Chapter-6

Effect of maltodextrin concentration and inlet temperature during spray drying on physicochemical and antioxidant properties of Amla (Emblica officinalis) juice powder
6.1. Introduction

Antioxidants are required to combat free radicals that are generated in the body as these free radicals accelerate hyper pigmentation syndrome, heart disease, stroke, atherosclerosis, diabetes and cancer\(^1\,^2\). Recently huge interest has been generated in finding naturally occurring antioxidants for use in foods or medicinal materials to replace synthetic antioxidants, which are being restricted due to their side effects like carcinogenicity, etc\(^3\,^4\).

It is well established that Amla is a good source of total phenolic content, antioxidants, flavones, tannins and other bioactive compounds\(^5\); these substances may contribute to the health effects of Amla. However, being a seasonal fruit its availability as a fresh fruit is for a limited period. Hence spray drying of the fruit juice may be a good alternative to make its health promoting components available throughout the year.

Fruit juice powders have number of benefits over the liquid counterparts such as reduced volume or weight, reduced packaging, easier handling, transportation and longer shelf life\(^6\). Spray dried powders have good reconstitutinal characteristics, have low water activity and are suitable for storage. Spray drying technique is also appropriate for heat sensitive components. Maltodextrin, gum arabic and gelatin are successfully used as drying aids to facilitate drying. Maltodextrin is one of the common drying aids for spray drying owing to its beneficial role as a carrier or an encapsulating agent. Workers have used carrier agents to protect heat sensitive components like vitamin C in camu-camu\(^7\) and to increase stability of products in acerola powder\(^8\). However, there are no reported studies on spray drying of Amla juice powder to retain functional properties like total phenolic content and DPPH\(^*\) scavenging activity.

A study was conducted wherein Amla juice was spray dried with different maltodextrin concentrations at different inlet temperatures. The effect of spray drying on the physicochemical properties, total phenolic content and DPPH\(^*\) scavenging activity of spray dried Amla powder are reported in this chapter.
6.2. Materials and methods

6.2.1. Chemicals

Chemicals for present investigation i.e. Folin-Ciocalteu reagent (FCR), sodium carbonate, 2, 2, diphenyl picryl hydrazil (DPPH) and ethanol were purchased from Merck.

6.2.2. Raw materials

Amla of Chakaiya variety was procured from local market in Allahabad, India. The Amla was cleaned thoroughly under tap water to remove adhering dust and wiped with muslin cloth. The washed fruits were used for development of spray dried Amla juice powder.

6.2.3. Process standardization for development of spray dried Amla powder

Amla of Chakaiya variety containing 83.40±1.10% moisture content, 11.10±0.83% carbohydrates, 2.32±0.21% crude fibre on fresh wt basis and 26.5±1.12 g/100 g total phenolic content equivalent to gallic acid on dry wt basis were used. Amla fruits were cut into small pieces and pulped in a laboratory grinder. Juice was extracted by straining through double fold muslin cloth and concentrated upto 40% in rotary evaporator at 70°C temperature. Maltodextrin of varying concentration (5-9% w/v of initial juice) was added and stirred for 15 min in a stirrer. The juice was fed into spray dryer and spray dried at inlet temperatures ranging from 125°C to 200°C. The spray dried powder was packed into laminates and stored at 4°C temperature for further analysis.

Preliminary spray drying trials showed that at maltodextrin level of 3% most of the material stuck on chamber wall, while at concentration above 9% a significant decrease in the DPPH* scavenging activity in finished spray dried Amla powder was observed. Hence, levels of maltodextrin used in the study varied between 5% and 9%. Feed material for all the formulations came from a single batch of Amla juice.
6.2.3.1 Spray drying conditions of fresh Amla juice powder

The feed comprising of maltodextrin and juice was spray dried in Lab plant LU 20 lab spray dryer (Labultima, Mumbai, India). The inlet temperatures/measured outlet temperatures were 125°C/81°C, 150°C/93.5°C, 175°C/103°C and 200°C/119°C. The compressor pressure, air flow rate and feed rate were constant at 0.12 MPa, 75±1.5 m³/h and 13-15 mL/min, respectively. Drying conditions were selected on the basis of final moisture content in the finished spray dried Amla powder. All formulations for spray drying were carried out in duplicates.

6.2.4. Analytical methods

6.2.4.1. WSI (Water solubility index)

The WSI of the powder was determined using the method described by Anderson et al. (1969). Spray dried Amla juice powder (2.0 g) and distilled water (25 mL) were vigorously mixed in a 100 mL centrifuge tube, incubated in a 37°C water bath for 30 min and then centrifuged for 20 min at 7895 g (Sigma, 13 K, Germany). The supernatant was carefully collected in a pre-weighed beaker and oven dried at 103±2°C. The WSI (%) was calculated as the percentage of dried supernatant with respect to the amount of the original Amla powder.

6.2.4.2. Hygroscopicity

For hygroscopicity, 1.5 g of the powder was placed at 25°C in an airtight container containing saturated solution of sodium carbonate. Sample was weighed after 1 week and hygroscopicity was expressed as gram of adsorbed moisture per 100 g of powder.

6.2.4.3. Color characteristics of spray dried Amla powder

The color characteristics of the spray dried amla powder were analyzed by using Hunter Color Lab (Ultra scan VIS, USA) calibrated with white tiles. Obtained results were expressed as Hunter color values L, a and b where L denotes lightness and darkness, a redness and greenness and b yellowness and blueness. Powders were
packed in polyethylene pouches and were measured for color characteristics. The samples were analyzed in triplicates. Color intensity in terms of chroma was calculated by the formula \( (a^2 + b^2)^{1/2} \), whereas hue angle \( (H^\circ) \) was calculated by the formula \( H^\circ = \arctan(b/a) \). The hue values of 0°, 90°, 180° and 270° denote pure red, pure yellow, pure green and pure blue color respectively. The ratio of \( a/b \) was also estimated for color measurement of spray dried Amla powder 11.

6.2.4.4. Bulk density

Briefly, 2.0 g of Amla powder was added in 10 mL of graduated measuring cylinder and the mixture was vortexed for 1 min. Bulk density of the powder was calculated by measuring the ratio of mass of powder to the volume occupied by the powder 6.

6.2.4.5. Total phenolic content

Estimation of total phenolic content was performed by Folin-Ciocalteu method described by Liu et al. (2008)12 with some modifications. Briefly, 250 mg of sample was mixed with 10 mL of 60% acetone and the mixture was stirred for 30 min at 30° C. Then 60 μL of supernatant, 300 μL of Folin - Ciocalteau reagent and 750 μL of 20% sodium carbonate in water were added in 4.75 mL of water. The mixture was allowed to stand for 30 min. The absorbance was measured at 765 nm using double beam spectrophotometer (Evolution 600, Thermoscientific) and the results are expressed as mg of GAE. A standard curve of absorbance vs concentration was plotted using gallic acid standard at various concentration (ranging from 50-500 mg/L).

6.2.4.6. 2,2, Dipheryl picryl hydrazil (DPPH*) radical scavenging activity

The DPPH* scavenging activity of extract was determined by the method of Luo et al. (2009)13 with slight modifications. Briefly, 2 mL of extract were mixed with 2 mL methanolic solution containing 1mM DPPH. The mixture was shaken vigorously and then left to stand for 30 min in the dark. The absorbance was measured at 517 nm using double beam spectrophotometer (Evolution 600, Thermoscientific). The absorbance of control was obtained by replacing the sample with methanol. DPPH radical scavenging activity of the sample was calculated as follows:

\[
\text{DPPH}\text{* scavenging activity (\%)} = \left( \frac{\text{absorbance of control} - \text{absorbance of sample}}{\text{absorbance of control}} \right) \times 100
\]
6.2.5. Scanning electron micrograph (SEM)

Particle morphology was evaluated by SEM. Powders were attached to a double sided adhesive tape on SEM stubs, coated with 3-5 mA palladium under vacuum and examined with a JEOL scanning electron microscope (JSM-6390 LV, Japan, PN junction type, semi conducting detector). SEM was operated with 15 KV at magnifications of 1000X and 5500X.

6.2.6. Statistical analysis

Spray drying experiments were carried out in duplicate and analyses were carried out in triplicates. Obtained mean values were analyzed by analysis of variance (ANOVA). The graphs of mean value and error bar were created by using Excel version of 2003.

6.3. Results and discussion

6.3.1. Effects of spray drying conditions on physical properties of Amla powder

The effects of maltodextrin concentration, aspiration speed and different drying temperatures on the physical properties of the Amla juice powder are shown in Table 1. The levels of maltodextrin used for development of the Amla powder varied between 5 - 9% (w/v) which were less than 10-30% that were used by Tonon et al. (2008)\(^\text{14}\), Abadio et al. (2004)\(^\text{15}\), and Kha et al. (2010)\(^\text{16}\). Increase in the maltodextrin concentration resulted in a significant (5% probability level) decrease in moisture concentration in the finished powder. Moisture content of sample decreased from 5.6 to 3.8% as maltodextrin level increased from 5 to 9% (Table 6.1). Abadio et al. (2004)\(^\text{15}\) there was also found a decrease in moisture content in final pineapple juice powder with increase in the level of maltodextrin from 10-15 % (w/v). Increased level of maltodextrin increased the level of feed solids and reduced the level of total moisture for evaporation\(^\text{16-17}\).

Increase in drying temperature from 125°C to 200°C also resulted in significant decrease in the moisture content in amla powder from 6.3 to 3.8% (Table 6.1). Due to the increased rate of heat transfer into the particles at higher temperature, there was a greater driving force for moisture evaporation causing faster water removal\(^\text{18,19}\). The present findings were in agreement with the results obtained for
spray dried tomato powder\textsuperscript{6}, orange juice powder\textsuperscript{20}, cactus pear juice powder\textsuperscript{21}, black carrot powder\textsuperscript{22} and gac juice powder\textsuperscript{16}.

Maltodextrin level had significant (p<0.05) effect on the hygroscopicity of the amla powder. Hygroscopicity was lowest when 9% maltodextrin was used for encapsulation. Rodriguez-Hernandez et al. (2005)\textsuperscript{21} and Cai and Corke, (2000)\textsuperscript{10} also observed a reduction in hygroscopicity with increasing maltodextrin concentrations in spray dried cactus pear juice powder and betacyanin pigments, respectively. Maltodextrin is a material having the property of low hygroscopicity and its utility as a carrier material for spray drying has been established\textsuperscript{14}. Inlet temperature also influenced the hygroscopicity of the powder significantly. The highest hygroscopicity value of 56.32 g/100 g for Amla powder was obtained at 125\degree C inlet temperature. When inlet temperature of processing was increased the hygroscopicity of Amla powder was decreased. The present findings are in agreement with Moreira et al. (2009)\textsuperscript{23} but contradicts the findings of Goula et al. (2004)\textsuperscript{6} and Tonon et al. (2008)\textsuperscript{14} in their work on spray drying of tomato pulp and acai juice powder, respectively. Amla powder showed greater tendency to adsorb moisture which may be due to the presence of higher level of carbohydrates in Amla fruit. Aspiration speed showed no significant effect on hygroscopicity of the powders.

Drying temperature did not show any significant effect on WSI of the Amla powder (Table 6.1) at 5% probability level (Table 6.1). Sousa et al. (2008)\textsuperscript{24} also observed that drying conditions had no significant effect on WSI of tomato powder\textsuperscript{24}. In the present study, WSI of Amla powder ranged from 91.34 to 94.98%. These values were higher when compared with 17.65-26.3\% in spray dried tomato powder\textsuperscript{24}, 36.91-38.25\% in gac powder\textsuperscript{16} and 81.56\% in pineapple juice powder\textsuperscript{15}. The excellent WSI of the Amla juice powder could be due to the high content of free phenolics\textsuperscript{25}, the significant level of carbohydrates and the low level of fat (0.05\% in fresh fruit) in the juice. According to Kumar et al. (2008)\textsuperscript{25}, 97.67\% of total phenolics are present in free form in Amla fruit\textsuperscript{25}. The dissolved solids in the Amla juice are highly water soluble. These soluble solids are mostly carbohydrates and free phenolics. Thus, free phenolics and soluble carbohydrates account for the high WSI in Amla juice powder. The high WSI observed in Amla powder makes it suitable for reconstitution. While maltodextrin level did not have significant effect on the bulk density of Amla powder

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at 5% probability level, the drying temperature showed a significant effect (Table 6.1). A decrease in the density of the powder was observed with an increase in the inlet temperature. This finding is consistent with those by Walton and Mumford, (1999)\textsuperscript{27}, Cai and Corke, (2000)\textsuperscript{10}, Goula et al. (2004)\textsuperscript{6} and Kha et al (2010)\textsuperscript{16}. As explained by Jumah et al. (2000)\textsuperscript{27}, Walton, (2000)\textsuperscript{28} and Chegini and Ghobadian, (2005)\textsuperscript{20}, the high rate of drying being rapid at very high temperatures meant that there was less droplet shrinkage, giving lower powder density. This explanation is supported by our findings. Aspiration speed had no significant effect on the bulk density of the powder.

Table 6.1 Physicochemical properties of spray dried Amla powder

<table>
<thead>
<tr>
<th>Particular</th>
<th>MC (%)</th>
<th>Hygroscopicity</th>
<th>WSI (%)</th>
<th>Bulk density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Maltodextrin concentration (MDC)</strong> (%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>5</td>
<td>5.6 ±0.1\textsuperscript{a}</td>
<td>53.01±1.32\textsuperscript{a}</td>
<td>93.28±2.33\textsuperscript{a}</td>
<td>0.52±0.01\textsuperscript{a}</td>
</tr>
<tr>
<td>7</td>
<td>4.07±0.15\textsuperscript{b}</td>
<td>47.74±1.04\textsuperscript{b}</td>
<td>94.11±1.87\textsuperscript{a}</td>
<td>0.49±0.03\textsuperscript{a}</td>
</tr>
<tr>
<td>9</td>
<td>3.83±0.42\textsuperscript{c}</td>
<td>46.03±0.98\textsuperscript{c}</td>
<td>93.32±2.41\textsuperscript{a}</td>
<td>0.51±0.04\textsuperscript{a}</td>
</tr>
<tr>
<td><strong>Inlet temperature (°C) (DT)</strong></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td>125</td>
<td>6.32±0.31\textsuperscript{a}</td>
<td>56.32±1.22\textsuperscript{a}</td>
<td>94.98±1.98\textsuperscript{a}</td>
<td>0.61±0.02\textsuperscript{a}</td>
</tr>
<tr>
<td>150</td>
<td>5.29±0.35\textsuperscript{b}</td>
<td>49.82±2.12\textsuperscript{b}</td>
<td>93.14±1.67\textsuperscript{a}</td>
<td>0.55±0.01\textsuperscript{b}</td>
</tr>
<tr>
<td>175</td>
<td>4.54±0.28\textsuperscript{c}</td>
<td>47.02±1.98\textsuperscript{c}</td>
<td>92.36±2.03\textsuperscript{a}</td>
<td>0.52±0.01\textsuperscript{c}</td>
</tr>
<tr>
<td>200</td>
<td>3.83±0.42\textsuperscript{d}</td>
<td>46.74±1.46\textsuperscript{d}</td>
<td>94.34±1.90\textsuperscript{a}</td>
<td>0.49±0.03\textsuperscript{d}</td>
</tr>
<tr>
<td><strong>Aspiration speed (AS)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>3.83±0.32\textsuperscript{a}</td>
<td>46.74±1.33\textsuperscript{a}</td>
<td>94.89±2.37\textsuperscript{a}</td>
<td>0.49±0.03\textsuperscript{a}</td>
</tr>
<tr>
<td>40</td>
<td>4.01±0.29\textsuperscript{b}</td>
<td>47.08±2.09\textsuperscript{a}</td>
<td>93.28±2.01\textsuperscript{a}</td>
<td>0.52±0.18\textsuperscript{a}</td>
</tr>
<tr>
<td>50</td>
<td>4.32±0.24\textsuperscript{c}</td>
<td>47.34±1.49\textsuperscript{a}</td>
<td>91.43±2.52\textsuperscript{a}</td>
<td>0.52±0.23\textsuperscript{a}</td>
</tr>
<tr>
<td><strong>Significant interaction</strong></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>MDC</td>
<td>**</td>
<td>***</td>
<td>ND</td>
<td>NS</td>
</tr>
<tr>
<td>DT</td>
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<td>ND</td>
<td>***</td>
</tr>
<tr>
<td>MDCxDT</td>
<td>**</td>
<td>**</td>
<td>NS</td>
<td>NS</td>
</tr>
</tbody>
</table>

ND : not detected, NS : non significant; **, *** : significant at p = 0.05 & p = 0.01 respectively. The values of the same column with different superscript differ significantly.
6.3.2. Particle morphology

Fig. 6.1 shows the SEM micrographs of Amla juice powder produced with 9% maltodextrin at different inlet temperatures. It was observed that the number of particles in a given amount of the powder increased with an increase in inlet temperature. Similar findings were also reported by Tonon et al. (2008)\textsuperscript{14}. Inlet temperature had no effect on the surface smoothness of the particles. This however contradicts the observation of Allamila- Beltran et al. (2005)\textsuperscript{29}, Nijdam and Langrish (2006)\textsuperscript{30} and Tonon et al. (2008)\textsuperscript{14}. SEM study (Fig 6.1) revealed that the average size of particles in the powder that was dried at higher inlet temperature was smaller than the particles in powder dried at lower inlet temperature. Similar finding was observed by Cai and Corke, (2000)\textsuperscript{10} for spray drying of amaranthus betacyanin pigments. Probably, the particle size got fixed as large sized globules when there was more water in the material that was being dried. At higher inlet temperature, due to rapid rate of drying the particles got fixed as smaller sized globules. Image of spray dried Amla juice powder is shown in Fig 6.2.

6.3.3. Color characteristics of powder

Fig. 6.3 shows the effect of different concentrations of maltodextrin and inlet temperatures on color characteristics of spray dried powder. In general, the color attributes of the powder was significantly affected by maltodextrin and temperature. The lightness of the powders was significantly affected by maltodextrin concentration (p<0.01) when powders were produced at inlet temperatures below 200\degree C. Highest value of \(a/b\) and lowest value of hue angle were obtained with 5\% concentration of maltodextrin at 200\degree C while no specific trend was observed for 7\% and 9\% levels of maltodextrin. No specific trend could be observed for chroma of the powder when produced at different inlet temperatures and maltodextrin levels. Spray drying temperature showed statistically significant effect on the color characteristics i.e lightness, \(a/b\), chroma and hue value. A significant effect on lightness of the powder was observed when inlet temperature was increased from 125\degree to 200\degree C temperature.
Effect of maltodextrin concentration and inlet temperature during spray drying on physicochemical and antioxidant properties of Amla (Emblica officinalis) juice powder

Fig 6.1 Micrographs of particles at different inlet temperature and constant maltodextrin level (9%) at magnifications of (a) 150°C, 1000 X, (b) 150°C, 5500 X, (c) 175°C, 1000X, (d) 175°C, 5500 X, (e) 200°C, 1000X, and (f) 200°C, 5500 X.
An increase in inlet temperature produced significantly (p<0.001) lighter product than powder produced at lower inlet temperature. Greatest degree of lightness of spray dried Amla powder at highest inlet temperature indicates that the pigments had undergone oxidation. Similar results were observed by Sousa et al. (2008) in spray dried tomato powders. The probable reason for higher degree of lightness of the Amla powder at higher inlet temperature may be attributed to the reduced rate of oxidation of the tannins. Tannins react slowly with iron in the absence of oxygen and form dark coloured complex. Probably, during the rapid rate of drying in a spray dryer, tannins get very little time to react with iron as availability of oxygen is reduced and therefore the powder colour had lessened.

Loss of redness of samples increased, resulting in low a/b value and high hue angle, when inlet temperature was increased from 125° to 175°C, however above 175°C temperature high a/b and low hue angle was found in the powder. Similar findings were reported by Graboswki et al. (2006) for sweet potato powder, by Abadio et al. (2004) for pineapple juice powder and by Kha et al. (2008) for gac juice powder. Higher maltodextrin level and higher inlet temperature resulted in low a/b value and high hue angle which are in concurrence with Chen et al. (1995). The probable reason may be that spray drying increased the surface area causing rapid pigment oxidation (Desobry et al., 1997). Reduced formation of iron tannate complex at high inlet temperature is also speculated which needs further studies for confirmation.

Fig 6.2 Image of spray dried Amla powder.
Effect of maltodextrin concentration and inlet temperature during spray drying on physicochemical and antioxidant properties of Amla (Emblica officinalis) juice powder

Fig. 6.3 The color characteristics of spray dried Amla powder (a) lightness, (b) ratio of $a/b$, (c) chroma, and (d) hue angle at different inlet temperatures and maltodextrin level.
6.3.4. Effect of spray drying conditions on total phenolic content of Amla powder

Fig. 6.4a shows the effect of processing conditions on total phenolic content (TPC) of spray dried powder. Drying temperature and maltodextrin concentration showed significant effect on TPC of spray dried powder. TPC was significantly (p<0.001) reduced when inlet temperature was increased from 125°C to 175°C temperature, however above 175°C there was a reverse trend. The reason for increased TPC content in the powder above 175°C may be because of the polymerisation as well as synthesis of polyphenols at 200°C which increases the total phenolic content of the powder.

TPC content of the powders was significantly reduced when the concentration of maltodextrin was increased from 5 to 9%. This can be explained to be due to the concentration effect of maltodextrin.

![Graph (a)](image)

**Fig. 6.4a** Effect of spray drying conditions on (a) total phenolic content, and (b) DPPH* scavenging activity of Amla powder.
6.3.5. Effect of spray drying conditions on free radical scavenging activity of powder

Fig. 6.4b shows the effect of spray drying conditions on DPPH\(^*\) scavenging activity of amla powder. Maltodextrin level and drying temperature showed statistically significant effect (p<0.01) on the DPPH\(^*\) scavenging activity of the powder. DPPH\(^*\) scavenging activity of the powder was significantly affected by increased inlet temperature. Overall, on increasing the inlet temperature from 125\(^\circ\)C to 200\(^\circ\)C a significant decrease in DPPH\(^*\) scavenging activity was observed. Similar results were observed in spray dried gac juice powder by Kha et al. (2008)\(^\text{16}\). The possible explanation for the low free radical scavenging activity may be because of the exposure to higher temperatures which adversely affected the structure of phenolics causing its break down and/or synthesis into different forms.

Increase in the maltodextrin concentration which itself has no free radical scavenging activity, resulted in lower DPPH\(^*\) scavenging activity. Powders containing 5\% maltodextrin level showed significantly higher free radical scavenging activity (dry wt basis) than 7 and 9\% of maltodextrin. This may be the dilution effect of maltodextrin when its concentration was raised. Obtained results contradicted the result of Kha et al. (2008)\(^\text{16}\) who found that on varying the maltodextrin level from 10-20\% there was no significant effect on total antioxidant activity of gac juice powder.

6.4. Conclusion

The effect of spray drying conditions on physicochemical properties of Amla powder was evaluated. Maltodextrin concentration (5-9\%) and drying temperature (125\(^\circ\)C to 200\(^\circ\)C) significantly affected moisture content, bulk density, hygroscopicity, color attributes, TPC and DPPH\(^*\) scavenging activity. However WSI was not significantly influenced by varying the concentration of maltodextrin or inlet temperature. The developed spray dried powder showed excellent water solubility that is essential for reconstitution. Amla juice powder dried at 175\(^\circ\)C with 7\% maltodextrin was adequately effective to produce powder with less hygroscopicity, acceptable color in terms of \(L, a\) and \(b\) and potent free radical scavenging activity. Spray dried Amla juice powder thus can be made into a health promoting reconstituted drink.
References


