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Spray-drying of coconut treacle into an amorphous powder

J. M. M. A. Jayasundera* and A. R. Kulatunga

Coconut Processing Research Division, Coconut Research Institute, Bandirippuwa Estate, Lunuwila, 61150, Sri Lanka

Abstract

We have investigated the amount of drying aid required to produce amorphous coconut treacle powder through spray-drying. Maltodextrin (DE-10) was used as the drying aid. Three different formulations of coconut treacle: maltodextrin: water (30%: 20%: 50%, 35%: 15%: 50% and 40%: 10%: 50%) were spray dried at inlet and outlet temperatures of 165°C and 65°C, respectively. Powder recovery in a pilot scale spray-dryer was used as a measure of the ease of spray-drying for a given formulation. The spray-dried coconut treacle powder was characterized for moisture content, water activity, sugar profile and particle morphology. The best powder recovery (58.6±3.5%) was obtained for the formulation of coconut treacle: maltodextrin: water in the ratio of 35%:15%:50%. The initial moisture content and water activity value of the spray-dried coconut treacle powder were 1.04±0.003% and 0.43±0.00%, respectively. It was interesting to note that both the moisture content and the water activity of treacle powder did not vary significantly (p>0.05) on storage of one year. The scanning electron micrograph of spray-dried coconut treacle powder showed that the particles were spherical in shape. The morphology of these particles apparently gives a good indication of the coconut treacle powder being amorphous. The sugar profile of this coconut treacle powder showed that it had a total sugar content of 88.2±0.05% out of which 46.6±0.02% was sucrose, 23.0±0.02% was glucose and 18.6±0.01% was fructose.

Key words: Coconut treacle, Spray-drying, Maltodextrin, Stickiness, Drying aid

Introduction

There are many food products that have very high sugar contents. Although there is a growing interest to convert them into more useable and stable forms such as powders, it is very difficult to convert such sugar-rich foods into particulate form due to the presence of a high proportion of low molecular weight sugars in their composition (Bhandari et al., 1997; Adhikari et al., 2007). This results into low glass transition temperatures of sugars, which is attributed to be the main reason for stickiness (Vega et al., 2005). Many foods are either crystalline or amorphous or a mixture of both depending on the composition of materials and the processing technology used (Boonyai, 2005). Amorphous powders have a higher solubility compared to that of crystalline powders.

Spray-drying is a well-established and widely-used method for converting a wide range of liquid foods into amorphous powders (Adhikari et al., 2009a; Adhikari et al., 2009b; Jayasundera et al., 2010). The process involves spraying of finely atomized solutions into a chamber where hot and dry air rapidly evaporates the solvent and quickly produces dried particles. Spray-dried powders can be stored at ambient temperature for prolonged periods without compromising the powder stability (Jayasundera et al., 2011). They are also cheaper to transport and easier to handle in manufacturing plants. Spray-drying is economical compared to freeze-drying as the latter is a batch process with long processing time and is also six times as expensive as spray-drying in terms of operational cost and nine times as expensive as spray-drying in terms of capital cost (Chavez, and Ledeboer, 2007; Santivarangkna et al., 2007). Spray-drying has many applications, particularly in the food, pharmaceutical and agrochemical industries (Adhikari et al., 2007; Vega et al., 2005; Bhandari et al., 1993; Maa et al., 1998; Maa and Hsu, 1997).

However, one prevalent problem in spray-drying is the stickiness that occurs when sugar-rich foods such as fruit juices, sweet toddy and honey
are spray-dried. The stickiness results in depositions onto the internal dryer wall and unacceptable clumping of particles. Both of these lead to inferior product quality and economic loss (Bhandari and Howes, 1999; Ozmen and Langrish, 2003).

To minimize the stickiness problem, both process-based and material science-based approaches are used. Process-based approaches include the mechanical scraping of the chamber wall, introduction of cold air at the bottom and the use of low temperature / low humidity air. An example of the material science based approach involves the addition of drying aids such as corn starch, gum Arabic and maltodextrin to reduce the stickiness of the powders (Downton et al., 1982; Werner et al., 2007).

There is an increasing demand in coconut sap sugar as an alternative sweetener both in local and international markets. It is also reported that sap sugar has a low glycemic index of about 35 (Arcot and Brand-Miller, 2005) making it a natural, safe sweetener and ideal for diabetics.

Coconut treacle which is made out of coconut inflorescence juice or the sap is a sugar-rich food with almost 65-75% sugars and 25-35% water and other minor constituents such as organic acids, mineral salts, vitamins, proteins, phenolic compounds and free amino acids (Ouchemoukh et al., 2007). Sugars predominate in the composition of coconut treacle and among them 47.73% are sucrose, 11.83% glucose and 11.53% fructose.

Coconut treacle poses multiple problems in handling during processes involving mass production due to its viscous and sticky nature thereby leading to a growing demand for dried treacle powder by both consumers and the food industry.

However, no studies have been carried out to convert coconut treacle into an amorphous powder due its inherent stickiness. Therefore, this study was aimed at spray-drying coconut treacle into an amorphous powder with the addition of maltodextrin (DE-10) as a drying aid and to characterize the powder with respect to moisture content, water activity value, crystalline/amorphous nature and sugar profile. Further, the keeping quality of the coconut treacle powder was determined.

Materials and Methods
Maltodextrin DE-10 (food grade) was imported from Qingyuan Foodstuff Company Limited, China. Coconut treacle was prepared from coconut sweet toddy collected at Bandirippuwa Estate, Lunuwila, Sri Lanka.

Solution preparation
Three different formulations of coconut treacle: drying aid: water (30%: 20%:50%, 35%: 15%: 50% and 40%:10%:50%) were prepared by heating the solutions at 45±5°C and gently agitating them with a magnetic stirrer and subsequently spray-dried.

Powder production
Spray-drying of solutions was carried out on a pilot scale spray-dryer (L-8, Ohkawara Kakohki Co Ltd., Yokohama, Japan) with a water evaporating capacity of 1L/hr. The inlet and outlet temperatures were maintained at 165°C and 65°C, respectively. The powders were collected from cyclone and the cylindrical part of the dryer chamber by lightly sweeping the chamber wall (Bhandari et al., 1997a). The yield was calculated as the ratio of the mass of solids collected to the mass in feed solution on a dry basis. Depending on the powder recovery the best formulation was selected for further studies.

Moisture
Moisture content of spray-dried coconut treacle samples was determined according to standard oven method (AOAC, 1999).

Water activity
Water activity of powder samples was determined by using Hydro-thermometer data logger (water activity meter). The temperature was maintained at 24.5± 0.5°C.

X-ray diffraction
X-ray diffraction (XRD) studies were carried out on a Rigaku Ultima IV diffractometer with CoKα1 radiation. Diffractograms were taken between 8° and 50° (20).

Scanning electron microscopy (SEM)
Surface morphology of powder samples were observed by using LEO (LEO 1420VP) SEM operated at an accelerated voltage of 5.49 kV and the current of 2.387 A at a working distance of about 20 mm. The samples were gold plated by using gold sputter.

Sugar profile
Sample preparation
One gram of coconut treacle powder was dissolved in 50 ml of water. It was filtered through 0.45 µm syringe and injected to HPLC system (Shimadzu LC-10AS single pump, Shimadzu Corporation, Japan).

HPLC conditions
Twenty micro liters of diluted coconut treacle sample was injected onto a Supeleo Gel C 610H
analytical column, (300mm x 7.8mm), Supelco, USA at a temperature 30°C with a flow rate of 1.0 ml/min. Mobile phase was 0.1% H₃PO₄ (BDH, UK) and detection was carried out on a Shimadzu RID-10A, Shimadzu Corporation, Japan. All injections were made in triplicate.

**Keeping quality**

The spray-dried coconut treacle powder packed in triple laminated aluminium pouches was evaluated for the changes in moisture content and water activity at monthly intervals for a period of one year at ambient conditions (30±2°C).

**Statistical analysis**

All results were statistically analysed by one-way analysis of variance (ANOVA). Differences were considered significant at P < 0.05.

**Results and discussion**

**Powder recovery**

Figure 1 shows the visual observation of coconut sweet toddy powder.

The recovery of spray-dried treacle powder was determined (Figure 2). A zero powder recovery was observed in the case of formulation of treacle: drying aid: water in the ratio of 40:10:50 since all sugar solids in the formulation were lost as wall deposits. This indicates that 10% of drying aid was not sufficient to overcome stickiness. It is reported that wall deposition is a common occurrence in spray-drying and accounts for the major loss of product (Masters, 1991). Maltodextrin (DE-10) used in this research was to improve the yield of product by reducing surface stickiness of low molecular weight sugar and thus reduce wall deposition. When the drying aid percentage was increased from 10% to 15% and 20% the total recoveries rose to 60.2±2.90% and 57.8±3.5%, respectively. It is reported that the benchmark cyclone recovery for a marginally successful spray-drying process of sugar-rich material is more than 50% (Bhandari et al., 1997a; Bhandari et al., 1997b). Therefore, spray-drying of coconut treacle with both 15% and 20% maltodextrin (DE-10) separately yielded a higher amount of powder recovery compared to the bench mark recovery value. Since there was no significant difference (P>0.05) between the recoveries of treacle: drying aid: water in the ratio of 30:20:50 and 35:15:50, the formulation with the lower percentage of drying aid (15%) was selected for further analysis.

![Figure 1. Coconut treacle powder.](image)

![Figure 2. Powder recovery of spray-dried treacle powders.](image)
Crystalline/amorphous nature of spray-dried treacle powder

It can be seen from X-ray diffractogram in Figure 03 that the coconut treacle powder was amorphous. The amorphous nature of the powder may be due to very rapid evaporation and particle formation process as indicated by the low $a_w$ values (Table 01). The scanning electron micrograph (Figure 04) showed that particles of treacle: drying aid: water (35:15:50) powder were mostly spherical. The morphology of these particles gives a clear indication of the amorphous crystalline nature of the powders, spherical particles being amorphous and hexagonal particles being crystalline (Jayasundera et al., 2011; Ando et al., 2007).

Figure 3. XRD pattern of coconut treacle powder.

Figure 4. Scanning electron micrograph of coconut treacle powder.
Sugar profile
The sugar profile of coconut treacle powder showed that it had a total sugar content of 88.2±0.05% out of which 46.6±0.02% was sucrose, 23.0±0.02% was glucose and 18.6±0.01% was fructose (Figure 5).

Physical parameters of spray-dried coconut treacle
Table 01 shows the physical parameters of spray-dried coconut treacle on storage. The initial moisture content and water activity value of the spray-dried coconut treacle powder were 1.04±0.12% and 0.43±0.02%, respectively. The moisture content varied between 1.04±0.12% and 1.66±0.08% during the storage of one year. One of the characteristics of spray-dried products is the low moisture content which is less than 5% (Masters, 1991). Therefore, moisture content of treacle powder is well within the range. Water activity ($a_w$) is a very important index for spray-dried powder as it determines the shelf life of powder produced. Water activity measures the activity of free water in a food system which is responsible for any biochemical reaction. Higher water activity indicates that there is more free water available for biochemical reactions and thus the shelf life is shorter. Basically, a food with $a_w$ less than 0.6 is microbiologically stable and if any spoilage occurred, it is induced by chemical reactions rather than by micro-organisms (Goula, and Adamopoulos, 2005; Kampaipo et al., 2012). From the results (Table 1), the $a_w$ of treacle powder was in the range of 0.43±0.02 and 0.46±0.01 during the one year storage period. Therefore, the results reveal that spray-dried treacle was microbiologically stable during storage. It is reported that a spray drying process can produce a good quality final product with low water activity and reduce the weight, resulting in easy storage and transportation (Obón et al., 2009).

Table 1. Physical parameters of spray-dried treacle on storage.

<table>
<thead>
<tr>
<th>Time (months)</th>
<th>Moisture content (%) ±SD</th>
<th>Water activity ($a_w$) ±SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.04±0.12</td>
<td>0.43±0.02</td>
</tr>
<tr>
<td>1</td>
<td>1.08±0.09</td>
<td>0.43±0.01</td>
</tr>
<tr>
<td>2</td>
<td>1.14±0.10</td>
<td>0.44±0.02</td>
</tr>
<tr>
<td>3</td>
<td>1.15±0.08</td>
<td>0.44±0.01</td>
</tr>
<tr>
<td>4</td>
<td>1.24±0.14</td>
<td>0.44±0.02</td>
</tr>
<tr>
<td>5</td>
<td>1.37±0.11</td>
<td>0.45±0.03</td>
</tr>
<tr>
<td>6</td>
<td>1.39±0.13</td>
<td>0.45±0.01</td>
</tr>
<tr>
<td>7</td>
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</tr>
<tr>
<td>8</td>
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<td>0.46±0.01</td>
</tr>
<tr>
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</tr>
<tr>
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<td>0.46±0.01</td>
</tr>
<tr>
<td>12</td>
<td>1.66±0.08</td>
<td>0.46±0.01</td>
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</table>

Conclusions
More than 55% of amorphous coconut treacle powder was produced with 15% maltodextrin and the powder could be stored in triple laminated aluminium pouches for more than a year under ambient conditions (30±2°C). The amount of sucrose present in the powder was higher than the amounts of either glucose or fructose.

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References


