CHEMICAL ENGINEERING TRANSACTIONS

VOL. 57, 2017

A publication of AIDIC

The Italian Association of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Sauro Pierucci, Jiří Jaromír Klemeš, Laura Piazza, Serafim Bakalis

ISBN 978-88-95608- 48-8; **ISSN** 2283-9216

DOI: 10.3303/CET1757147

Production of Chocolate Powdered Beverage with Enhanced Instant Properties

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Instant properties of chocolate powder such as solubility and dispersibility are important factors for the quality and consequently consumer acceptance of the final powdered beverage. With the aim of producing a novel powdered chocolate beverage with enhanced instant solubility, we studied the effect of spray drying process on the components of the chocolate powder. Spray drying of chocolate beverage formulation (complete recipe provided and protected by Domori s.r.l., Italy) was performed using a mixture of cocoa powder and sucrose 30:70 (w/w). Liquid feed was prepared by dissolving 17.5 g of powder in 100 mL of deionized water at 60 °C and mixed until complete homogenization. The solution was fed to a mini spray dryer B-290 (Büchi, Huddersfield, UK). In this work an inlet temperature of 150 °C was used. Maltodextrin concentration (0, 3.5 and 7 % w/w) and drying air flow rate (22, 27 and 32 m^3/h) were varied. Powders were stored at 4 °C in closed dark vessels before analysis.
The results of full factorial design showed that operating at a drying air flow rate of 27 m^3/h and a maltodextrin concentration of 3.5 % w/w, powder with solubility of 74.2 ± 6.8% and dispersibility of 58.7 ± 2.9% was achieved. At this operative condition, powder recovery yield was almost consistent with the industrial one (45.5 ± 2.5%). Therefore, spray drying presents a useful way to produce chocolate powder with better instant solubility, which can also be dissolved in less amount of water (70 mL instead of 120 mL).

1. Introduction

For several years, the powdered beverage manufacturing has been considered one of the slowest growing food industries worldwide (Shittu and Lawal, 2007). Developing niche and healthy products as well as increasing the knowledge about product formulation are necessary to overcome this trend. Unfortunately, sufficient information about the effect of physical properties and chemical composition of the ingredients on the final characteristics of the beverage is at present scanty in the literature. Among the powdered drinks, instant hot chocolate plays an important role. Usually, this product mainly consists of milk, cocoa, sugar, emulsifier and small amount of flavours. Instant properties of chocolate powder, such as wettability and solubility, characterize the quality of the end use instant beverage and, as a consequence, of the consumer acceptance. According to the definition given by Schubert (1993), instant beverages are based on agglomerated powders that are supposed to be completely dissolved or dispersed in the specific amount of liquid (e.g. water or milk) after stirring for a short time. A good instantized beverage has no floating particles on the surface nor sediment at the bottom of the container after minimum stirring. The high fat content (10-25%) of the cocoa powder is one of the reasons which affects the wettability of the instant product (Wieland, 1972). The other factor can be attributed to the capillary structure of cocoa powder, which could obstacle the water penetration by trapping air pockets (Omobuwajo et al., 2000). To overcome those drawbacks, agglomeration has been considered as major factor to enhance the solubility of chocolate powder drink (Omobuwajo et al., 2000) since it increases the particle size thus shortening wetting time. However, not all the agglomeration processes (agglomeration of wetted single particles, agglomeration by drying, combination of both these methods, and agglomeration by compression) resulted in good instant solubilisation. The agglomerate size and structure should be characterized by high porosity (Kyaw Hla and Hogekamp, 1999), good dispersibility (Schubert, 1993) and also acceptable strength for handling and packaging (Hogekamp et al., 1996).

Please cite this article as: Aliakbarian B., Casazza A.A., Nani A., Perego P., 2017, Production of chocolate powdered beverage with enhanced instant properties, Chemical Engineering Transactions, 57, 877-882 DOI: 10.3303/CET1757147

Spray drying represents an alternative process to improve the reconstitution properties of chocolate powder beverage. Spray drying is the most widely used commercial method for food drying processes, giving a high quality powder product with enhanced instantaneous solubility with relatively low cost (Paini et al., 2015). Working with very short time of heat contact and high rate of evaporation, this process ensures the nutritive value of the components. Maltodextrin is one of the most commonly used carriers in the process of spray drying due to its high solubility in water (Cano-Chauca et al., 2005). To the best of our knowledge, the use of spray drying and maltodextrin as carrier has not been vastly investigated for the preparation of chocolate powdered beverage. As is evidenced throughout the world, the demand of novel and healthy foods is growing. For this reason, the aim of this study was to produce a novel chocolate powdered beverage with enhanced instantized properties, which can be dissolved completely in lower amount of water (70 mL) compared to the traditional quantity (about 120 mL). Effect of spray drying operative parameters such as drying air flow rate, the concentration of maltodextrin and its effect on the physico-chemical and instantizing properties of main components of the chocolate drink were investigated.

2. Materials and methods

2.1 Reagents and ingredients

Cocoa and sucrose powders were kindly furnished by the Italian company Domori s.r.l.. Maltodextrin (16.5-19.5 dextrose equivalents), acetic acid, methanol and acetonitrile (HPLC grade) were purchase from Sigma-Aldrich (Milan, Italy).

2.2 Spray drying process

We used spray drying process to produce soluble chocolate powders. Spray drying of chocolate beverage formulation (recipe provided and protected by Domori s.r.l., Italy) was performed using a mixture of cocoa powder and sucrose 30:70 (w/w). Liquid feed was prepared by dissolving 17.5 g of powder in 100 mL of deionized water at 60 °C and mixed until complete homogenization. The solution was fed to a mini spray dryer B-290 (Büchi, Huddersfield, UK). Spray dryer was run with water for 10 min before and after each experiment. In this work an inlet temperature of 150 °C was used. Preliminary studies performed in our laboratory indicated that an inlet temperature lower than 130 °C causes the formation of a wet film on the walls of the chamber, while a temperature higher than 160 °C leads to an excessive denaturation of the product (Aliakbarian et al., 2015a; Paini et al., 2015). Maltodextrin (MD) concentration and drying air flow rate ranges were selected based on literature review and taking into account the minimum alteration of the product sample (Cano-Chauca et al., 2005; Jinapong et al., 2008;). In experimental design tests, aspiration rate was changed from 22 to 32 m^3/h and different percentages (0, 3.5 and 7% w/w) of MD were added to the initial formulation, reducing the sucrose percentage from 70 to 66.5 and 63.0 %, respectively. Powders were stored at 4 °C in closed dark vessels before analysis.

2.3 Powder characterization

Powder recovery yield (%) after spray drying process was defined as the ratio of the mass of powder recovered from the cyclone to the solids originally presented in the feed solution. We did not consider powder attached to the heating vessel as part of the yield because of the very dark color and the burned aspect. The moisture content of the spray dried samples, expressed as percentage, was calculated based on the weight loss of the sample (0.5 g) after drying at 105 °C for 6 h. Solubility, as an important instant property of the powder, was determined based on the method described by (Shittu and Lawal, 2007) with some modifications. Briefly, approximately 2.0 g of each sample were mixed with 20 mL of water at 80 °C and stirred for 30 min. The suspension was then centrifuged (ALC PK131, Alberta, Canada) at 9500 rpm for 10 min and the supernatant was collected. The weight of the solids in the supernatant after drying at 105 °C was used as the solubility index and expressed as percentage of solubility in water. For dispersibility index, about 10.0 g of each powder were used. Samples were manually stirred with 100 mL of water at 27 °C for 1 min. The suspension was maintained firmed for 30 min. Fifty millilitres of the supernatant containing dispersed solids were separated and weighed. The dispersibility index expressed as the percentage was calculated using the following equation (Eq(1)).

$$Dispersibility\ (\%) = 2 * (((M2 - M1))/M0) * 100) \qquad (1)$$

Where, M2 is the weight of 50 mL of supernatant (g), M1 is weight of 50 mL of distilled water (g), and M0 is the powdered sample (g) (Shittu and Lawal, 2007). Scanning electron microscopy (SEM 515, Philips, Netherlands) was used to evaluate the microstructure of the particles. Appropriate amounts of powders were coated with a 30 nm-thick gold layer and observed in secondary electrons (5.0 kV) at 300x magnification. In order to quantify the sugar and cocoa percentages in the spray dried powder, the sucrose after spray drying process was analysed by HPLC. For the analysis a HPLC 1100 (Hewlett Packard, Palo Alto, USA) equipped with a Hi-Plex H column (300 x 7.7 mm) (Phenomenex, Torrance, CA, USA) was used following the methodology described by (Aliakbarian et al., 2015b). The mobile phase was 0.5% v/v of sulfuric acid 1 M in Milli-Q water, the flux was set at 0.5 mL/min and the column temperature was set at 50 °C. Before the injection, 1.0 g of spray dried sample was dissolved in 100 mL of hot water (70 °C), mixed for 30 min and centrifuged at 10000 rpm for 10 min. The sucrose concentration was evaluated comparing the peak sequence and intensity to a 0.5 g/L sucrose standard solution. The cocoa powder percentage in the product was theoretically calculated using the following equation (Eq. 2):

$$Cocoa\ (\%) = 100 - sucrose\ (\%) - M_D\ (\%) \qquad (2)$$

2.4 Statistical analysis

All the experiments were carried out in triplicate (n = 3). ANOVA and Tukey's post hoc test ($p < 0.05$) were used ("Statistica 10.0", StatSoft, Tulsa, USA).

3. Results and discussions

Table 1 shows the matrix design of the independent variables according to the 3^2 full factorial design and the experimental results. In every sample, the moisture content resulted in being less than 7% (almost in the acceptable range supporting microbiological stability). The highest moisture content was achieved using the maximum air drying flow rate (32 m^3/h) and MD concentration (7% w/w). This moisture content can be attributed to the protecting effect of MD as coating agent by interpolating more quantity of water in the particles.

Table 1: Full factorial design matrix and the responses of the dependent variables

Runs	Independent variables		Responses			
	X1	X2	Recovery (%)	Moisture (%)	Solubility (%)	Dispersibility (%)
1	22.0	0.0	18.3±1.7b	2.9±0.01f	66.0±5.2a	67.8±0.1b,d
2	27.0	0.0	44.2±2.5a	0.0±0.03b	75.0±7.8a	54.4±1.2a
3	32.0	0.0	45.4±4.1a	3.6±0.05a	69.0±10.1a	43.0±4.1c
4	22.0	3.5	33.2±3.2c	1.0±0.04c	75.5±8.9a	23.8±0.9e
5	27.0	3.5	45.5±2.5a	4.9±0.02g	74.2±6.8a	58.7±2.9a,b
6	32.0	3.5	49.2±5.2a	2.0±0.03d	78.0±11.2a	56.5±4.7a
7	22.0	7.0	23.3±1.7b	2.7±0.06e	73.0±12.5a	58.4±3.4a,b
8	27.0	7.0	48.2±4.8a	3.5±0.08a	72.0±11.4a	74.5±5.6d
9	32.0	7.0	45.2±3.4a	6.7±0.09h	74.5±13.8a	40.8±3.2c

X1: Drying Air flow rate (m^3/h); X2: Maltodextrin concentration (% w/w). Different letters within the columns indicate significant difference between data at $p < 0.05$. Data are expressed as mean value ± standard deviation, n = 3.

As concern the solubility, powders showed good solubility properties, with values ranging from 66 % (without addition of MD) to 78 % when 3.5 % w/w of MD was used. Addition of higher quantity of MD did not increase the solubility of the powders ($p<0.05$). Regard to the dispersibility index, using an intermediate air drying flow rate (27 m^3/h), the increase of the concentration of MD from 0.0 to 7.0 % w/w enhanced this property of the final product up to 37.02%. Results of regression analysis demonstrated that the drying air flow rate significantly influenced ($p<0.05$) the recovery yield of the powdered product (Figure 1). For this reason, the effect of those variables has been studied using response surface modelling (RSM). RSM, as a combination of statistical and mathematical techniques, enables the evaluation of different parameters and their interaction on response variables (De Faveri et al., 2007). This methodology has been widely used for the optimization of food processes such as olive oil (Aliakbarian et al., 2008), chocolate-flavored and peanut soy beverages (Deshpande et al., 2008) and winery industry (Casazza et al., 2012).

Statistical prediction revealed that at critical values of drying air flow rate and MD concentration of 29.65 m^3/h and 4.0 % w/w, respectively, the powder recovery yield will be maximum and equal to 54.43%. At this condition, working with reasonable amount of drying aid, the recovery yield is industrially acceptable (Abad and Turon, 2012).

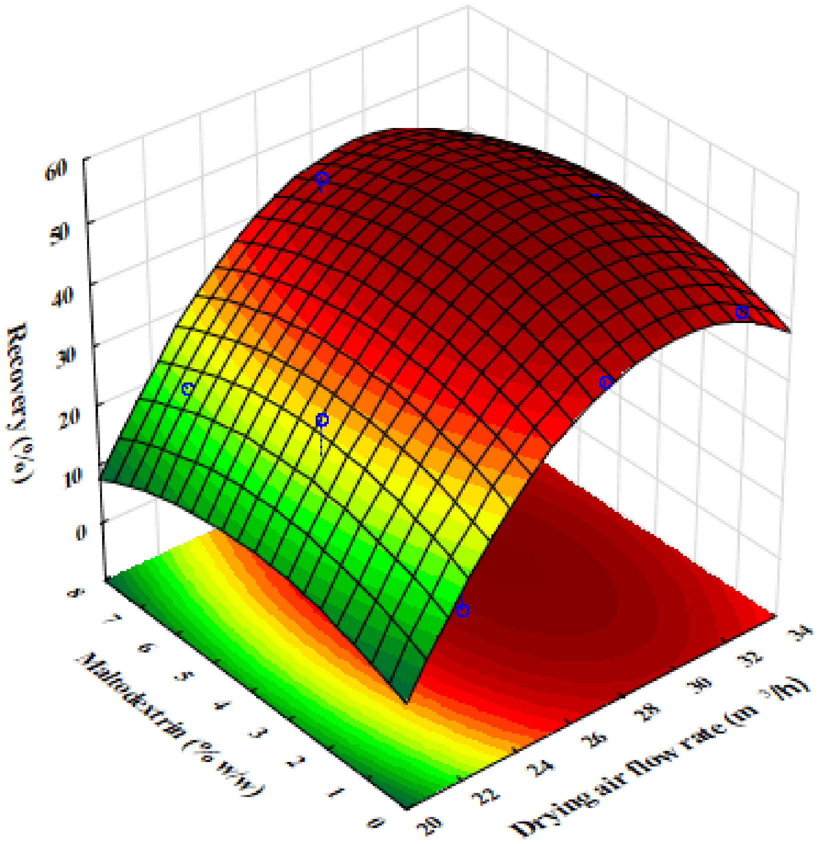


Figure 1: Response surface of powder recovery yield (%) as a simultaneous function of drying air flow rate (m^3/h) and maltodextrin concentration (% w/w) (Recovery (%) = -314.00 + 24.26 X1-0.41 X12 + 3.39 X2 - 0.42 X22).

SEM images were used to evaluate the effect of MD concentration and the drying air flow on the structure of the microparticles. Figure 2 shows the presence in all systems of hexagonal particles (sucrose) among spherical ones, whether or not the addition of MD. In the presence of 3.5% w/w of MD and 27 m^3/h of drying air flow (Fig. 2 b), the particles showed a good degree of uniformity without strong adhesion among them. This can be due to the crystallization effect of spray drying in the presence of MD as a carrier to induce this phenomenon (Cano-Chauca et al., 2005). While aggregation of the particles has been noted in the samples containing 7% w/w of MD (Fig. 2 c, and f). This aggregation was more evident with a drying air flow equal to 32 m^3/h (Fig. 2 f). This result is validated by the moisture content (6.66%) in these samples as well. Such a result could be due to the greater quantities of water that can be entrapped inside high amounts of drying agent, which generates bigger particles. The same behavior was also noticed by Paini et al. (2015) in the spray drying of olive pomace extract using MD concentration of 500 g/L and 10 mL/min of feed flow at 150 °C. At this condition, the authors observed bigger microparticles than the ones obtained with 100 g/L of MD (keeping the same operative conditions). When they used 100 g/L of MD, 99% of the microcapsules showed a diameter range less than 20 μm, while with 500 g/L of MD only 29% were in the same range of dimension.

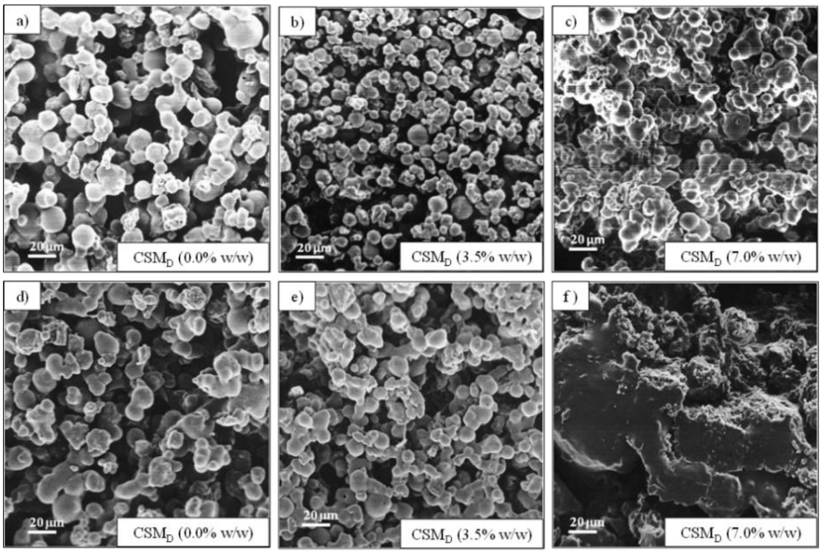
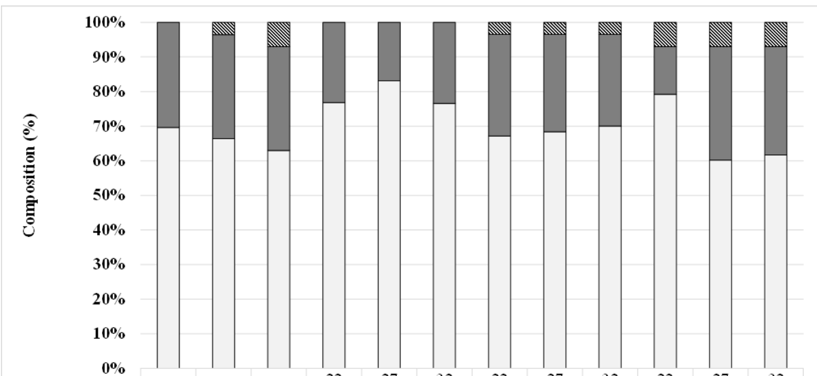


Figure 2: SEM images (300x) of the particles obtained by spray drying at inlet temperature of 150 °C varying maltodextrin (MD) concentration (0.0%, 3.5 and 7.0 w/w) and drying air flow (27 and 32 m^3/h). Images on top (a, b, and c) refer to drying air flow of 27 m^3/h. Images on the bottom (d, e, and f) refer to drying air flow of 3^2 m^3/h. C: cocoa, S: sucrose.

To assess the effect of spray drying process on the final chocolate powder formulation, we calculated the sucrose percentage by HPLC before and after the drying process.

As can be seen in Figure 3, the change in sucrose percentage was more evident in the samples without MD with an increment of 7, 13, and 7% at an air flow of 22, 27, and 32 m^3/h, respectively when compared to sample A (before the drying process). Moreover, a notable increment of sucrose compared to the sample before spray drying (C) was noticed (16%) using 7% w/w of MD and 22 m^3/h of drying air flow. The best results were obtained using 3.5% w/w of MD. In this condition and varying the drying air flow, the content of sucrose remain almost the same as the initial composition before spray drying (1% of difference was noticed with respect to sample B).

Considering the sucrose percentage in the products and taking into account the hypothesis that MD will be totally recovered after the spray drying process, the content of cocoa resulted to be notably decreased in samples without the addition of MD and in the one containing 7% of MD spray dried at 22 m^3/h. The variation of cocoa quantity in the final product can cause unpleasant taste and as consequence consumer dissatisfaction.



	A	B	C	22 m3/h	27 m3/h	32 m3/h	22 m3/h	27 m3/h	32 m3/h	22 m3/h	27 m3/h	32 m3/h
Maltodextrins	0.0	3.5	7.0	0.0	0.0	0.0	3.5	3.5	3.5	7.0	7.0	7.0
Cocoa	30	30	30	23	17	23	29	28	26	14	33	31
Sucrose	70	66	63	77	83	77	67	68	70	79	60	62

Figure 3: The composition percentage of the product containing principally sucrose and cocoa powder and maltodextrin as a carrier quantified by HPLC. In the spray drying process maltodextrin was varied from 0 to 7% w/w and drying air flow from 22 to 32 m^3/h. Samples A, B and C are analyzed before spray drying process.

4. Conclusions

The approach taken in this study was to exploit the effect of spray drying process on instant properties of chocolate powder beverage. We demonstrated the ability of producing particles with good solubility (more than 70%) and dispersibility (higher than 50%) remaining in an acceptable industrial range of product yield (around 50%) and moisture content (less than 6%). The use of maltodextrin resulted to protect the composition of spray dried component (when operating at 27 m^3/h as drying air flow) producing uniform and separate particles. These findings can be potentially used in powder beverage production industries to enhance the solubility properties.

Acknowledgments

The authors wish to thank Dr. Alberto Lagazzo for his support and help for SEM imaging.

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