

**EFFECT OF SPRAY DRYING OF FOUR FRUIT JUICES ON  
PHYSICOCHEMICAL, PHYTOCHEMICAL AND ANTIOXIDANT PROPERTIES**

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**4.1. Introduction**

Fruits are rich sources of vitamins, minerals and bioactive compounds. Daily consumption of fruits is recommended mainly for their positive health benefits. <sup>[1, 2]</sup> Although fruits are essential in the human diet, ensuring their availability throughout the season is not possible. In general, fruits are seasonal and highly perishable in nature. There are many processing and preservation techniques that can ensure the availability of fruits in its various processed forms. However, the techniques applied must maintain or enhance their nutritional quality. <sup>[3]</sup> Out of the various processing techniques, spray drying is a technique applied to increase the shelf life of the fruit. Spray drying uses a technique where the feed material along with the carrier material is atomized into fine droplets and these droplets are then dried quickly under high temperatures. At the end of the process a fine powder of the feed material is obtained. <sup>[4]</sup> In food industries, the manufacturers use the spray drying method to dry different fruit juices. The spray dried fruit powder provides more stability to the nutrients and other active substances present in the fruit. <sup>[5]</sup> Also, spray dried powder can be readily reconstituted, has low water activity and is suitable for transport and storage at room temperature. <sup>[6]</sup> This lowers the storage and transportation costs as compared to the raw fruits.

But one of the major drawbacks in spray drying of fruit juices is the issue of stickiness and flow problems of the powder. <sup>[7]</sup> These problems occur due to the presence of high levels of low molecular weight sugars and organic acids. This results in low glass transition temperatures ( $T_g$ ) of the dried powder. To overcome this, an adjunct or a carrier material like maltodextrin, gum, starch or gelatin is used as an additive to the feed material during drying. <sup>[8]</sup> Use of the additives increases the glass transition temperature and reduces the stickiness and hygroscopicity of the powder; thus positively affects the yield and efficiency. <sup>[9]</sup> Among the additives, maltodextrin is cheap, widely available and has high solubility in water with low viscosity and bland flavor. <sup>[9-11]</sup> and hence is commonly used as

a carrier material. According to Ferrari et al. <sup>[12]</sup>, maltodextrin is more efficient in protecting the flavor, colour and bioactive compounds under adverse surrounding conditions. Although spray drying is an efficient method that prolongs shelf life, ensures availability during the off seasons and gives value addition to the product, processing treatments can have both positive and negative effects on physicochemical, phytochemical and antioxidant properties. The effects in turn are dependent on the type, nutritional composition and bioactive content of the fruit.

Therefore, this chapter presents the results of the study conducted to determine the effect of spray drying on the physicochemical, phytochemical and antioxidant properties of the juices of four fruits viz. carambola (*Averrhoa carambola* L.), watermelon (*Citrullus lanatus*), Khasi mandarin orange (*Citrus reticulata* Blanco) and pineapple (*Anona comosus* L.Merr). The results are interpreted and discussed at the end of the chapter in the light of the reported studies.

## **4.2. Materials and methods**

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

### **4.2.1. Materials**

The fruit samples viz. carambola (*Averrhoa carambola* L.), watermelon (*Citrullus lanatus*), Khasi mandarin orange (*Citrus reticulata* Blanco) and pineapple (*Anona comosus* L.Merr) were procured from the local fruit market, Tezpur, Assam during the season. Khasi mandarin is a citrus species grown in the sub-mountainous tract along the Indo-Bangladesh border regions of the north-eastern part of India. The four fruits selected for this study were chosen from the thirteen studied fruits reported in chapter 3 based on their easy availability and suitability for juice extraction.

### **4.2.2. Fruit juice preparation**

The fruit samples were washed and sorted properly and the juice was extracted using a household juicer (Philips juicer). The juice was strained in a muslin cloth and then 20% maltodextrin ( $\leq 20$  DE, Himedia) was added and mixed well by homogenization ((UltraTurex 25, IKA). Initially, three different levels of maltodextrin were taken for spray drying viz. 15, 20 and 25% at 185°C inlet temperature. Out of the three concentrations, 20%

MD gave an average good yield and end product quality for all the four fruit juice samples. Tze et al. <sup>[13]</sup> used 20% maltodextrin in spray dried pitaya fruit powder. Similarly, Dailami <sup>[14]</sup> also reported the use of 20% maltodextrin for the production of dragon fruit powder. The maltodextrin mixed homogenized juice of each sample was separated into two lots, one lot was marked as unprocessed fresh juice sample and the other lot was taken for spray drying. Each of the unprocessed fresh juice with maltodextrin was analyzed for phytochemical and antioxidant properties. To ensure that the measurements of both fresh fruit juice and spray dried powder were done for the same quantity of material, the dry matter content of the sample was determined and the final values were expressed in dry weight basis.

#### **4.2.3. pH, total solid content and viscosity of the feed samples**

pH of the feed samples (unprocessed fresh juice with maltodextrin) was measured using a pH meter (Eutech, Merck) at 27°C. The total solid content of the feed samples was determined by measuring °Brix using a portable refractometer (0-32%). Viscosity of the feed sample maintained at 10°B after dilution with water was measured by a viscometer (LabTech, LTT30) at 30°C and 30 rpm using spindle no.2.

#### **4.2.4. Spray drying of the juice-maltodextrin mixture**

The juice-maltodextrin mixtures were spray dried at an inlet temperature of 185°C and outlet temperature maintained at 88°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 0.1 mm. The obtained powders were kept in airtight containers and stored at room temperature for various analyses.

#### **4.2.5. 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.

$$\text{Yield (\%)} = \frac{W_p}{F_s} \times 100 \quad \text{Eq. 4.1}$$

Where,  $W_p$  is the weight of the solids of dried powder and  $F_s$  is the solid content of the feed material.

## 4.2.6. Physicochemical properties of the spray dried powder

### 4.2.6.1. Moisture content

The moisture content of the prepared fruit powder samples were determined by AOAC<sup>[15]</sup> method. Briefly, 5 g of the fruit powder sample was taken in previously dried and weighed covered dishes. 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.

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_2} \times 100 \quad \text{Eq. 4.2}$$

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.

### 4.2.6.2. Bulk density, tapped density, Hausner's ratio and Carr index

The bulk density and tapped density were calculated by weighing 1 g of sample powder into a graduated 10 mL cylinder and measuring the volume occupied by the sample. For tapped density, the cylinder was tapped manually for 50 times and then the volume occupied by the sample was taken.<sup>[16]</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>[17, 18]</sup> Based on the values of HR and CI (Table 4.1), the flowability and cohesiveness of the sample powders were classified.<sup>[19]</sup>

$$\text{Hausner's ratio, HR} = \frac{TD}{BD} \quad \text{Eq. 4.3}$$

$$\text{Carr index, CI} = \frac{TD - BD}{TD} \times 100 \quad \text{Eq. 4.4}$$

Where, TD is tapped density and BD is bulk density

**Table 4.1.** Classification of powder flowability based on Carr index (CI) and powder cohesiveness based on Hausner ratio (HR)

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

#### 4.2.6.3. Colour properties of the fresh feed and reconstituted samples

Colour values ( $L$ ,  $a$ ,  $b$ ) were measured using a Hunter colour spectrophotometer (Hunter Colour Lab UltrascanVis). The ' $L$ ' value indicates degree of lightness. ' $L$ ' value in the range between 0-50 indicates darkness and 51-100 indicates lightness. Similarly, ' $a$ ' means measure of red (positive values) and green colour (negative values); ' $b$ ' measures the yellow (positive value) or blue (negative values) colours.<sup>[20]</sup> The colour 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 colour change ( $\Delta E$ ) of the samples was calculated according to Santipanichwong and Suphantharika.<sup>[21]</sup>

$$\Delta E = \sqrt{(L_0^* - L_0)^2 + (a_0^* - a)^2 + (b_0^* - b)^2} \quad \text{Eq. 4.5}$$

Where,  $\Delta E$  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.

#### 4.2.6.4. pH and titratable acidity of the powdered samples

pH of the sample was measured using a pH meter (Eutech, Merck). Briefly, 1 g of sample was dissolved in 5 mL deionised water and pH was measured at 27°C. Titratable acidity was determined by titration method.<sup>[15]</sup> To 1 g of sample dissolved in deionised

water, 2-3 drops of phenolphthalein indicator was added and titrated against 0.1N sodium hydroxide. Titratable acidity was expressed as citric acid equivalent.

$$\text{Titratable acidity (\%)} = \frac{\text{Titre value} \times 0.1\text{N NaOH} \times 64 \times 100}{\text{Sample volume taken} \times \text{weight sample} \times 1000} \quad \text{Eq. 4.6}$$

#### 4.2.6.5. Solubility

The solubility was determined according to the method described by Chau et al. [22] 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.

$$\text{Solubility (\%)} = \frac{W_f}{S} \times 100 \quad \text{Eq. 4.7}$$

Where,  $W_f$  is the final weight (g) of supernatant after drying and S is the weight (g) of sample.

#### 4.2.6.6. Hygroscopicity

The hygroscopicity property of the sample powders was determined according to Cai and Corke [23] with some modifications. Briefly, 2 g of spray dried powder samples in pre-weighed glass vials were kept 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 expressed as g moisture per 100 g solids.

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

The powder samples, prior to SEM observation, were mounted on stubs with double-sided adhesive tape, followed by coating the samples with a thin layer of gold. The SEM images were then obtained using a JSM-6390LV scanning electron microscope (JEOL, Japan) at 15 kV and 1000 x magnification.

#### 4.2.8. Particle size distribution of the powdered samples

The particle size distribution was determined using a particle 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 quantity of powder sample was suspended in water and analyzed at 25°C. The particle size (µM) was depicted with respect to its intensity (%) while particle size distribution was represented by span value and calculated using the equation given below.

$$\text{Span} = \frac{D_{90} - D_{10}}{D_{50}} \quad \text{Eq. 4.8}$$

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

#### **4.2.9. Phytochemical content and antioxidant activities of the fresh juice and dried powder**

##### **4.2.9.1. Sample extraction**

The samples were extracted in 80% acetone with a ratio of 1:10 (sample: solvent) and incubated in a shaking incubator (Certomat 1S, Sartorius) at 20°C for 90 min. After the incubation period, the crude extract was centrifuged at 3000 rpm (Hettich-Zentrifugen, Germany) for 15 min. The extracts were then stored at -20°C until further analyses.

##### **4.2.9.2. Determination of total phenolic content**

Total phenolic content in the sample extracts was assessed using the Folin–Ciocalteu assay <sup>[24]</sup> with slight modification. For the analysis, 20 µL each of extract, gallic acid standard or blank were taken in separate test tubes and to each 1.58 mL of distilled water was added, followed by 100 µL of Folin–Ciocalteu reagent, mixed well and within 8 min, 300 µL of sodium carbonate was added. The samples were vortexed immediately and the tubes were incubated in the dark for 30 min at 40°C. The absorbance was then measured at 765 nm in a UV-Vis spectrophotometer (Cecil, Aquarius 7400). The results were expressed in mg GAE/ 100g.

##### **4.2.9.3. Determination of total flavonoid content**

The flavonoid content was determined by aluminium trichloride method. <sup>[25]</sup> Briefly, 0.5 mL of the extract was mixed with 1.5 mL of 95% ethanol, 0.1 mL of 10% aluminium trichloride, 0.1 mL of 1M potassium acetate, and 2.8 mL of deionised water. After incubation at room temperature for 40 min, the reaction mixture absorbance was measured at 415 nm against deionised water blank in a UV-Vis spectrophotometer (Cecil,

Aquarius 7400). Results were expressed as mg quercetin equivalent (mgQE/100g) per 100g sample.

#### **4.2.9.4. Determination of ferric reducing antioxidant property (FRAP)**

FRAP activity of the samples was measured by the method of Benzie and Strain. [26] Briefly, a 40  $\mu$ L aliquot of properly diluted sample extract was mixed with 3 mL of FRAP solution. The reaction mixture was incubated at 37°C for 4 min and the absorbance was determined at 593 nm in a UV-Vis spectrophotometer (Cecil, Aquarius 7400) against a blank that was prepared using distilled water. FRAP solution was pre warmed at 37°C and prepared freshly by mixing 2.5 mL of a 10 mM 2,4,6-TPTZ [2,4,6-tri(2-pyridyl)-1,3,5-triazine] solution in 40 mM hydrochloric acid with 2.5 mL of 20mM ferric chloride and 25 mL of 0.3 M acetate buffer (pH 3.6). A calibration curve was prepared, using an aqueous solution of ferrous sulfate (1-10 mM). FRAP values were expressed as  $\mu$ M Fe (II) per 100 g of sample.

#### **4.2.9.5. Determination of DPPH activity**

Radical scavenging activity of the sample extracts was measured by determining the inhibition rate of DPPH (2, 2-diphenyl-1-picrylhydrazyl) radical. [27] Precisely, 100  $\mu$ L of extracts were added to 1.4 mL DPPH radical methanolic solution ( $10^{-4}$ M). The absorbance at 517 nm was measured at 30 min against blank (100  $\mu$ L methanol in 1.4 mL of DPPH radical solution) using a UV-Vis Spectrophotometer (Cecil Aquarius 7400). The results were expressed in terms of radical scavenging activity.

$$\text{Radical scavenging activity (\%)} = [(A_o - A_s) / A_o] \times 100 \quad \text{Eq. 4.9}$$

Where,  $A_o$  is absorbance of control blank, and  $A_s$  is absorbance of sample extract.

#### **4.2.10. Statistical analysis**

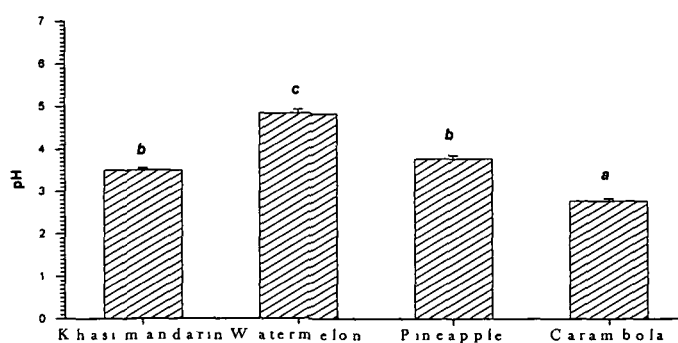
All experiments were carried out at least in triplicates and reported as mean  $\pm$  standard deviation of mean (S.E.M) using SPSS version 11.5. The physicochemical properties were statistically analyzed by Duncan's multiple range tests using one-way ANOVA while the colour, phytochemical content and antioxidant properties were subjected to paired-comparison t-test ( $p \leq 0.05$ ).



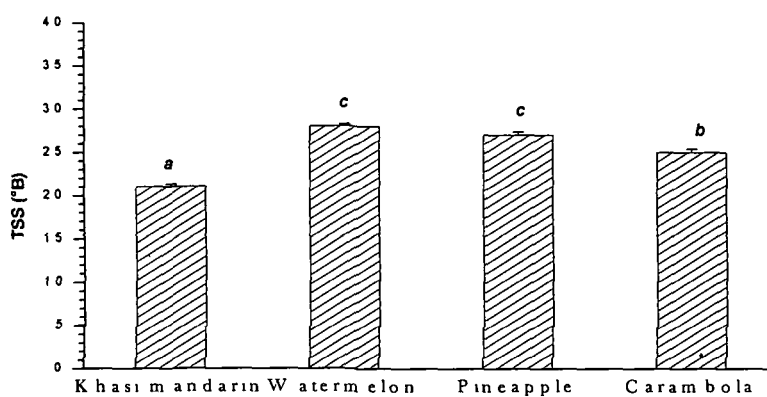
### 4.3. Results and discussion

#### 4.3.1. Total solid content, pH and viscosity of the feed sample

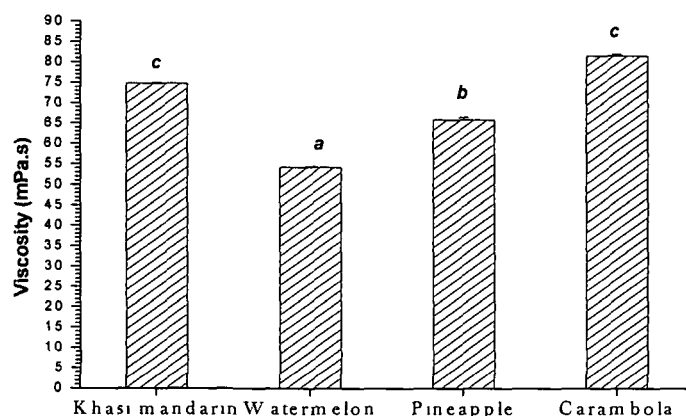
The pH value of the feed samples varied in the range of 2.77-4.83 (Fig.4.1a). However, no significant difference was observed in carambola and Khasi mandarin pH values. Similarly, the total solid content of the feed samples (Fig.4.1b) varied from 21°B-28°B and no significant variation was there between pineapple and watermelon juices. As represented in Fig.4.1c, viscosity of the samples (10°B) at the time of spray drying was in the range between 54.30-81.87 mPa.s. The variation in pectic colloidal substances in the studied juice samples may affect their viscosity even when the TSS was maintained at 10°B [28].



(a)



(b)



(c)

**Fig 4.1.** (a) pH, (b) Total solid content (°B), and (c) Viscosity of the raw juice feed samples for spray drying.

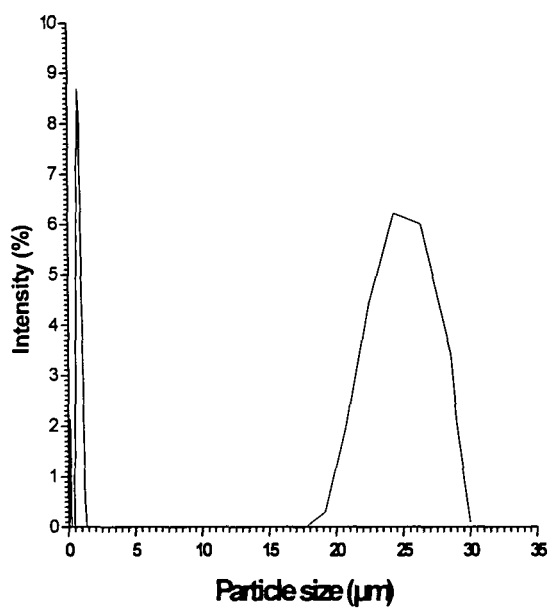
# Results are mean±S.D of triplicates. Same letter between the bars means no significant difference at  $p \leq 0.05$  by DMRT.

#### 4.3.2. Particle size distribution, cohesiveness and flow ability of the powdered sample

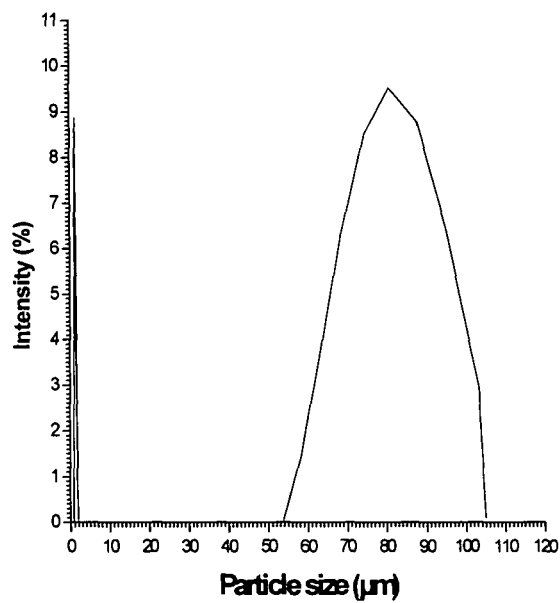
The bulk density (Table 4.2) of the samples ranged from 0.259-0.363 g/mL. The watermelon and pineapple showed slightly higher bulk density values than the remaining two samples. Same is the case in tapped density also. This may be due to the small particle size of the watermelon and pineapple samples (Fig.4.2).

The Hausner's ratio ranged between 1.22-1.57, while Carr index range was between 18.49% and 35.73%. On the basis of their Hausner's ratio and Carr index (Table 4.1), pineapple and Khasi mandarin had low cohesiveness compared to watermelon and carambola. As a result of which, the flowability as per Carr index in watermelon and carambola is bad while it is fair for Khasi mandarin and good for pineapple.

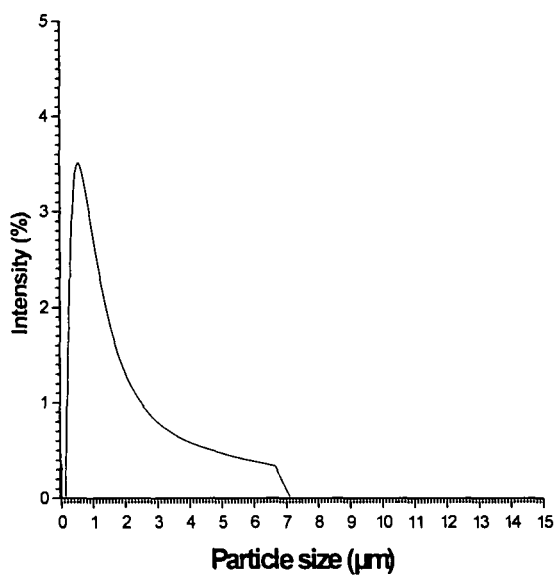
Particle size analysis showed (Fig.4.2) that watermelon (0.1-4  $\mu\text{M}$ ) and pineapple (0.2-7.2  $\mu\text{M}$ ) particle size are small compared to that of carambola (53-104  $\mu\text{M}$ ) and Khasi mandarin (18-30  $\mu\text{M}$ ) samples. The span value for the samples also varied (Table 4.2). In case of watermelon and pineapple span was 3.83 and 3.09, respectively, whereas it was 52.85 and 31.32, respectively for carambola and Khasi mandarin. It can be assumed that low viscosity values in watermelon and pineapple had lead to formation of smaller particles <sup>[29]</sup>.



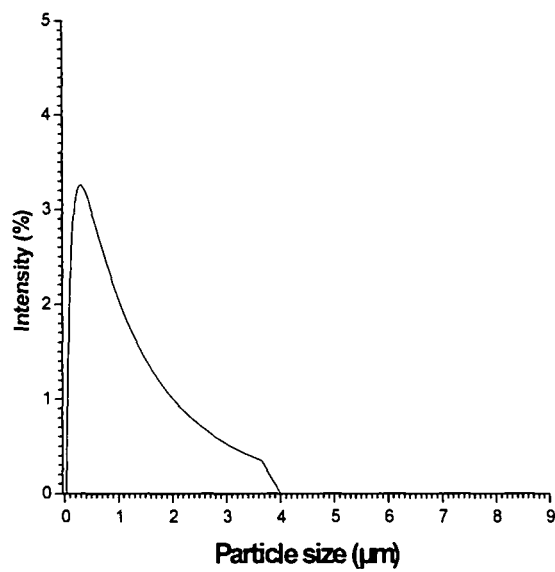
(a)



(b)



(c)



(d)

**Fig.4.2.** Particle size distribution of the spray dried juice powder against intensity. (a) Khasi mandarin (18-30  $\mu\text{M}$ ), (b) carambola (53-104  $\mu\text{M}$ ), (c) pineapple (0.2-7.2  $\mu\text{M}$ ) and (d) watermelon (0.1-4  $\mu\text{M}$ ).

**Table 4.2.** Bulk density, tapped density, Hausner's ratio, Carr index and span value of the spray dried fruit juice powders

Sample	Bulk density (g/mL)	Tapped Density (g/mL)	Hausner's ratio	Carr index (%)	Span value
Khasi mandarin	0.287±0.015 <sup>a</sup>	0.394±0.003 <sup>a</sup>	1.37	27.16	31.32±0.11 <sup>b</sup>
Watermelon	0.363±0.003 <sup>b</sup>	0.569±0.005 <sup>c</sup>	1.57	36.20	3.83±0.17 <sup>a</sup>
Pineapple	0.357±0.009 <sup>b</sup>	0.438±0.003 <sup>b</sup>	1.22	18.49	3.09±0.07 <sup>a</sup>
Carambola	0.259±0.011 <sup>a</sup>	0.403±0.002 <sup>a</sup>	1.56	35.73	52.85±0.19 <sup>c</sup>

\*\* Means ± S.D. of triplicates values with the same letter between the rows are not significantly different at  $p \leq 0.05$  by DMRT.

#### 4.3.3. Physicochemical parameters of the spray dried fruit juice powders

Fruit juices from carambola, watermelon, pineapple and Khasi mandarin, respectively were spray dried with 20% maltodextrin as carrier agent and were studied for the physicochemical parameters (Table 4.3). Highest yield was observed in Khasi mandarin spray dried juice. Watermelon and carambola juice powders showed no significant difference in the yield. The moisture content of the four spray dried samples ranged from 3.99% and 5.47%. All the fruit samples showed acidic pH in the range of 1.98-5.04 with carambola powder having 1.98 pH and watermelon showing relatively high pH of 5.04. The titratable acidity of carambola showed highest value of 0.70% while the acidity value for watermelon powder was very low. The low pH and high titratable acidity in carambola could be due to presence of oxalic acid that is predominant in the juice [30]. The solubility percentage of the samples ranged between 57.57% and 76.75%. Higher sugar content in watermelon and pineapple compared to Khasi mandarin and carambola besides the low moisture content might have contributed to their increased solubility [31]. Another reason could be that the particle size of watermelon and pineapple was small compared to the remaining two samples. Similarly, low viscosity values of watermelon and pineapple feed samples could result in low moisture content of the end product. There is no significant difference between Khasi mandarin and pineapple powders. Similarly, hygroscopicity of watermelon and carambola also showed no significant variation in values. However, when

the Khasi mandarin and pineapple hygroscopicity results were compared with that of watermelon and carambola values significant difference was observed.

**Table 4.3.** Physicochemical parameters of the spray dried fruit juice powder samples

Sample	Yield (%)	Moisture content (%)	pH	Titrateable acidity (%)	Solubility (%)	Hygroscopicity (g/100g)
Khasi mandarin	85.27±0.31 <sup>c</sup>	5.41±0.17 <sup>c</sup>	3.81±0.10 <sup>b</sup>	0.36±0.02 <sup>b</sup>	58.41±0.14 <sup>a</sup>	12.99±0.19 <sup>b</sup>
Watermelon	58.87±0.23 <sup>a</sup>	3.99±0.10 <sup>a</sup>	5.04±0.12 <sup>c</sup>	0.18±0.09 <sup>a</sup>	76.75±0.18 <sup>c</sup>	11.68±0.11 <sup>a</sup>
Pineapple	72.00±0.29 <sup>b</sup>	4.91±0.12 <sup>b</sup>	3.71±0.19 <sup>b</sup>	0.42±0.04 <sup>c</sup>	68.84±0.32 <sup>b</sup>	12.62±0.23 <sup>b</sup>
Carambola	57.45±0.11 <sup>a</sup>	5.47±0.19 <sup>c</sup>	1.98±0.10 <sup>a</sup>	0.70±0.05 <sup>d</sup>	57.57±0.25 <sup>a</sup>	11.44±0.27 <sup>a</sup>

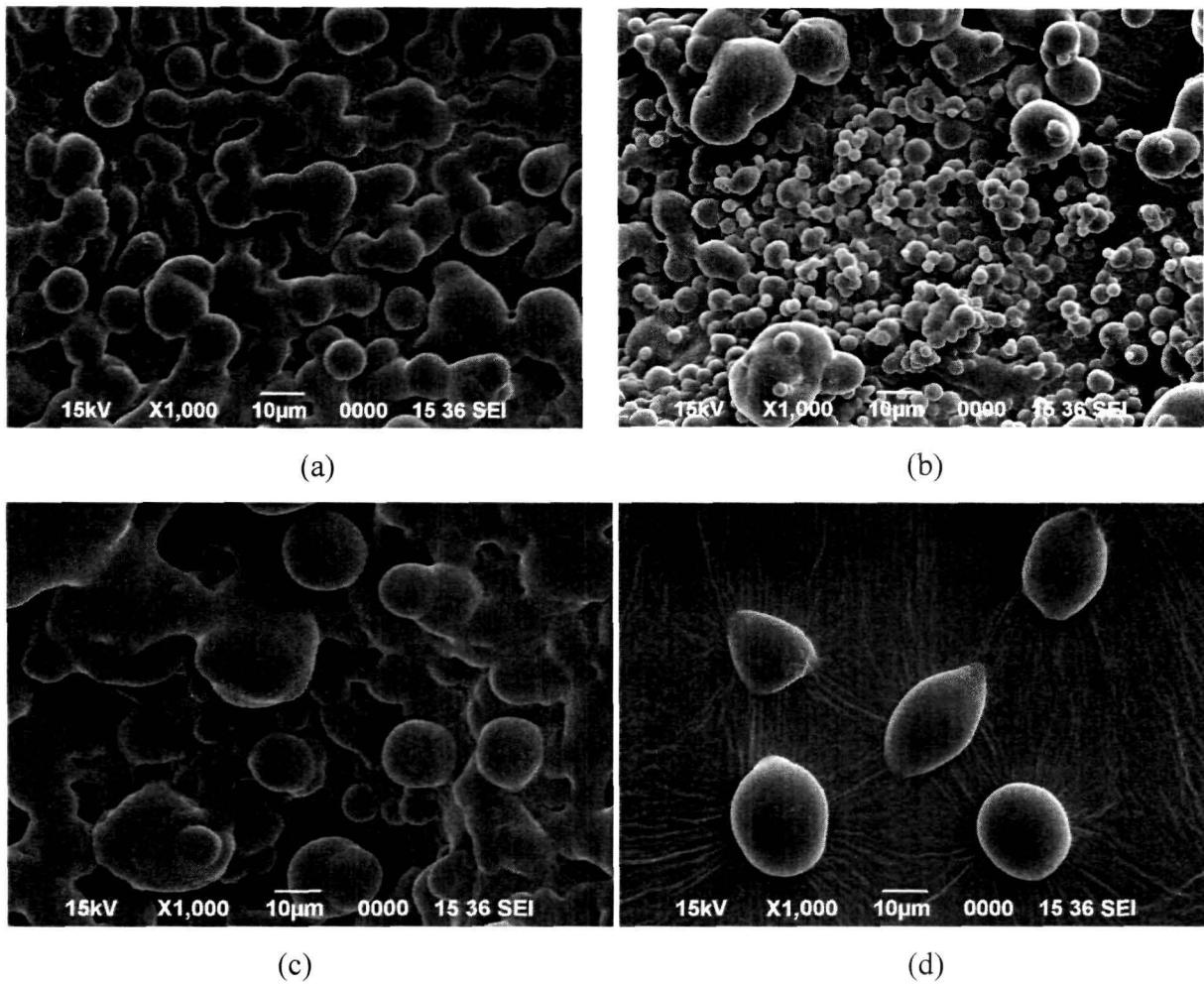
\*\* Means± S.D of triplicates values with the same letter between the rows is not significantly different at  $p \leq 0.05$  by DMRT.

#### 4.3.4. Surface morphology study of the spray dried juice powder

The surface morphology study showed varied shapes and sizes of the dried powder materials (Fig.4.3). In Khasi mandarin and pineapple powders, the particles were found to be fused and clumped together to one another. In carambola, both small and large spherical particles were observed. Particle size and shape in watermelon powder differed widely than the rest of the three sample powders. Large, globular and ellipsoidal particles in watermelon powder were observed at 1000 x magnification.

#### 4.3.5. Colour of the spray dried fruit juice powders

The colour comparison for 'L', 'a' and 'b' values between feed and reconstituted samples showed significant difference ( $p \leq 0.05$ ) during paired t-test except in 'L' value of watermelon (Table 4.4). Increased 'L' values were observed in reconstituted samples while a decrease was observed in the 'a' values. Similarly, increases in 'b' values were observed for the reconstituted samples except in pineapple. The overall colour change ( $\Delta E$ ) was much more prominent in Khasi mandarin.



**Fig. 4.3.** SEM image of the spray dried fruit juice powders. (a) Khasi mandarin, (b) carambola, (c) pineapple and (d) watermelon

**Table 4.4.** Colour parameters of the spray dried fruit juice powders

Sample	<i>L</i>		<i>a</i>		<i>b</i>		Overall colour difference, $\Delta E$
	FS	RS	FS	RS	FS	RS	
Khasi mandarin	23.39±0.11*	31.35±0.19*	0.17±0.03*	-0.47±0.02*	1.25±0.04*	6.20±0.10*	9.39±0.11
Watermelon	22.95±0.23	23.12±0.09	0.99±0.03*	0.51±0.09*	0.15±0.06*	1.17±0.08*	1.14±0.11
Pineapple	22.18±0.12*	24.55±0.18*	0.07±0.02*	-0.33±0.05*	0.24±0.10*	-0.04±0.02*	2.42±0.11
Carambola	22.59±0.27*	25.37±0.11*	0.11±0.05*	-0.16±0.02*	0.19±0.08*	1.01±0.02*	2.91±0.09

\* denotes statistically significant difference at  $p \leq 0.05$  during paired t-test between FS and RS values

# FS denotes feed sample; RS denotes reconstituted sample.

#### 4.3.6. Phytochemical content and activity of spray dried fruit juice

The results (Table 4.5) showed a significant difference in phytochemical content between the untreated and spray dried fruit juice. In Khasi mandarin orange powder, an increase in TPC, TFC, FRAP and DPPH was observed. In watermelon powder, a decrease in all the phytochemical parameters was observed. The TPC, TFC values in carambola powder sample showed no significant change on drying but increase in FRAP and DPPH was observed. However, in case of pineapple powder, a significant decrease in TPC and TFC occurred. While, no change in FRAP values took place, a significant increase in DPPH activity was observed compared to the untreated juice sample.

The varied results could be due to the reaction of the various phenolic compounds present in the fruit juices to the heat applied during the drying process. Although, phenolic compounds are generally heat labile, individual phenolic compounds have different degree of tolerance to heat and the structural degradation and rearrangement hence in turn affect its content and activity. The conformational change in phenolics could either render them more soluble and extractable in the extracting solvent or make them less soluble thus affecting their quantification. However, in carambola which is rich in proanthocyanidins that are relatively heat stable, on drying showed an enhanced FRAP and DPPH activity. Moreover, removal of moisture led to concentration of the bioactive compounds in some cases when compared with that of the raw samples. Application of heat in some cases could cleave the phenolic-sugar glycosidic bonds resulting in the formation of phenolic aglycones, which have high reactivity with Folin Ciocalteu reagent and thus lead to an increased value of total phenolics <sup>[32]</sup>. Hence, the change in phenolic content and antioxidant activity depends upon individual constituent phenolic acid and their susceptibility to heat and conformational changes.

The use of spray drying involves thermal treatment and therefore, loss of some phenolic acids is inevitable. The extent of degradation of phenolic compounds depends on the food matrix and processing conditions <sup>[33]</sup>. As reported by Mrkic et al. <sup>[34]</sup>, during drying, in some cases oxidation reactions may occur due to which, oxidized polyphenols may exhibit higher antioxidant activity than non-oxidised polyphenols. Also, high temperature conditions could trigger maillard reactions producing maillard products which can act as

antioxidants and can enhance their activity alone or in combination with other natural phenolic compounds [35]. Therefore, in some cases, although loss of total phenolic content was observed, the FRAP and DPPH values increased.

**Table 4.5.** Phytochemical content and antioxidant activity of fresh juice and spray dried fruit juice powder (dry basis)

<b>Samples</b>	<b>Fresh juice</b>	<b>Spray dried juice powder</b>
<b>Khasi mandarin</b>		
TPC(mgGAE/100g)	79.64±0.12*	332.78±0.27*
TFC(mgQE/100g)	8.41±0.09*	34.16±0.21*
FRAP(uM/100g)	1220.12±0.19*	1538.42±0.23*
DPPH (%)	59.97±0.17*	77.79±0.11*
<b>Watermelon</b>		
TPC(mgGAE/100g)	97.92±0.23*	25.75±0.21*
TFC(mgQE/100g)	9.76±0.16*	2.14±0.05*
FRAP(uM/100g)	85.08±0.25*	18.08±0.11*
DPPH (%)	53.23±0.27	48.94±0.18
<b>Carambola</b>		
TPC(mgGAE/100g)	644.65±0.22	618.52±0.17
TFC(mgQE/100g)	18.08±0.20	16.93±0.12
FRAP(uM/100g)	3155.57±0.14*	5210.17±0.19*
DPPH (%)	78.56±0.11*	92.24±0.27*
<b>Pineapple</b>		
TPC(mgGAE/100g)	225.99±0.32*	149.32±0.28*
TFC(mgQE/100g)	7.77±0.11*	14.59±0.10*
FRAP(uM/100g)	679.38±0.29	627.99±0.23
DPPH (%)	59.97±0.27*	77.79±0.20*

\* denotes statistically significant difference at  $p \leq 0.05$  during paired t-test.



#### 4. 4. Conclusion

Spray drying of the juice samples had both positive and adverse effects on the final product's quality and antioxidant activity. The physicochemical properties such as yield, colour, solubility, moisture content and hygroscopicity of the final product varied depending on the source of fruit juice. The phytochemical content and antioxidant activities also showed variations. But unlike the rest of the samples, solubility was highest in spray dried watermelon. In watermelon and pineapple powders, decrease in TPC and TFC was observed. However, carambola showed no significant changes in TPC, TFC values but exhibited increased FRAP and DPPH values. Therefore, it could be inferred that depending on the fruit juice type the increase or decrease in phytochemicals and their antioxidant activities differed. All the fruit juice powders had phytochemical and antioxidant activities. Therefore, spray drying is a convenient method to process and convert fruit juice into powder that has convenience of reconstitution just prior to consumption and also provides health benefitting compounds.

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