### CHAPTER 8 DEVELOPMENT OF A CARAMBOLA POMACE FIBRE FORTIFIED MIX FRUIT BEVERAGE POWDER AND ITS CHARACTERIZATION

#### 8.1. Introduction

Fruits in their natural form are good sources of micronutrients and phytochemicals. But as most of the fruits are seasonal in nature, most of them are not available throughout the year. With the technological advancement in the food industries, many processing methods have developed to ensure the availability of the seasonal fruits in the form of processed products which are generally read-to-use. Unlike the fresh fruits which are highly perishable in nature and their transportation is a major problem for the suppliers, the processed products are more stable with increased shelf life and easy to transport and supply. The popular processed fruit products available in the market are different ready-todrink and ready-to-serve fruit juice drinks in addition to spray dried powders of some citrus fruits. Compared to other processed fruit drinks which are prepared by conventional pasteurisation, the products which were processed using high pressure processing, membrane processing or spray drying are more stable, compact in size and easy to transport. The spray dried products could be easily reconstituted before consumption and can be quite comparable to their original unprocessed counterpart.

With an increase in awareness for health among the consumers, there has been a rapid growth in the functional food segments. Researchers and food manufacturers are developing newer food products with health promoting properties. In the present time, low calorie breakfast cereals with added dietary fibre are the most popular products among the consumers in the functional food segments. Dietary fibre helps in the regulation of serum cholesterol and glucose level. It acts as a bulking agent; regulates energy intake; improves satiety; controls weight gain; and maintains health of the digestive system through its fermentation in the colon by the bacteria present that produces many secondary bioactive metabolites with health promoting properties.<sup>[1-4]</sup> The dietary fibres derived mainly from the fruit by-products are rich in polyphenols, carotenoids and minerals. <sup>[5-10]</sup> The carambola pomace derived fibre rich fractions have been reported to have serum cholesterol and

glucose lowering properties under *in vitro* and *in vivo* conditions. <sup>[11-13]</sup> However, till date their application in the development of a functional beverage has not been reported.

The use of dietary fibre as fortificant in cereal based functional food is very prevalent and well accepted. However, fibre fortification in fruit based products is very sparse. Some work has been reported on using fibre in fruit smoothie.<sup>[14, 15]</sup> Sun-Waterhouse<sup>[16]</sup>, reported a study on addition of polyphenols and fibre from apple to fruit smoothies. But as the dietary fibre, specifically insoluble dietary fibre is insoluble in water and therefore, their use in the beverage industry is very limited. This is mainly because, fortification of fibre into a liquid medium has many shortcomings and challenges like immiscibility, separation and sedimentation problems during storage which would ultimately affect the overall acceptability and appearance of the fortified juice product.<sup>[17-19]</sup> To overcome these problems, the manufacturers are then needed to use some techniques like microencapsulation, emulsification using natural emulsifiers or high pressure homogenisation. Another method is to use chemical additives which are not always preferred by the consumers who are looking for a natural healthy food product with functional properties.

In chapter 7, it has been reported that out of the six fibre sources that were studied for their functional properties, only carambola pomace fibre was found to have good TPC content; high water- and oil-holding capacities and other functional properties; and good color. The reported results indicated that carambola pomace could be used as a source of fibre to develop the functional fruit beverage.

Therefore, considering the above aspects, a study was carried out to optimize the development of a functional mix fruit beverage powder fortified with fibre rich fraction of carambola pomace using response surface methodology and study the quality of the obtained powder product. This chapter has the report of the detailed study.

#### 8.2. Materials and methods

All the chemicals used were of analytical grade and supplied by Merck, India, Himedia Laboratories and Sigma Aldrich, India.

#### 8.2.1. Materials

The fruits samples viz. carambola (Averrhoa carambola L), watermelon (Citrullus lanatus var lanatus), and pineapple (Anona sativas L. Merr) were procured from the local fruit market, Tezpur, Assam during the season.

#### 8.2.2. Fruit juice mix preparation for spray drying

The fruit samples were washed and sorted properly and the juice was extracted using a household juicer (Philips). The juice was strained through a muslin cloth and kept aside. The juice mix was obtained by blending the fruit juice in the ratio 90:5:5 (pineapple: carambola: watermelon). This was obtained after initial trials based on taste. To the mixed juice, 10% of fibre rich carambola pomace was added along with maltodextrin ( $\leq$  20 DE, Himedia) in the required amount (15-35%) and homogenized (UltraTurex 25, IKA). After adding the different concentrations of maltodextrin to the mixed juice fibre, the final °Brix of all the feed mixtures were diluted to 10°B to maintain a constant feed flow rate.

The total solid content or °Brix was measured using a portable refractometer, (0-32%, Erma, Japan).

#### 8.2.3. Viscosity and pH of the mix fruit juice fibre-maltodextrin feed sample at 10°B

Viscosity of the feed sample was measured by a viscometer (LabTech, LTT30) at 30°C and 30 rpm using spindle no.2. pH was measured using an electronic pH meter (Eutech, Merck) at 27°C.

#### 8.2.4. Spray drying of the mix fruit juice fibre-maltodextrin

The homogenized mix fruit juice fibre-maltodextrin feed mixture at different concentration of maltodextrin was spray dried at an inlet temperature of 165 °C - 185 °C in a laboratory scale spray drier ((Lab plant system, UK). The feed rate was maintained at 7 mL/min and the nozzle size of the atomizer was 1mm. The obtained powder was kept in an airtight container and stored at room temperature for the various analyses.

### 8.2.5. Experimental design for optimization using central composite rotatable design (CCRD) by response surface methodology (RSM)

For the optimization of the spray drying process, CCRD model was applied. Two independent variables were taken viz., maltodextrin concentration and inlet temperature at 5 levels (Table 8.1). The CCD consisted of 13 experiments including 5 centers, 4 axial and 4

factorial points. The design independent variables were maltodextrin concentration  $(X_1, \%)$  and inlet temperature  $(X_2, °C)$  while, the dependent or response variables were moisture content, yield, solubility, bulk density, and hygroscopicity.

Experimental data obtained were fitted into a second order polynomial model. The generalized second order polynomial order equation used was:

$$Y_{1} = a_{0} + a_{1}X_{1} + a_{2}X_{2} + a_{11}X_{1}^{2} + a_{22}X_{2}^{2} + a_{12}X_{1}X_{2}$$
 Eq. 8.1

Where,  $Y_1$  (i= 1-3) is predicted response for moisture content, yield, solubility, bulk density, and hygroscopicity. The  $a_0$  is the fitted response at the center point;  $a_1$  and  $a_2$  are linear terms;  $a_{12}$  is the interaction effect,  $a_{11}$  and  $a_{22}$  are squared effects.  $X_1$  and  $X_2$  are the independent variables.

Design expert 6.0 software was used to generate response surfaces and the plot. The data were statistically analyzed by ANOVA. The p values of  $\leq 0.01$  were considered to be statistically significant.

#### 8.2.6. Determination of the response variables

#### **8.2.6.1.** *Moisture content*

Moisture content was determined based on AOAC method.<sup>[20]</sup> The spray dried sample was taken in a previously dried and weighed covered dish. The sample was allowed to dry in a hot air oven (Jiotech, South Korea) at 105°C for 8 h till a constant weight was attained. The final weight of the dish containing the sample was measured both before and after drying and moisture content was calculated.

Moisture content (%) = 
$$\frac{W_1 - W_2}{W_2} \times 100$$
 Eq. 8.2

Where,  $W_1$  is the weight of the sample with the dish before drying;  $W_2$  is the final weight of the sample with dish after drying

#### 8.2.6.2. Yield of powder

The yield of the spray drying process was calculated by taking into consideration the total solid content of the feed sample with maltodextrin and weight of the final dry powder.

Yield (%) = 
$$\frac{Weight of solids dried powder (g)}{Solid content of the feed material (°B)} \times 100$$
 Eq. 8.3

#### 8.2.6.3. Solubility

The solubility was determined according to the method described by Chau et al.<sup>[21]</sup> Briefly, samples were mixed with distilled water (1:10 w/v), stirred for 1 h at room temperature and centrifuged at 1500 rpm for 10 min. The supernatant was collected, dried and weighed.

Solubility (%) = 
$$\frac{Weight (g) of supernatant after drying}{Weight (g) of sample} \times 100$$
 Eq. 8.4

#### 8.2.6.4. Bulk density

The bulk density and tapped density were calculated by weighing 1g of sample powder into a graduated 10 mL cylinder and measuring the volume occupied by the sample.<sup>[22]</sup> The results are expressed as g/mL.

Bulk density 
$$(g/mL) = \frac{Weight of sample}{Volume occupied}$$
 Eq. 8.5

#### 8.2.6.5. Hygroscopicity

The hygroscopicity property of the sample powders was determined according to Cai and Corke <sup>[23]</sup> with some modifications. Briefly, 2 g of spray dried powder samples were placed in pre-weighed glass vials and placed in a desiccator containing saturated salt solution of sodium chloride (relative humidity of 75.09 %) maintained at 30°C and kept for 7 days. After the incubation period, sample vials were weighed and hygroscopicity was expressed as expressed as g moisture per 100 g solids.

# 8.2.7. Characterization of the obtained spray dried fibre fortified mix fruit beverage powder

#### 8.2.7.1. Determination of proximate content.

#### 8.2.7.1.1. Moisture content

It was determined by the method as mentioned in 8.2.6.1.

#### 8.2.7.1.2. Crude protein content

The crude protein content was determined by modified Micro-Kjeldahl method of AOAC <sup>[20]</sup> using a digestion and distillation system (KelPlus, Pelican Equipment, Chennai, India). Briefly, 250 mg of the fibre sample was weighed and then digested with 10 mL concentrated sulphuric acid in a digestion tube at 350°C till a bluish green colour appeared.

The digested sample was then distilled using 40 mL of 40 M sodium hydroxide with 20% of 20 mL boric acid with 1-2 drops of methyl red indicator. During distillation the ammonium vapor produced is passed over the boric acid solution which results in change of its colour from pink to light yellow. After completion of the distillation process the distillate was titrated against 0.1N hydrochloric acid and the end point was noted when a light pink colour appeared. The nitrogen content of the sample was calculated. The crude protein was calculated by multiplying a factor of 6.25 with nitrogen content.

Nitrogen content (%, db) = 
$$\frac{S - B \times normality \ of \ HCL \times 14 \times 100}{Weight \ of \ sample \times 1000}$$
 Eq. 8.6

Where, S is the titre value of sample and B is the titre value of blank

Crude protein content (%, db) = Nitrogen content (%)  $\times$  6.25 Eq. 8.7

#### 8.2.7.1.3. Crude lipid content

The crude lipid content was determined as per AOAC method <sup>[20]</sup> using a Soxhlet apparatus (Socs Plus, Pelican Equipment, India). Briefly, 2 g of sample was weighed and extracted with petroleum ether (60°C-80°C boiling range) for 2 h at 100°C and recovered for 2 h at 200°C in a pre-weighed glass jar. Once the recovery process was over, the glass jars were put into a hot air oven at 105°C to remove any trace of moisture, cooled in a desiccator and then the final weight was measured. The total fat content was calculated.

Crude lipid content (%, db) = 
$$\frac{W_2 - W_1}{S} \times 100$$
 Eq. 8.8

Where,  $W_1$  is the initial weight of the glass jar;  $W_2$  is the final weight of the glass jar; S is the weight of the sample.

#### 8.2.7.1.4. Ash content

The ash content was determined by AOAC method <sup>[20]</sup>. Briefly, 5g of sample was weighed into a pre-weighed sintered crucible ( $W_1$ ), charred on a hot plate and ashed at 625°C for 6 h in a muffle furnace (Labtech). The ashed sample with the crucible was then cooled in a desiccator and the final weight of the crucible ( $W_2$ ) was measured and the total ash content was calculated.

Ash content (%) = 
$$\frac{W_2 - W_1}{S} \times 100$$
 Eq. 8.9

Where,  $W_1$  is the initial weight of the empty crucible;  $W_2$  is the final weight of the crucible after ashing; S is the weight of the sample.

#### 8.2.7.1.5. Total reducing sugar

The total reducing sugar was measured by Nelson Somogyi method.<sup>[24]</sup> The alkaline copper taratarate solution was prepared mixing 4 mL of solution A and 96 mL of solution B. The solution A was made by dissolving 2.5 g anhydrous sodium carbonate, 2.0 g sodium carbonate, 2.5 g potassium sodium tartarate and 20 g anhydrous sodium sulphate in 80 mL of distilled water and the volume was made up to 100 mL. Similarly, the solution B was prepared by dissolving 15.0 g copper sulphate in distilled water. To this solution 1 drop of concentrated suphuric acid was added and volume was made up to 100 mL. For the preparation of the arsenomolybdate reagent, 2.5 g of ammonium molybdate in 45 mL distilled water was dissolved. Then added 2.5 mL concentrated sulphuric acid and mixed well. After that 0.3 g disodium hydrogen arsenate was dissolved in 25 mL distilled water. Both the solution was mixed well and incubated at 13°C for 24 h. A standard glucose solution (100  $\mu$ g/mL) was prepared for obtaining the calibration curve.

Sample aliquot of 0.1 mL was taken in a test tube and volume made up to 2mL with distilled water. Then added 1 mL of alkaline copper tartarate solution and placed in a boiling water bath for 10 min. After 10 min the test tubes were cooled and then 1 mL of arsenomolybic acid reagent was added to the tubes. The final volume up to 10mL was made up with distilled water and after an incubation period of 10 min, the absorbance of the test samples was taken at 620 nm. The reducing sugar concentration was calculated from standard glucose calibration graph.

#### 8.2.7.1.6. Total dietary fibre

The dietary fibre estimation was done by an enzymatic gravimetric method. <sup>[20]</sup> The beverage powder (1 g) was homogenized in 40 mL of 0.05 M 2-(*N*-morpholino) ethanesulfonic acid (MES) buffer (pH 8.2), followed by the addition of 50  $\mu$ L heat stable  $\alpha$ -amylase (product code A 3306, termamyl, Sigma) and was incubated at 95°C for 15 min with occasional stirring. The contents were cooled to 60°C and rinsed with 10 mL of water. Then added 100  $\mu$ L of protease (product code P 3910, Sigma) and incubated at 60°C for 30 min. Then 5mL 0.561 M hydrochloric acid was added into the beakers while stirring. The

pH was adjusted to 4-4.7 at 60°C, by adding 1 M hydrochloric acid. Lastly, 300  $\mu$ L of amyloglucosidase (product code A 9913, Sigma) was added and incubated for another 30 min at 60°C with constant stirring. To the digested test solution, 225 mL 95% ethanol was added at 60°C. The ratio of ethanol to test solution was kept at 4:1. The digested solution and ethanol mixture was allowed to precipitate at room temperature for 1 h. The precipitate was collected by vacumm filtration through a dried and weighed crucible containing 0.5 g of celite. (Drying and weighing of crucible was done by adding 0.5 g of celite into the crucible and washing it with 20 mL of 95% ethanol and 20 mL acetone. It was then dried in oven at 105°C for 30 min and weighed).

The residue retained on the crucible was then washed with 20 mL of 95% ethanol and 20 ml of acetone, the crucible was dried at 105°C overnight and the final weight was recorded. Total dietary fibre was calculated by the given equation:

TDF (%) = 
$$\frac{R_1 + R_2/2 - P - A - B}{M_1 + M_2/2} \times 100$$
 Eq. 8.10

Where,  $R_1$  and  $R_2$  are the residue weight of the duplicate test samples;  $M_1$  and  $M_2$  are the weight of the test samples in duplicate; P is the weight of protein; A is the weight of ash content; and B is the test blank

# 8.2.7.2. Color comparison of the reconstituted beverage with the feed solution before drying

Color values (L, a, b) were measured using a Hunter color spectrophotometer (Hunter Color Lab UltrascanVis). The 'L' value indicates degree of lightness. 'L' value in the range between 0-50 indicates dark and 51-100 indicates light. Similarly, 'a' means measure of red (positive values) and green color (negative values); 'b' measures the yellow (positive value) or blue (negative values) colors <sup>[25]</sup>. The color change of the samples was determined by comparing the L, a, b values of the reconstituted samples with that of the fresh feed sample just before spray drying. The quantity required (water/g) of the powder samples for the reconstitution was calculated to obtain 10°B. The overall color change ( $\Delta E$ ) of the samples was calculated. <sup>[26]</sup>

$$\Delta E = \sqrt{(L_0^* - L_0)^2 + (a_0^* - a)^2 + (b_0^* - b)^2}$$
 Eq. 8.11

Where,  $\triangle E$  is the overall change in colour;  $L_0^*$  is the 'L' value of fresh feed;  $L_0$  is the 'L' value of reconstituted sample;  $a_0^*$  the 'a' value of fresh feed;  $a_0$  is the 'a' value of reconstituted sample;  $b_0^*$  is the 'b' value of fresh feed; and  $b_0$  is the 'b' value of reconstituted sample.

#### 8.2.7.3. Particle size distribution

The particle size distribution was determined using a particle size analyzer (NanoPlus zeta potential & particle size analyzer, Particulate Systems). The particle size was analyzed based on the principle that when laser beams are irradiated to particles under the Brownian motion, scattered light from the particles shows fluctuation corresponding to individual particles. The fluctuation is observed according to the pinhole type photon detection method, so that particle size and particle size distributions are calculated. A small powder sample was suspended in water and analyzed at 25°C. The particle size ( $\mu$ M) was depicted with respect to its intensity (%) while particle size distribution was represented by calculating Span.

Span = 
$$\frac{D_{90} - D_{10}}{D_{50}}$$
 Eq. 8.12

Where,  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  are the diameters of sample at the 10th, 50th, and 90th percentiles.

# 8.2.7.4. Bulk density (BD), tapped density (TD), Hausner's ratio (HR) and Carr Index (CI)

Bulk density was measured as given in 8.2.6.4. For tapped density, 1 g of sample was taken in a cylinder and then it was tapped manually for 50 times and the volume occupied by the sample was noted. <sup>[22]</sup>

From the bulk and tapped density values, the Hausner's ratio (HR) and Carr index (CI) were calculated to determine the cohesiveness and flowability property of the powder samples. <sup>[27, 28]</sup> Based on the values of HR and CI (Table 8.1), the flowability and cohesiveness of the sample powders were classified. <sup>[29]</sup>

$$HR = \frac{TD}{BD}$$

$$CI = \frac{TD - BD}{TD} \times 100$$
Eq. 8.13

Hausner's ratio	Cohesiveness	
<1.2	Low	
1.2-1.4	Intermediate	
>1.4	High	
Carr Index (%)	Flowability	
<15	Very good	
15-20	Good	
20-30	Fair	
35-45	Bad	
>45	Very bad	

**Table 8.1.** Classification of powder flowability based on Carr index (CI) and powder cohesiveness based on Hausner ratio (HR)

#### 8.2.7.5. Dissolution test and pH of the beverage powder

Dissolution test of the beverage powder was done by dissolving the sample powder in distilled water and the time taken to completely dissolve the powder in the water was recorded. pH of the sample was measured using a pH meter (Eutech, Merck). Briefly, 1g of sample was dissolved in 5 mL deionised water and pH was measured at 27°C.

#### 8.2.7.6. Water activity

The water activity of the powdered samples was measured using a water activity meter (Aqua LAB, Dew Point, 4TE) at 25°C.

### 8.2.7.7. Surface morphology of the spray dried powder by scanning electron microscopy (SEM)

The powder samples, prior to SEM observation, samples were mounted on stubs with double-sided adhesive tape and coated with a thin layer of gold. The SEM images were then obtained using a JSM-6390LV scanning electron microscope (JEOL, Japan) at 15 kV at 2000x magnification.

#### 8.2.7.8. Sensory analysis using 9-point Hedonic scale

The sensory profiling of the developed beverage powder was done using 9-point Hedonic scale. There were 11 panel members and each member was given the original fresh mix fruit juice and reconstituted beverage coded as A and B, respectively. The panel members then rated the two samples for color, odour, taste, aroma, appearance and overall acceptability using the 9-point scale.

1=dislike extremely	6= like slightly
2= dislike very much	7= like moderately
3= dislike moderately	8= like very much
4= dislike slightly	9= like extremely
5= neither like nor dislike	

#### 8.2.7.9. Statistical analysis

All experiments were carried out at least in triplicates and reported as mean  $\pm$  standard deviation of mean (S.E.M). The optimization data were analyzed by ANOVA using Design Expert 6.0 software. The 'L', 'a', 'b' values were subjected to paired-comparison t-test ( $p \le 0.05$ ).

#### 8.3. Results and discussion

#### 8.3.1. Optimization and fitting of the model

RSM was applied to determine the effect of maltodextrin concentration and inlet temperature on responses for moisture content, yield, solubility, bulk density, and hygroscopicity. The results of the experiments performed for CCRD of the variables along with the responses are given in Table 8.2. The ANOVA results for the model responses are presented in Table 8.3. For the good fit of a model, the  $R^2$  value should be 0.80.<sup>[30]</sup> In the present study,  $R^2$  values for the three responses were higher than 0.80 which implied the adequacy of the applied regression model. The lack of fit for all fitted models was found to be not significant (p>0.05). The lack of fit measures the failure of the model to represent data in the experimental domain at points which are not included in the regression.<sup>[31]</sup> Therefore, it can be assumed that the selected model can be used for the optimization of variables and development of the spray dried fibre fortified mix fruit beverage powder.

### 8.3.2. Effect of independent variables on responses in the spray dried beverage powder 8.3.2.1. *Moisture content*

From the response plot (Fig.8.1a), it can be inferred that maltodextrin alone or in interaction with inlet temperature had a positive effect. The regression equation obtained for moisture content is given below.

$$Y = +66.44 + 0.42X_1 - 1.40X_2 + 1.08X_1^2 + 0.32X_2^2 + 1.13X_1X_2$$
 Eq. 8.15

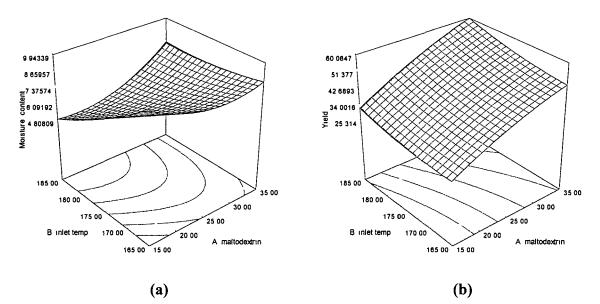


Fig 8.1. 3D plot of the response variables (a) moisture content and (b) yield

#### 8.3.2.2. Yield

From the response plot (Fig.8.1b), it showed that increase in temperature and maltodextrin concentration increased the yield value. The regression equation obtained for yield is given below.

$$Y = +42.07 + 1291X_1 + 4.45X_2 - 2.37X_1^2 + 1.81X_2^2 + 1.18X_1X_2$$
 Eq. 8.16  
8.3.2.3. Solubility

From the response plot (Fig.8.2a), it can be suggested that with increase in inlet temperature and maltodextrin, the solubility increases. The regression equation obtained for solubility is given below.

$$Y = +75.10 + 9.08X_1 + 2.02X_2 + 0.22X_1^2 + 3.79X_2^2 + 0.33 X_1X_2$$
 Eq. 8.17

Sl.no.	Maltodextrin X <sub>1</sub> (%)	Inlet Temperature X2(°C)	Moisture Content (%, wb)	Yield (%)	Solubility (%)	Bulk density (g/mL)	Hygroscopicity (g/100g)
1	15.00 (-1)	165.00 (-1)	9.71±0.11	25.72±0.18	68.18±0.15	0.389±0.09	14.89±0.10
2	25.00 (0)	175.00 (0)	6.51 ±0.07	41.83±0.17	74.51±0.13	0.413±0.06	14.63±0.14
3	25.00 (0)	175.00 (0)	6.52±0.14	42.78±0.19	74.89±0.19	0.417±0.03	14.54±0.09
4	25.00 (0)	175.00 (0)	6.75±0.18	41.86±0.07	77.2±0.09	0.456±0.06	14.62±0.22
5	10.86 (-1.41)	175.00 (0)	8.07±0.04	19.57±0.23	63.94±0.11	0.351±0.06	15. <b>89±0</b> .17
6	25.00 (0)	175.00 (0)	6.70±0.11	41.04±0.29	74.78±0.18	0.422±0.03	14.33±0.12
7	35.00 (+1)	165.00 (-1)	8.62±0.16	49.12±0.11	86.39±0.13	0.386±0.08	11.99±0.10
8	15.00 (-1)	185.00 (+1)	5.12±0.09	30.45±0.13	69.46±0.17	0.286±0.07	14.69±0.17
9	35.00 (+1)	185.00 (+1)	8.55±0.03	58.49±0.11	87.8±0.15	0.291±0.13	10.50±0.09
10	39.14 (+1.41)	175.00 (0)	8.79±0.16	56.30±0.19	89.44±0.08	0.417±0.05	10.10±0.06
11	25.00 (0)	189.14(+1.41)	4.62±0.12	53.87±0.12	88.62±0.18	0.239±0.02	12.59±011
12	25.00 (0)	175.00 (0)	5.77±0.09	42.86±0.18	74.13±0.11	0.419±0.07	14.61±0.09
13	25.00 (0)	160.86 (-1.41)	9.22±0.19	38.7±0.23	79.07±0.05	0.396±0.06	13.22±0.05

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 Table 8.2. Independent variables and the response variables

\*Mean ± S.D of triplicate values

	DF	Moisture Content	Yield	Solubility	Bulk density	Hygroscopicity
Model	5	30.39	1568.93	792.55	0.048	37.94
Lack of fit	3	0.86	9.46	25.32	1.440E-003	0.24
Pure error	4	0.69	2.29	5.85	1.213E-003	0.063
R <sup>2</sup>	-	0.9515	0.9926	0.9622	0.9474	.9921

Table 8.3. ANOVA values for the fitted models and lack of fit

#### 8.3.2.4. Bulk density

From the response plot (Fig.8.2 b), a slight decrease in the bulk density of the powder was observed. The regression equation obtained for bulk density is given below.  $Y = 0.43+0.012X_1-0.053X_2-0.024X_1^2-0.057X_2^2-0.002X_1X_2$  Eq. 8.18

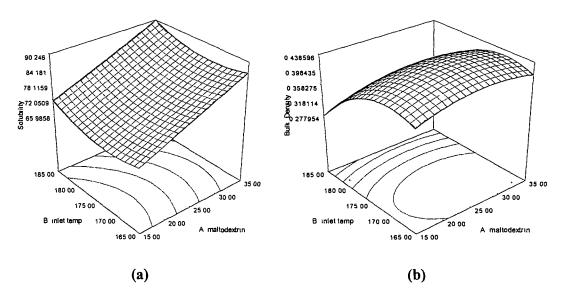


Fig 8.2. 3D plot of the response variables (a) solubility and (b) bulk density

#### 8.3.2.5. Hygroscopicity

From the response plot (Fig.8.3), increase in maltodextrin decreased the hygroscopicity of the powder while no effect of inlet temperature was observed. The regression equation obtained for hygroscopicity is given below.

$$Y = +14.55 - 1.91X_1 - 0.32X_2 - 0.76X_1^2 - 0.80X_2^2 - 0.32X_1X_2$$
 Eq. 8.19

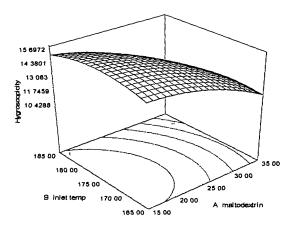


Fig 8.3. 3D plot of the response variable hygroscopicity

Inlet temperature and maltodextrin concentration influenced the moisture content, bulk density, and hygroscopicity. The decrease in moisture content with increase in temperature may be due to greater temperature gradient between the atomized feed particles and the hot air inside the chamber of the spray drier. The large difference in temperature gradient ensures better evaporation efficiency of the droplets.<sup>[32]</sup> Similar results had been reported during spray drying of anthocyanin of black carrot. <sup>[33]</sup> Likewise, increase in maltodextrin increases the total soluble solid content of the feed and thus decrease the moisture content. Increase in temperature also increases the yield of the powder due to greater heat and mass transfer efficiency.<sup>[23,34,35]</sup> The solubility increases with increased maltodextrin to encapsulate feed droplets and this affects the surface stickiness of the particles due to transformation into glassy state during drying. This change in surface stickiness helps in reduction of the agglomerate formation.<sup>[37]</sup>

Increase in temperature and maltodextrin concentration decreases the bulk density of powder. The increase in maltodextrin helps in minimizing the thermoplastic particles from sticking together. In addition to that, the increase in volume of air trapped in the particles may occur with increase in maltodextrin concentration.<sup>[38]</sup>

Hygroscopicity is however, inversely related to temperature. This is because; at high temperature generally low moisture content is obtained. With decrease in moisture content, the capacity to absorb moisture by the powder sample from its ambient surrounding increases. This is due to greater water concentration gradient between the powder and the surrounding. Thus, this phenomenon leads to an increase in hygroscopicity. <sup>[34, 39]</sup> However, increase in maltodextrin decreases the hygroscopicity of the powder. <sup>[40]</sup>

#### 8.3.3. Verification of the predictive model

The suitability of the model for the responses was determined under the optimum conditions of inlet temperature (175°C) and maltodextrin concentration (25%). The experimental values for the responses were found to be quite comparable and in agreement with that of the predicted value (Table 8.4).

Responses	Predicted value	Actual value
Moisture content (%)	6.44	6.12±0.23
Yield (%)	42.07	41.89±0.34
Solubility (%)	75.10	74.57±0.11
Bulk density (g/mL)	0.4254	0.416±0.08
Hygroscopicity (g/100g)	14.55	14.38±0.17

Table 8.4. Responses, predicted and actual values of the optimized model

#### 8.3.4. pH, total solid content and viscosity of the feed sample

The pH value of the feed sample was 3.62±0.08. Similarly, the total solid content of the feed sample was 30°B and the viscosity of the samples (10°B) at the time of spray drying was 33.50 mPa.s.

### 8.3.5. Proximate composition of the beverage powder and dissolution test, pH and water activity $(a_w)$ of the powder

The proximate composition of the beverage powder is presented in Table 8.5. The moisture content was 6.12%. The lipid content was very low and the final total dietary fibre in the sample powder was 9.29%. Similarly, the protein and ash content were 1.75% and 1.32%, respectively. The reducing sugar content in the powder sample was 9.97%.

The time taken for dissolution of the beverage powder in distilled water was  $48.52\pm0.31$ s while, pH value of the beverage powder was  $3.52\pm0.04$ . The water activity (a<sub>w</sub>) of the obtained powder was 0.34. Generally, a<sub>w</sub> < 0.6 implies that the powder is relatively stable from microbial spoilage. This is because, a<sub>w</sub> measures the amount of free

water available in the powder sample that can be used by the microbes for different biochemical reactions.<sup>[32, 40]</sup>

Moisture	Protein	Lipid	Ash	Reducing	Total
content	content	content	content	sugar	dietary fibre
6.12±0.23	1.75±0.14	0.273±0.09	1.32±0.07	9.97±0.17	9.29±0.19

Table 8.5. Proximate composition of the beverage powder (%, db)

\*Mean ±S.D. of triplicates data

#### 8.3.6. Bulk density, tapped density and flow property of the powder

The bulk density (Table 8.6) of the sample was 0.416 g/mL and the tapped density was 0.506 g/mL. The Hausner's ratio ranged between 1.21, while Carr index range was 17.79%. According to the classification of powder, cohesiveness and flowability mentioned in Table 8.1 <sup>[30]</sup>, the beverage powder had intermediate cohesiveness whereas flowability property as per Carr index value was relatively good.

Table 8.6. Bulk density, tapped density and flow property of the beverage powder

Bulk density	Tappe density	Hausner's	Carr
(g/mL)	(g/mL)	Ratio	Index
0.416±0.08	0.506±0.03	1.21±0.07	17.79±0.02

\*Mean ±S.D. of triplicates data

### 8.3.7. Change in color L a b values when the reconstituted beverage was compared to the fresh feed sample

The color comparison for 'L' and 'b' values between feed and reconstituted samples showed significant difference ( $p \le 0.05$ ) during paired t-test (Table 8.7). Increased 'L' values were observed in reconstituted samples while no significant change was observed in the 'a' value. Similarly, significant increase in 'b' value was observed for the reconstituted samples. Overall color change ( $\Delta E$ ) value was 6.47±0.09.

L		а	!	b		
FS	RS	FS	RS	FS	RS	△E
22.76±0.17*	29.02±0.11*	-0.07±0.01	-0.0.05±0.02	-0.24±0.02*	1.55±0.12*	6.47±0.09

**Table 8.7.** Color,  $L \ a \ b$  values and overall colour ( $\triangle E$ ) difference of the reconstituted beverage and fresh feed sample

\*Statistically significant difference at p≤0.05 during paired t-test between FS and RS values. # FS denotes feed sample; RS denotes reconstituted sample

#### 8.3.8. Particle size distribution and Span

Particle size analysis (Fig.8.4.) showed that the beverage powder size was distributed within the range 0.90-1.50  $\mu$ M and 43.50-102.00  $\mu$ M. The span value for the sample was 1.55±0.09. A smaller span value indicates a narrower particle size distribution of the powder sample. The possible reason could be low viscosity value of the feed which leads to formation of smaller particles. <sup>[41]</sup>

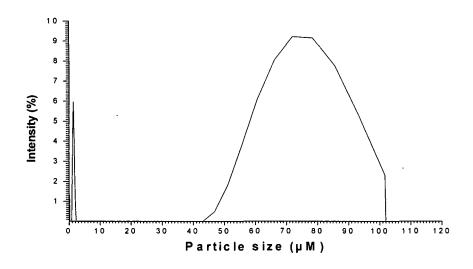


Fig. 8.4. Particle size distribution of the beverage powder

#### 8.3.9. Surface morphology of the beverage powder

The surface morphology study of the dried powder material is represented in Fig.8.5. Both small spherical and large ellipsoidal particles were observed at 2000 x magnification.

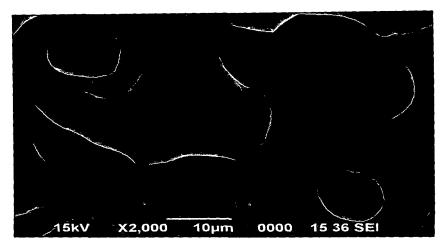


Fig. 8.5. SEM of spray dried powder mix fruit juice and carambola pomace

#### 8.3.10. Sensory analysis results of the reconstituted beverage powder

The 9 point Hedonic scale sensory analysis by the panel members showed overall acceptability of 7 for the reconstituted beverage which meant it was liked moderately (Table 8.8). Similarly, the other parameters like color, appearance and aroma were also liked moderately when compared with that of the fresh mixed juice beverage. However, both the sample showed same Hedonic value of 8 for taste.

Parameters	Fresh mixed juice	Reconstituted from	
		beverage powder	
Color	8	7	
Appearance	8	7	
Aroma	6	7	
Taste	8	8	
Overall acceptability	8	7	

Table 8.8. Sensory analysis of beverage using 9 point Hedonic scale

#### 8.4. Conclusion

The mixed fruit juice and fibre drink was optimized at inlet temperature of 175°C and 25% maltodextrin concentration. The actual values for the response variables for the optimum conditions were in tandem with that of the predicted values. The final product contained 9.29% of total dietary fibre. The time taken for dissolution of the beverage powder in distilled water was 48.52±0.31s. Beverage powder size range was distributed

in the range of 0.90-1.50 $\mu$ M and 43.50-102.00  $\mu$ M. The Span value for the sample was 1.55±0.09. The beverage powder had intermediate cohesiveness whereas flowability property as per Carr index value was relatively good. The color comparison for 'L' and 'b' values between feed and reconstituted samples showed significant difference (p≤0.05) during paired t-test. Overall color change ( $\Delta E$ ) value was 6.47±0. The sensory analysis by 9 point hedonic scale showed overall acceptability of the product at 7 (liked moderately). It can be concluded that, the proposed optimization model of CCRD through RSM could be used for the development of a functional mix fruit beverage powder fortified with fibre rich fraction of carambola pomace.

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