Optimization of process parameters for the production of spray dried *Ber* (*Ziziphus jujube* L.) powder

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Abstract The study covers effect of operating variables of spray dryer i.e. inlet temperature (170, 180, 190, 200 and 210 °C), aspirator blower capacity (40, 50, 60, 70 and 80 %) and feed pump capacity (9, 12, 15, 18, and 21 %) and processing parameters of feed i.e. total soluble solid (TSS) of feed (7.5 %) and encapsulating material, maltodextrin (4, 6, 8, 10, and 12 %), aerosil (1.0 %), citric acid (0.25 %) upon the physical properties (colour, packed density and hygroscopicity) of powder were observed. After complete evaluation and analysis of all the attributes for physical properties i.e. colour, packed density (0.45 g/cc) and hygroscopicity (0.17 g/g dry matter), it was concluded that best quality of *Ber* powder were obtained at inlet air temperature 190 °C, aspirator blower capacity 60 %, feed pump capacity 15 %, encapsulating material 8 %.

Keywords Colour · Density · Hygroscopicity · Spray dryer · Maltodextrin

Introduction

India ranks second in fruit production next to China and contribute 10 % of total fruits production in the world (Ojha 2009). The total production of fruits has gone up from 32.98

to 53.53 million tonnes over a period of last 12 years from the year 1992–93 to 2004–05 (Kantwa 2009). But only 3 % of total fruits produced are used for processing and rest 25 to 35 % of the production goes waste because of inadequate handling, storage and processing technology. Among the horticultural commodities, fruits and vegetables owe its own role, contributing over 24 % of countries agricultural GDP (John 2008). The recommended requirement of fruits per capita per day is 120 g, while in India, availability of fruits per capita per day is 75 g (Rathore 2010). The excellent human resources with large network in research and development under the National Agricultural Research Scheme (NARS) on horticulture have been asset that help in development and transfer of technologies to respective users (Shikhamany 2006). Dried juice products today are used mainly as convenient foods and have long storage life at ambient temperatures (Pap 1995).

Spray dryers are employed in the transformation of liquid materials (solutions, suspensions, and pastes) into solid products with desired moisture and used in many sectors like chemical, pharmaceutical, mineral and food industries (Mazza et al. 2003). The rotating air broom system attached at the conical bottom of the spray dryer reduced the sticking by 33 % for banana powder (Sudhagar 2000). Conventionally, *Ber* is considered as a poor man's fruit. *Ber* fruits are consisted moisture content (81.6 %), sugar (5.4 g), protein (0.8 g), fat (0.07 g) and citric acid (1.1 g) Per 100 g of edible Portion of fresh fruits. It is a rich source of vitamin C, A and B-complex. *Ber* fruit is richer than apple in protein, phosphorus, calcium, carotene and vitamin C. Fully ripe fruits have a food value of 20.9 calories (Tiwari 1995). Storage experiments in India showed that slightly under ripe fruits ripen and keep for 8 days under wheat straw, 7 days under leaves, and 4 days in carbide (Morthon and Miami 1987). Spray-drying, a

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well-characterized pharmaceutical unit operation is ideally suited to producing solid amorphous dispersions due to its rapid drying kinetics (Dobry et al. 2009). Spray drying has not only contributed in drying of fluids but also has played a vital role in encapsulation and microencapsulation of valuable foods and functional nutraceutical ingredients (Murugesan and Orset 2011). The moisture content of the powder varied with the maltodextrin level, inlet air temperature, and air flow rate. Using scanning electron microscope images, larger particle size was observed with the increase of maltodextrin concentration. Powder bulk density also increased from 0.41 to 0.68 g/cm³, depending on the level of maltodextrin, whereas the drying air temperature and flow rate were not as effective (Zareifard et al. 2011). Adding a carrier increased the Glass transition temperature (T_g) of the powder and its storage stability (Yousefi et al. 2010). Maltodextrin is an effective drying aid for spray drying of watermelon juice. Addition of maltodextrin reduced the stickiness of the products and altered the physicochemical properties of the spray dried powders (Quek et al. 2007).

Materials and methods

Experimental techniques used in blending the encapsulating material in fruit juice, process of fruit juice preparation, working principle of in Lab Spray Dryer (LU-222 Advanced Model, Twin cyclone) as per experimental plan, drying of fruit juice in existing spray dryer and procedure for determining physical properties.

Preparation of *Ber* juice

The *Ber* juice was prepared by two methods, boiling and without boiling. It was found that sedimentation of *Ber* pulp residue was less in juice prepared by boiling than without boiling. Preparation of *Ber* juice by without boiling method steps is same as boiling method except boiling and meshing steps. Prepared *Ber* juice composition was TSS (7.5 %), pH (3.2) and citric acid (1.1 mg). Procedure for the preparation of juice by boiling method mass balance of raw material to *Ber* juice for the spray drying is described Fig. 1.

Preparation of powder

Preheated samples at temperature of 50 °C (Garg and Chandra 2005) were taken and fed to the Spray Dryer as per experimental plan. Drying of incoming liquid feed process is accomplished by the atomization of the liquid feed stream into droplets and their dispersion in a hot gaseous phase. Due to small size of the droplets, the heat and mass transfer per unit volume is considerably high,

Fig. 1 Flow chart of preparation of *Ber* fruit juice

providing the possibility of achieving high particle drying rates. The dried particles are recovered in collection pot 1st (coarse particle, 40–60 μm), pot 2nd (fine particle, 20–40 μm) - and 3rd (ultra fine particle, 5–15 μm) respectively by gravity and through the use of cyclones separator. Initially, sample was collected for the determination of physical properties for a specific period of time and kept in sealed glass bottle and sealed. Working principle of Spray Dryer flow diagram is given in Fig. 2.

Experimental plan

In the present study the effect of processing parameters of feed i.e. total soluble solid (TSS) of feed (7.5 %) and encapsulating material, maltodextrin (EM) i.e. 4, 6, 8, 10, and 12 %, aerosil (1.0 %), citric acid (0.25 %) and operational parameters of Lab Spray dryer i.e. inlet temperature (IAT) i.e. 170, 180, 190, 200 and 210 °C, aspirator blower capacity (ABC) i.e. 40, 50, 60, 70 and 80 % and feed pump capacity (FPC) i.e. 9, 12, 15, 18, and 21 % on physical

Fig. 2 Flow diagram of working principle of Lab Spray Dryer (LU 222 Advanced Model, Twin cyclone)

properties i.e. Colour, Packed Density (PD) and Hygroscopicity (HY) of powder were observed.

Experimental design

Inlet air temperature (IAT), aspirator blower capacity (ABC), feed pump capacity (FPC) and encapsulating material of maltodextrin (EM). In this study central composite rotatable design (CCRD) with full replicate of four independent variables with five levels of each was chosen. The level of coded variables and design matrix is presented in Tables 1 and 2 respectively. The details of levels of variable with experimental plan given in Table 2, show the experimental plan consisted of 32 treatment combinations of each independent chosen variable. The observations recorded were analyzed and best fit regression equations were developed.

Analysis of powder

Colour The colour of spray dried product was determined by Hunter L*a* and b* colour Flex spectrophotometer model 45°/0° Hunter Association Laboratory, Virginia.

Packed bulk density Packed bulk density was calculated from the weight of powder contained in the cylinder after being tapped by hand on a bench for 100 times from a height of 10 cm (Goula and Adamopoulos 2004).

Hygroscopicity About 1 g of powder was spread evenly on Petri dishes (9 cm diameter) to allow for a high surface area between humid air and powder. Samples of each powder in the dishes were placed in desiccator under the conditions: 23 °C and 76 % relative humidity using HNO₃ solution. Ten minutes interval was selected to get the kinetics of moisture sorption (Kahtani, and Hassan 1990). The gain in weight of the samples was considerably lower after 90 min. Although hygroscopicity is based on the equilibrium moisture content, to compare hygroscopicities, the weight increase per gram of powder solids after being subjected to the atmosphere with relative humidity of 76 % for 90 min was determined (Papadakis et al. 1998).

Table 2 Experimental design matrix for levels of parameter

Run	Coded values				Actual values			
	X ₁	X ₂	X ₃	X ₄	X ₁	X ₂	X ₃	X ₄
1	−1	−1	−1	−1	180	50	12	6
2	1	−1	−1	−1	200	50	12	6
3	−1	1	−1	−1	180	70	12	6
4	1	1	−1	−1	200	70	12	6
5	−1	−1	1	−1	180	50	18	6
6	1	−1	1	−1	200	50	18	6
7	−1	1	1	−1	180	70	18	6
8	1	1	1	−1	200	70	18	6
9	−1	−1	−1	1	180	50	12	10
10	1	−1	−1	1	200	50	12	10
11	−1	1	−1	1	180	70	12	10
12	1	1	−1	1	200	70	12	10
13	−1	−1	1	1	180	50	18	10
14	1	−1	1	1	200	50	18	10
15	−1	1	1	1	180	70	18	10
16	1	1	1	1	200	70	18	10
17	−2	0	0	0	170	60	15	8
18	2	0	0	0	210	60	15	8
19	0	−2	0	0	190	40	15	8
20	0	2	0	0	190	80	15	8
21	0	0	−2	0	190	60	9	8
22	0	0	2	0	190	60	21	8
23	0	0	0	−2	190	60	15	4
24	0	0	0	2	190	60	15	12
25	0	0	0	0	190	60	15	8
26	0	0	0	0	190	60	15	8
27	0	0	0	0	190	60	15	8
28	0	0	0	0	190	60	15	8
29	0	0	0	0	190	60	15	8
30	0	0	0	0	190	60	15	8
31	0	0	0	0	190	60	15	8
32	0	0	0	0	190	60	15	8

X₁=Inlet air temperature, °C, X₂=Aspirator blower capacity, % (100 %=85 m³/h)

X₃=Feed pump capacity, % (100 %=1,000 ml/h), X₄=Encapsulating material maltodextrin, % (V/W basis)

Table 1 Level of coded variables

Sr. No.	Independent variables	Code	Coded and actual values				
			−2	−1	0	1	2
1.	Inlet Temperature °C)	X ₁	170	180	190	200	210
2.	Aspirator blower capacity, % (100 %=85 m ³ /h)	X ₂	40	50	60	70	80
3.	Feed pump capacity, % (100 %=1,000 ml/h)	X ₃	9	12	15	18	21
4.	Encapsulating material, % (V/W basis)	X ₄	4	6	8	10	12

Results and discussion

In spray drying process various operating condition like inlet air temperature, aspirator blower capacity, feed pump capacity etc., influence the preparation of *Ber* juice powder in a complex fashion and effect are listed in Table 3. Moisture content of the powder was varied from 1.6 to 5.5 % wet basis within the experimental region. The standard deviation, coefficient of variation, mean and predicted residual error sum of square (PRESS), values, coefficient of determination and predicted R^2 , adequate precision, F-value and P-value of are given in Table 4.

Table 4 The standard error, mean, coefficient of variation, predicted residual error of sum of squares (PRESS), coefficient of determination, adjusted and Pred R-Squared and adequate precision values for developed models

Particulars	Model No. 1	Model No.2
SD	0.017504	0.062517
Mean	0.259688	0.363438
C.V.%	6.740217	17.20149
PRESS	0.02755	0.380254
R^2	0.928942	0.687071
Adj. R^2	0.870424	0.429365
Pred. R^2	0.624126	−0.79093
Adeq precision	16.82823	6.746143
F-Value	2.666102	15.87049
P-value	0.0001	0.0001

Table 3 Spread Sheet of different data of various dependent & independent parameter

	Run	Block	IAT	ABC	FPC	EM	Colour			PD	HY	OT
							L	a	b			
	1	1	180	50	12	6	54.26	8.21	18.24	0.35	0.14	102.5
	2	1	200	50	12	6	58.23	9.25	19.56	0.26	0.24	110.4
	3	1	180	70	12	6	56.32	8.56	18.23	0.36	0.15	102.3
	4	1	200	70	12	6	55.23	7.23	20.24	0.25	0.23	112.6
	5	1	180	50	18	6	55.65	8.23	18.23	0.34	0.16	103.3
	6	1	200	50	18	6	61.24	9.26	18.24	0.24	0.25	108.5
	7	1	180	70	18	6	58.23	8.98	20.89	0.34	0.14	104.2
	8	1	200	70	18	6	65.23	9.12	19.25	0.26	0.23	109.8
	9	1	180	50	12	10	54.96	7.89	21.23	0.35	0.15	101.8
	10	1	200	50	12	10	59.23	9.89	18.23	0.23	0.24	110.8
	11	1	180	70	12	10	54.85	8.22	20.14	0.34	0.13	103.2
	12	1	200	70	12	10	61.25	6.98	19.56	0.26	0.23	115.5
	13	1	180	50	18	10	64.25	9.25	20.56	0.34	0.15	104.2
	14	1	200	50	18	10	59.32	8.65	19.56	0.25	0.25	106.2
	15	1	180	70	18	10	58.45	7.89	20.56	0.35	0.16	101.2
	16	1	200	70	18	10	57.23	9.56	18.24	0.27	0.23	109.5
	17	1	170	60	15	8	65.43	8.56	24.35	0.38	0.12	100.5
	18	1	210	60	15	8	53.69	7.99	20.22	0.25	0.26	122.3
	19	1	190	40	15	8	63.11	9.32	19.22	0.42	0.22	105.6
	20	1	190	80	15	8	62.12	8.23	20.21	0.48	0.15	104.6
	21	1	190	60	9	8	63.56	7.54	21.23	0.45	0.19	105.6
	22	1	190	60	21	8	61.52	8.45	21.32	0.46	0.18	106.3
	23	1	190	60	15	4	64.56	6.23	24.56	0.38	0.19	105.2
	24	1	190	60	15	12	63.52	8.65	23.12	0.44	0.19	106.5
	25	1	190	60	15	8	63.31	7.96	20.98	0.45	0.17	105.7
	26	1	190	60	15	8	63.15	7.95	20.65	0.45	0.19	105.2
	27	1	190	60	15	8	63.12	7.12	20.45	0.46	0.19	106.7
	28	1	190	60	15	8	63.18	7.32	20.87	0.45	0.19	105.8
	29	1	190	60	15	8	63.61	7.87	20.98	0.43	0.18	104.2
	30	1	190	60	15	8	63.19	7.96	20.45	0.45	0.19	105.3
	31	1	190	60	15	8	63.11	7.65	20.32	0.44	0.19	105.9
	32	1	190	60	15	8	63.51	7.87	20.21	0.45	0.18	105.1

IAT Inlet air temperature °C, ABC Aspirator blower capacity % (100 %=85 m³/h), FPC Feed pump capacity, % (100 %=1,000 ml/h), EM Encapsulating material, % (V/W basis), PD Packed Density g/cc, HY Hygroscopicity g/g dry matter and OT Outlet temperature °C

Packed density of powder

The variation is encompassed by a polynomial equation of second order. The equation in coded values generated by multiple regression analysis using CCRD reads as follows:

$$\begin{aligned} \text{Packed Density (g/cc)} = & 0.45 - 0.04212\mathbf{X}_1 + 0.00792\mathbf{X}_2 \\ & + 0.000423\mathbf{X}_3 - 0.00464\mathbf{X}_4 + 0.00313\mathbf{X}_1\mathbf{X}_2 \\ & + 0.00313\mathbf{X}_1\mathbf{X}_3 + .000635\mathbf{X}_1\mathbf{X}_4 + 0.00195\mathbf{X}_2\mathbf{X}_3 \\ & + 0.001962\mathbf{X}_2\mathbf{X}_4 + 0.00446\mathbf{X}_3\mathbf{X}_4 - 0.05173\mathbf{X}_1^2 \\ & - 0.01771\mathbf{X}_2^2 - 0.0168\mathbf{X}_3^2 - 0.0278\mathbf{X}_4^2 \end{aligned}$$

An interaction between the different variables under study was revealed a poor value of R^2 i.e. 0.68. Adequacy of fit of presented model produced 'F' value (2.667) more than unity, which is clearly significant. Figure 3a, shows that the packed density decreased from 0.38 to 0.25 g/cc with increase in inlet air temperature from 170 to 210 °C, this may be because increasing the drying air temperature generally produces low moisture content in finished product. Packed density

increased from 0.42 to 0.48 g/cc with increase in aspirator blower capacity from 40 to 80 % due to a high aspirator speed leading to a shorter residence time in drying chamber resulting in a larger amount of residual moisture in the end product. Figure 3b, illustrate that packed density increased from 0.45 to 0.46 g/cc with increase in feed pump capacity from 9 to 21 % due to increase in moisture content of end product. Packed density increase from 0.38 to 0.44 g/cc with increase in encapsulating materials from 4 to 12 % due to increase the weight of end product.

Hygroscopicity of powder

The variation is explained by a polynomial equation of second order. The equation in coded value generated by multiple regression analysis using CCRD reads as follows:

$$\begin{aligned} \text{Hygroscopicity} = & 0.19 + 0.042\mathbf{X}_1 - 0.00916\mathbf{X}_2 - 0.000833\mathbf{X}_3 \\ & + 0.00166\mathbf{X}_4 - 0.0025\mathbf{X}_1\mathbf{X}_2 - 0.00125\mathbf{X}_1\mathbf{X}_3 \\ & + 0.000\mathbf{X}_1\mathbf{X}_4 - 0.00125\mathbf{X}_2\mathbf{X}_3 + .000\mathbf{X}_2\mathbf{X}_4 \\ & + 0.00125\mathbf{X}_3\mathbf{X}_4 + 0.001042\mathbf{X}_1^2 \\ & - 0.000208\mathbf{X}_2^2 + 0.00354\mathbf{X}_3^2 + 0.00354\mathbf{X}_4^2 \end{aligned}$$

Fig. 3 Effect of (a) Inlet Air Temperature and Aspirator Blower capacity, (b) Feed Pump Capacity and Encapsulating Material on Packed Density (a, b) and Hygroscopicity (c, d) of Ber juice Powder at constant atomization pressure (1.5 bar) and TSS of feed (7.5°Brix)

The coefficient of determination R^2 was 0.94 showing 94 % variability in the data. The analysis of variance (ANOVA) for model 4 was revealed that adequacy of fit 'F' value (15.87) more than unity, which is clearly significant.

Figure 3c, also depicts that increase in inlet air temperature from 170 to 210 °C, increases the hygroscopicity from 0.12 to 0.26 g/g drying matter, this may be because increasing the drying air temperature generally produces low moisture content in finished product resulting in an increase in the hygroscopicity. Hygroscopicity decreased from 0.22 to 0.15 g/g drying matter with increase in aspirator blower capacity from 40 to 80 % due to a high aspirator speed means a shorter residence time in the device and results in a larger amount of residual moisture in end product resulting in decrease in the hygroscopicity. It can be observed from Fig. 3d, hygroscopicity decreased negligibly from 0.19 to 0.18 g/g drying matter with increase in feed pump capacity from 9 to 21 % meaning thereby that there was no effect of variation of feed pump capacity on hygroscopicity of powder. Increase in encapsulating materials 4 to 12 % resulting hygroscopicity 0.19 g/g drying matter means there was no effect of variation of encapsulating material on hygroscopicity of powder.

Colour of powder

The colour of the spray dried *Ber* powder on L^* , a^* and b^* colour scale was observed. The values varied for " L^* " from 53.69 to 65.43, " a^* " from 6.45 to 9.89 and " b^* " from 18.23 to 24.56 which are presented in Table 3.

It can be observed from Table 3 that the whiteness in the samples vary between average i.e. 53.69 to good i.e. 65.43 where as all the samples are positively red. Similarly all the samples are yellowish with yellow colour varying between 18.23 to 24.56 on L^* , a^* and b^* colour scale. Whiteness of the samples decrease on the L^* , a^* and b^* colour values from 65.43 to 53.69 with the increase drying temperature, from 170 °C to 210 °C. The table further reveals that the *Ber* powder is "yellowish red" as the values are positive L^* , a^* and b^* colour scale. The maximum change in the red colour is brought at inlet air temperature 200 °C, aspirator blower capacity 50 %, feed pump capacity 12% and encapsulating material 10 %. However the minimum change in the red colour is brought at inlet air temperature 190 °C, aspirator blower capacity 80 %, feed pump capacity 15% and encapsulating material 8 %. The maximum change in the yellow colour is brought at inlet air temperature 190 °C, aspirator blower capacity 60 %, feed pump capacity 15% and encapsulating material 4 %. However the minimum change in the red colour is brought at inlet air temperature 180 °C, aspirator blower capacity 70 %, feed pump capacity 12% and encapsulating material 6 %. Therefore the colour of spray dried *Ber* powder is "whitish yellow-red". The best colour

adjudged by panel of judges during sensory evaluation with overall acceptability score was maximum 7.92 at the inlet air temperature 190 °C, aspirator blower capacity 80 %, feed pump capacity 15% and encapsulating material 8 % is "whitish yellow-red".

Optimization of process parameters through Response surface methodology on the basis of response parameters like, if required maximum packed density and minimum hygroscopicity of *Ber* powder then process parameters would be inlet air temperature 194.98 °C, aspirator blower capacity 65 %, feed pump capacity 15.2 % and encapsulating material 7 %. It can be also optimized for others conditions.

Conclusion

In this study the effect of operational parameters of spray dryer i.e. inlet temperature (170, 180, 190, 200 and 210 °C), aspirator blower capacity (40, 50, 60, 70 and 80 %) and feed pump capacity (9, 12, 15, 18, and 21 %) and processing parameters of feed i.e. total soluble solid (TSS) of feed (7.5 %) and encapsulating material, maltodextrin (4, 6, 8, 10, and 12 %), aerosil (1.0 %), citric acid (0.25 %) have been conducted for physical properties (Colour, packed density and hygroscopicity) of powder were observed. After complete evaluation and analysis of all the attributes for physical properties i.e. colour, packed density (0.45 g/cc and hygroscopicity (0.17 g/g drying matter), it was concluded that best quality of *Ber* powder were obtained at inlet air temperature 190 °C, aspirator blower capacity 60 %, feed pump capacity 15 %, encapsulating material 8 %.

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