determination, R^2 and Adjusted R^2 were 0.998 and 0.996 respectively (Table 3) for the polynomial regression model for beta carotene, whereas lack of fit was non-significant. The model F-value, 609.82 and Adequate Precision, 90.16 showed that the model can be used to predict β-carotene content. The feed flow rate at the linear level and interactive level did not reveal any significant effect (P<0.05) (Table 3) whereas the process variables, maltodextrin and temperature had the prominent effect on the β-carotene content (Table 3). Pumpkin is a rich source of antioxidant, β-carotene, which is one of the plant carotenoids, gets converted into Vitamin A in the body. Maltodextrin is devoid of this antioxidant and higher temperature affects carotene content therefore both the parameters were found to have dominating effect. The β-carotene content of powder decreased with the increase of maltodextrin concentration, which may be due to decrease in pumpkin juice concentration (Figure 1(d)). It also decreased with increase in inlet air temperature. High temperature may degrade the carotene content. The negative correlation of air inlet temperature and MD with β-carotene is in agreement with the results for gac powder (Kha, 2010).

Moisture content

The moisture content of powder varied from 1.55 to 3.89%. It is apparent that the moisture content of the powder decreased with the increase in temperature, while it increased with the increase in feed flow rate. In spray drying, the main variable which controls the moisture content of the resulting powder is inlet and outlet temperatures. In general, the temperature of the exhaust air leaving the drying chamber in spray drier controls the residual moisture in the powder and lower moisture can be achieved by higher outlet temperature and longer exposure time. Goula and Adamopoulos (2008), and Tonon *et al.* (2008) reported a reduction of powder moisture content when the air temperature in spray drying of tomato juice and acai pulp was increased. There is a greater temperature gradient between the atomized feed and drying air, which might result in a grater heat transfer, thus greater driving force for water evaporation and, therefore may yield powders with lower moisture content. Powder's moisture content increased with feed flow rate. With increase in maltodextrin concentration, moisture of the powder decreased. Lower moisture content can be reached by higher feed solids contents. An increase in solids in the feed may reduce free water amount for evaporation due to comparatively lesser amount of heat available for it (Kurozawa *et al*., 2008).

DPPH radical scavenging activity (%)

The antioxidant (DPPH radical scavenging) activity of powder varied from 34.58 to 55.84% (Table 2). Maltodextrin and temperature (Table 3) mainly affected the antioxidant activity ($P<0.05$). Figure 1(e) shows the effect of maltodextrin concentration and inlet temperature on DPPH radical scavenging activity of powder. With increase in maltodextrin concentration, DPPH radical scavenging activity was decreased. This decrease may be attributed due to decrease in pumpkin juice concentration in the feed mixture. The increase in inlet air temperature, which also caused decreased DPPH radical scavenging activity, may be due to degradation of some bioactive compounds. The negative correlation of air inlet temperature and MD with DPPH radical scavenging activity in terms of antioxidant activity is in agreement with the results for gac fruit powder (Kha, 2010).

Optimization of process variables for pumpkin juice powder

A numerical multi-response optimization technique was adopted to determine the optimum conditions for the spray drying of the pumpkin juice powder. The main criterion for constraints optimization was maximum β-carotene, DPPH radical scavenging activity, colour value (a^* value and b^* value), water solubility index, water absorption index and process

Table 4. Optimization of process parameters and the verification of optimized conditions

		Optimum value		
		(In range*)	(Targeted)	
Variables	M.D. Concentration (%)	14.92	15	
	Inlet air temperature (°C)	176.59	177	
	Feed flow rate (RPM)	10.47	10	
		Predicated value	Experimental value	Coefficient of variation
Responses	Water activity	0.15	0.14	4.88%
	L* value	95.05	96.54	1.10%
	a* value	4.59	4.91	4.76%
	b* value	20.36	21.71	4.54%
	WSI (%)	80.34	82.58	1.94%
	WAI (g/g)	11.36	10.09	8.37%
	Hygroscopicity (g/100g)	14.01	13.25	3.94%
	Bulk Density (g/ml)	0.28	0.29	2.48%
	Process yields (%)	65.77	64.53	1.35%
	ß carotene (mg/100gm)	3.24	3.06	4.04%
	Moisture Content (%)	2.93	3.25	7.32%
	DPPH radical scav. (%)	42.87	40.95	3.24%

Note: *As 14.92%, 176.59°C and 10.47 RPM are difficult to maintain hence maltodextrin concentration, inlet air temperature and feed flow rate were targeted as 15%, 177˚C and 10 rpm respectively, for carrying out the experiments to verify the predicted values.

yields; minimum value of hygroscopicity, moisture content and water activity of pumpkin juice powder while L^* value and bulk density were kept in range.

The optimum operating conditions for spray dried pumpkin juice powder were found as maltodextrin concentration; 14.92%, inlet air temperature; 176.59°C and Feed flow rate; 10.47 rpm. The responses predicted by the design expart-6 software for these optimum process conditions were water activity 0.15, L^* value 95.05, a^*value 4.59, b^* value 20.36, water solubility index 80.34%, water absorption index 11.36 g/g, hygroscopicity 14.01 g/100g, bulk density 0.28 g/ml, process yield 65.77%, beta carotene 3.24 mg/100g, moisture content 2.93% and DPPH radical scavenging activity 42.87%.

It was difficult to maintain the actual optimized conditions therefore these optimized values were targeted to facilitate the control of experiments under the precise conditions. The process variables, such as maltodextrin concentration, inlet air temperature and feed flow rate were targeted as 15%, 177˚C and 10 rpm respectively rather than their actual optimized values (14.92%, 176.59°C and 10.47 rpm) to carry out the experiments. The experimental values under the optimum process conditions were found as water activity 0.14, L^* value 96.54, a^*value 4.91, b^* value 21.71, water solubility index 82.58%, water absorption index 10.09g/g, hygroscopicity 13.25 g/100g, bulk density 0.29 g/ml, process yield 64.53%, beta carotene 3.06 mg/100g, moisture content 3.25%

and DPPH radical scavenging activity in terms of antioxidant activity 40.95%. The experimental values were very close to the predicted value (Table 4) with a COV lesser than 8.37%.

Conclusion

The quality parameters of the pumpkin powder were affected by changes in maltodextrin concentration, inlet air temperature and feed flow rate. With increase in maltodextrin concentration, water activity, process yields, water absorption index, Color a^* value , b^* value, hygroscopicity, moisture content, beta carotene content, DPPH radical scavenging activity were decreased whereas water soluble index and L^*-value were increased. The results also showed that with increase in inlet air temperature level, the colour value, a^* and b^*, water solubility index, hygroscopicity, process yield were increased and water activity, L^*value, water absorption index, beta carotene, DPPH radical scavenging activity, moisture content were decreased in spray dried pumpkin powder. The optimum process variables were 15% of maltodextrin concentration, 177˚C of inlet air temperature and 10 rpm of feed flow rate to produce spray dried powder of optimum quality. The closed correlation of experimental and predicted value revealed the suitability of the models for the prediction of responses.

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