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Influence of the process parameters and sugar granulometry on cocoa beverage powder steam agglomeration

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ABSTRACT

The objective of this research was to evaluate the effects of the steam agglomeration process variables on the characteristics of cocoa beverage powders. A pilot scale agglomerator was used in the tests. For the cocoa beverage powder formulated with granulated sugar, most common commercial product, the increase in solids feed rate (400–700 g/min) led to a decrease in the mean particle diameter (400 g/min – 564.70 μm, 700 g/min – 438.40 μm) and an increase in the dryer rotation (12–52 rpm) led to an increase in the product moisture (12 rpm – 1.52% w.b., 52 rpm – 1.88% w.b.). The changing from 1.0×10^2 to 1.8×10^2 kPa of the vapor pressure resulted in an increase in moisture of the cocoa beverage powder (1.0×10^2 kPa – 1.46% w.b., 1.8×10^2 kPa – 1.94% w.b.) and the intensification of the yellow color of the product (1.0×10^2 kPa – 14.51, 1.8×10^2 kPa – 15.17).

1. Introduction

In Brazil the majority of the cocoa beverage powders found on the market are formulated with sugar, which can be crystal or granulated, maltodextrin and cocoa powder, and can also contain skimmed milk powder and milk whey. The amount of cocoa used in the formulations generally varies from 5% to 20%, sufficient to make the powders insoluble and difficult to reconstitute in liquid, water or milk. The alkaline type of cocoa powder (pH between 6.2 and 7.5) is generally used in cocoa beverage powders, and although this shows improved dispersability as compared to natural cocoa powder (pH between 5.0 and 5.9), the presence of, on average, from 10% to 12% of cocoa butter, compromises reconstitution of the cocoa beverage powders formulated with this ingredient.

Agglomeration is an alternative process that can be used to improve the reconstitution properties of cocoa beverage powders in liquids. This process has been used as an "instantizer" for dairy products (hot chocolate, milk powder for ice-creams), other beverages (coffee, tea) and for starch-based products (soups, sauces and baby foods). Ingredients with wide applications in the food industry can also be agglomerated, such as enzymes and yeasts used in bakery products, maltodextrin, milk proteins and acacia gum, amongst others. The objective of agglomeration is to improve the dispersion and solubility properties of the powders in addition to reducing the fines in the final product and during handling on the manufacturing lines. The process also makes it possible to produce more attractive powdered products (both in appearance and sensory properties) and decrease the apparent density, as well as reducing the tendency to cake during storage (Iveson et al., 2001; Knight, 2001; Maurel, 1994; Palzer, 2005). Various techniques can be used to agglomerate cocoa beverage powders, amongst which the following stand out: steam agglomeration (Hogekamp et al., 1996; Hogekamp, 1999a,b; Hla and Hogekamp, 1999), fluid bed agglomeration (Kowalska and Lenard, 2005) and thermal agglomeration (Omobuwajo et al., 2000). High shear mixers have also been used for the wet granulation of cocoa powder (Vu et al., 2003).

Steam agglomeration is a complex process and involves many process variables and product characteristics. Of the process variables, the most important are the agglomerator feed conditions (solids flow, steam flow) and the drying conditions (temperature and residence time in the dryer). With respect to product characteristics, it is of fundamental importance to know the particle size and distribution, the moisture content and the intrinsic characteristics of the material (chemical composition and proportion of soluble components) in addition to the powder flow properties.

The main objective of this research was to evaluate the effects of the process variables in the steam agglomeration of cocoa beverage powder formulated with crystal and granulated sugar. The agglomerator used consisted of a moisturizing chamber and a rotary dryer. The following process variables were used: vapor

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Nomenclature

a^*	color parameter – red to green	R_d	dryer rotation (rpm)
b^*	color parameter – yellow to blue	T_a	air temperature of rotary dryer (°C)
D_p	mean particle diameter (μm)	U	moisture content (% wet basis)
I	insolubility (%)	V_s	solids feed rate (g/min)
L^*	color parameter – black to white	W	wettability (s)
P_v	vapor pressure (kPa)		

pressure, solids feed rate, drying air temperature and the rotational velocity of the dryer. This research proposed to study cocoa beverage powders formulated from the ingredients and additives normally used in commercial products using formulations similar to those available on the Brazilian market. The formulated products were analyzed both before and after steam agglomeration, with respect to their chemical, physical and physicochemical food powder characteristics: moisture content, mean particle diameter, wettability, insolubility and color.

2. Materials and methods

2.1. Materials

The cocoa beverage powders used in this study had the following composition: 80% sugar (crystal or granulated), 12% corn maltodextrin (17–19.9 D.E.) and 8% cocoa powder (alkaline; 11–12% cocoa butter). Natural, low viscosity soybean lecithin (T = 25 °C, 2900 cP) was also used.

2.2. Pre-treatments

The crystal sugar used in the formulation was ground in a hammer mill (Treu; 3.2 mm^2 plate) to a mean particle diameter of 300 μm. On the other hand, the granulated sugar was obtained milling crystal sugar twice and then it was sieved (1.19 mm mesh). The crystals that passed through the sieve were used in the formulations (mean particle diameter of granulated sugar: 100 μm). The components of the milk chocolate powder were then mixed in a ribbon blender (20 L of powder), mixing at 25 °C and 120 rpm for 20 min. Lecithinization was carried out in the same equipment under the same conditions used to mix the ingredients. The lecithin content sprayed in was 0.3%, as indicated by Vissotto et al. (2006) for agglomerated cocoa beverage powders.

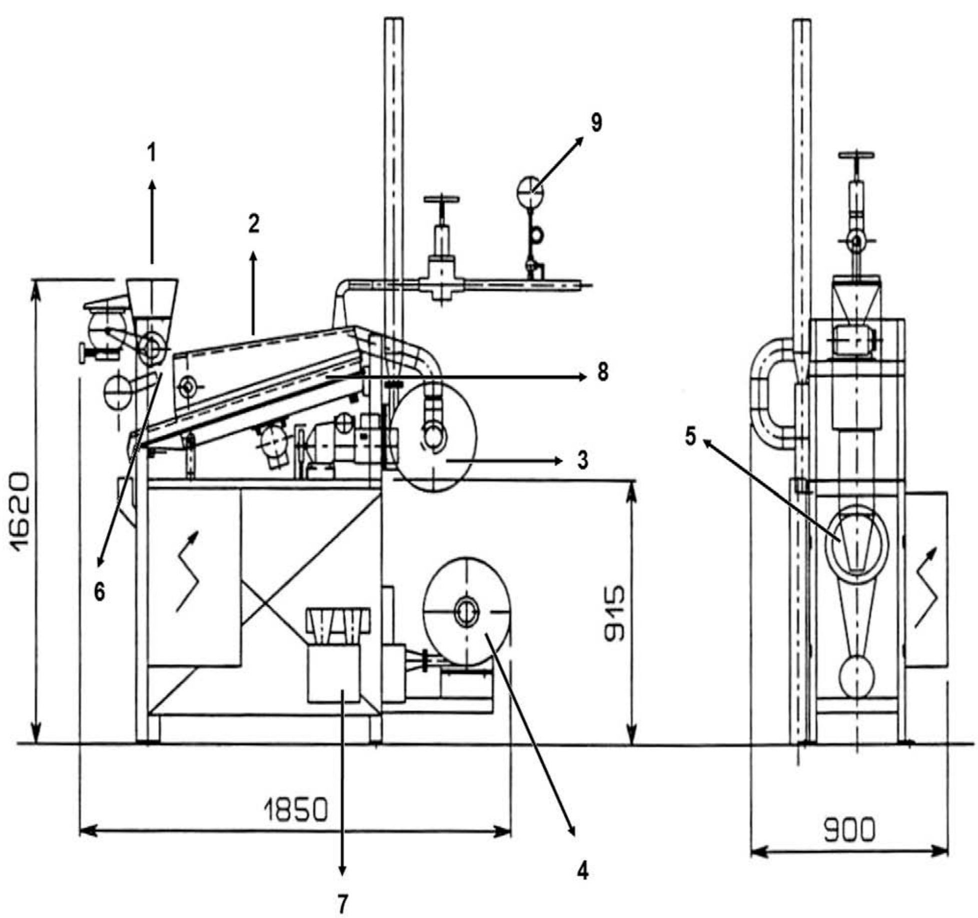

Fig. 1. Schematic design of the ICF Industrie Cibec pilot agglomerator, where (1) is the feed hopper, (2) the heated vapor and fines aspiration system, (3) the exhaust, (4) the air fan for the rotary heater, (5) the rotary heater, (6) the vapor diffuser, (7) the agglomerate classifier, (8) a 45° inclined vibrating screen, (9) steam manometer.

Table 1
Values and levels used in the fractionated factorial designs.

Variables	Levels		
	−1	0	+1
P_v[a] ($\times 10^2$ kPa)	1.0	1.4	1.8
T_a[b] (°C)	90	95	100
V_s[c] (g/min)	400	550	700
R_d[d] (rpm)	12	32	52

Constant drying air speed = 1.45 m/s.
[a] Vapor pressure.
[b] Rotary dryer air temperature.
[c] Solids feed rate.
[d] Dryer rotation.

2.3. Equipment

A steam agglomerator was used in this study (ICF Industrie CIBEC s.p.a., Italy; type: mini-instant pilot), (Fig. 1). The material was fed into the agglomerator by means of a vibrating dosing chute (M.V.L. Máquinas Vibratórias Ltda, Brazil; type: FTO), the powder flow being directed towards the center of the feed hopper (1), on the inside of which there was a rotary brush that forced the passage of the powder against a perforated screen (6.25 mm^2 mesh). The cocoa beverage powder, falling in a descending direction, was moistened by the steam coming from the diffuser (6). The granules formed were fed into the rotary dryer (5), and after the drying stage, were classified according to their size (7). The agglomerator operated in the following ranges of the variables: vapor pressure – 1.0×10^2 to 1.8×10^2 kPa; solids feed rate – 400–700 g/min; dryer rotation – 12–52 rpm; rotary dryer temperature – 25–100 °C.

2.4. Sample characterization

The moisture content was determined in triplicate using the Karl Fischer Equipment (Schoot Gerate, Titroline Alfha, Germany), according to the official A.O.A.C. International Method (2005) and Bruttel and Schilink (2003). The mean particle diameter, determined in triplicate, was obtained by sieving 100 g of cocoa beverage powder through seven steel sieves (mesh between 0.15 and 1.19 mm), using the mechanical agitation system (Produtest, Brazil). Wettability was evaluated by an adaptation of the method of Hla and Hogekamp (1999), being considered as the time necessary for 2.0 g of powder deposited on the surface of the liquid to become completely submersed in 400 g of distilled water at 25 °C. The color parameters were measured using CIELAB system (L^* a^* b^*; for 2° Standard Observer; Standard Illuminant D_{65}) in the colorimeter (Konica Minolta, model CR-410, Japan), equipped with an adaptor for granulated and powdered materials (CR-A50). In each case, 10 samples were evaluated for wettability, and for color, 10 replicates were carried out for each sample, analyzing three samples.

2.4.1. Insolubility

The objective of this analysis was to quantify the amount of insoluble compounds in the products studied, and consisted of adding 20.0 g cocoa beverage powder to 150 mL distilled water (at 25 °C) in a 600 mL beaker. The dilution used in this method was the same as that employed in the preparation of commercial hot chocolate drinks in Brazil. The powder was then mixed with the liquid using a digital mechanical stirrer (330 rpm, 20 s) and a "helix" especially developed for this analysis as described by Vissotto et al. (2006). The solution obtained was filtered in a vacuum system consisting of a Buchner funnel and vacuum conical flask. The mass retained on the paper was dried in an oven (60 °C) to constant weight, and subsequently weighed on an analytical balance. The insolubility (% dry basis) was obtained from the ratio between the mass of sample retained on the paper, that had not dissolved, and the initial mass of the sample. The determinations were carried out in triplicate.

2.5. Effects of the process variables

Two fractionated experimental designs from Resolution IV (2^{4-1}, plus 3 central points) were used to evaluate the effects of the process variables. The first design was applied to the products formulated with crystal sugar and the second for products formulated with granulated sugar, with a total of 22 trials. The variables studied were: vapor pressure (P_v), air temperature in the rotary dryer (T_a), solids feed rate (V_s) and dryer rotation (R_d). Table 1 shows the values and levels applied in the fractionated factorial designs. The responses determined for the experimental designs were: moisture content (U), mean particle diameter (D_p), insolubility (I), wettability (W) and the color parameters (L^* a^* b^*). The results were analyzed using the Statistica 7.0® software, the effects of the process variables on the responses being obtained and the significance evaluated at a 90% confidence level ($p \leqslant 0.10$) (Rodrigues and Iemma, 2005; Haaland, 1989).

2.6. Product quality at milder process condition

Considering the results obtained in the fractionated designs, further trials were carried out with the objective of evaluating the physical and physicochemical characteristics of the cocoa beverage powders produced under the most economic steam agglomeration conditions, that is, lowest vapor pressure and dryer rotation ($P_v = 1.0 \times 10^2$ kPa; $R_d = 12$ rpm) that also represent less aggressive ones. Solids feed rate was fixed at 400 g/min. With respect to the drying temperature, operational ranges lower than the lowest ones used in the prior experimental designs were evaluated (T_a = 40, 60 and 80 °C), with a view to observing the characteristics of the products obtained and the drying efficiency under milder temperature conditions. The cocoa beverage powder formulation used in this study was: 80% granulated sugar, 12% corn maltodextrin (17–19.9 DE) and 8% cocoa powder.

3. Results and discussion

3.1. Effects of the process variables on steam agglomeration

According to the experimental designs proposed, steam agglomeration trials were carried out considering the following process variables: vapor pressure, rotary dryer air temperature, solids feed rate and dryer rotation. The responses obtained – mean particle diameter, moisture content, wettability, insolubility and color (L^* a^* b^*), for the first design (cocoa beverage powder formulated with crystal sugar) and second design (cocoa beverage powder formulated with granulated sugar) are presented in Tables 2 and 3, respectively, as well as the characterization of the cocoa beverage powders before steam agglomeration (control). Table 4 shows the effects of the process variables on the responses and their mean values (obtained by the arithmetic mean of 11 tests presented in Tables 2 and 3), and Figs. 2 and 3 show the significance ($p \leqslant 0.10$) of the effects in the form of Pareto Diagrams for the first and second designs, respectively.

For the cocoa beverage powder formulated with crystal sugar, an increase in vapor pressure from 1.0×10^2 to 1.8×10^2 kPa resulted in growth of the granules, the effect being statistically significant ($p \leqslant 0.10$) (Fig. 2) and equal to 25.35 µm (Table 4).

Table 2
Results of the experimental design (2^{4-1}, plus 3 central points) for the evaluation of the variables of vapor pressure (P_v), rotary dryer temperature (T_a), solids feed rate (V_s), dryer rotation (R_d) in the steam agglomeration process for cocoa beverage powder formulated with crystal sugar.

Trials	P_v	T_a	V_s	R_d	First design – crystal sugar						
					D_p[a] (μm)	U[b] (% w.b.)	W[c] (s)	I[d] (%)	Color		
									L^*[e]	a^*[f]	b^*[g]
1	−1	−1	−1	−1	451.81 ± 3.40	1.65 ± 0.04	1.66 ± 0.05	7.15 ± 0.30	48.28 ± 0.11	11.82 ± 0.02	14.42 ± 0.05
2	+1	−1	−1	+1	512.65 ± 3.53	1.41 ± 0.06	1.45 ± 0.10	7.35 ± 0.35	46.65 ± 0.21	11.65 ± 0.08	14.22 ± 0.06
3	−1	+1	−1	+1	511.70 ± 3.60	1.33 ± 0.03	1.07 ± 0.10	7.67 ± 0.42	47.48 ± 0.20	11.81 ± 0.05	14.31 ± 0.07
4	+1	+1	−1	−1	511.01 ± 3.48	1.25 ± 0.05	1.50 ± 0.10	7.60 ± 0.37	46.86 ± 0.11	11.68 ± 0.04	14.15 ± 0.08
5	−1	−1	+1	+1	416.28 ± 3.65	1.52 ± 0.06	1.10 ± 0.10	7.00 ± 0.36	49.82 ± 0.20	11.56 ± 0.02	14.35 ± 0.02
6	+1	−1	+1	−1	413.18 ± 3.43	1.20 ± 0.04	1.16 ± 0.05	7.33 ± 0.39	49.01 ± 0.24	11.68 ± 0.02	14.69 ± 0.08
7	−1	+1	+1	−1	409.03 ± 3.27	1.06 ± 0.04	1.13 ± 0.05	7.36 ± 0.34	50.37 ± 0.24	11.46 ± 0.08	14.47 ± 0.07
8	+1	+1	+1	+1	453.38 ± 3.72	1.28 ± 0.05	1.41 ± 0.08	7.74 ± 0.37	48.49 ± 0.11	11.68 ± 0.04	14.38 ± 0.05
9	0	0	0	0	457.72 ± 3.65	1.61 ± 0.03	1.19 ± 0.10	7.65 ± 0.43	48.10 ± 0.20	11.74 ± 0.05	14.48 ± 0.06
10	0	0	0	0	483.11 ± 3.44	1.41 ± 0.06	1.14 ± 0.05	7.41 ± 0.38	48.13 ± 0.24	11.95 ± 0.02	14.69 ± 0.06
11	0	0	0	0	486.39 ± 3.52	1.34 ± 0.04	1.17 ± 0.08	7.90 ± 0.37	47.80 ± 0.24	11.86 ± 0.08	14.43 ± 0.08
Control[h]	–	–	–	–	438.12 ± 3.02	3.65 ± 0.06	19.63 ± 1.55	7.69 ± 0.40	57.76 ± 0.20	7.80 ± 0.10	10.87 ± 0.07

Result: mean ± standard error.
[a] Mean particle diameter.
[b] Moisture content.
[c] Wettability.
[d] Insolubility.
[e] Luminosity.
[f] Red.
[g] Yellow.
[h] The control is the experimental condition carried out without agglomeration.

Table 3
Results of the experimental design (2^{4-1}, plus 3 central points) for the evaluation of the variables of vapor pressure (P_v), rotary dryer temperature (T_a), solids feed rate (V_s), dryer rotation (R_d) in the steam agglomeration process for cocoa beverage powder formulated with granulated sugar.

Trials	P_v	T_a	V_s	R_d	Second design – granulated sugar						
					D_p (μm) [a]	U (% w.b.) [b]	W (s)[c, i]	I (%)[d]	Color		
									L^*[e]	a^*[f]	b^*[g]
1	−1	−1	−1	−1	508.87 ± 2.88	1.45 ± 0.04	–	8.22 ± 0.30	46.63 ± 0.24	11.99 ± 0.06	14.29 ± 0.11
2	+1	−1	−1	+1	522.47 ± 2.90	1.90 ± 0.05	–	8.14 ± 0.15	47.83 ± 0.09	12.09 ± 0.03	14.99 ± 0.02
3	−1	+1	−1	+1	521.29 ± 2.42	1.55 ± 0.04	–	7.44 ± 0.40	48.36 ± 0.09	12.00 ± 0.02	14.67 ± 0.02
4	+1	+1	−1	−1	546.02 ± 3.00	1.74 ± 0.05	–	7.68 ± 0.40	47.26 ± 0.28	12.10 ± 0.06	14.88 ± 0.10
5	−1	−1	+1	+1	450.42 ± 3.10	1.78 ± 0.04	–	7.86 ± 0.30	48.79 ± 0.22	11.93 ± 0.06	14.70 ± 0.10
6	+1	−1	+1	−1	445.86 ± 2.70	1.72 ± 0.05	–	7.80 ± 0.15	47.37 ± 0.09	12.02 ± 0.03	14.77 ± 0.02
7	−1	+1	+1	−1	468.33 ± 2.26	1.45 ± 0.05	–	8.17 ± 0.15	48.43 ± 0.22	11.78 ± 0.02	14.69 ± 0.05
8	+1	+1	+1	+1	481.44 ± 3.40	1.84 ± 0.05	–	7.95 ± 0.30	47.70 ± 0.28	12.08 ± 0.03	15.03 ± 0.11
9	0	0	0	0	529.30 ± 3.74	1.74 ± 0.03	–	7.96 ± 0.40	48.55 ± 0.09	12.15 ± 0.02	15.12 ± 0.02
10	0	0	0	0	508.79 ± 2.65	1.76 ± 0.04	–	7.87 ± 0.30	49.95 ± 0.24	11.94 ± 0.03	15.25 ± 0.04
11	0	0	0	0	534.31 ± 3.14	1.80 ± 0.03	–	7.85 ± 0.15	47.43 ± 0.09	12.16 ± 0.02	14.87 ± 0.02
Control[h]	–	–	–	–	396.35 ± 2.58	3.53 ± 0.03	11.79 ± 1.56	7.04 ± 0.30	57.87 ± 0.25	7.83 ± 0.08	11.18 ± 0.11

Result: mean ± standard error.
[a] Mean particle diameter.
[b] Moisture content.
[c] Wettability.
[d] Insolubility.
[e] Luminosity.
[f] Red.
[g] Yellow.
[h] The control is the experimental condition carried out without agglomeration.
[i] For all samples the wetting time was less than 1 s, not being possible to quantify with the methodology employed.

Considering that the increase in vapor pressure implied an increase in the feed rate of the steam and hence in the size of the droplets, this favored the formation of a greater number of inter-particle liquid bridges, leading to the production of larger granules. The increase in vapor pressure also implied a decrease in luminosity of the hot chocolate (color parameter L^*), the effect being statistically significant and equal to 1.24 (Fig. 2 and Table 4).

The air temperature of the rotary dryer was a statistically significant process variable ($p \leqslant 0.10$) for the responses of mean particle diameter, moisture content and insolubility, as can be seen from the results presented in the Pareto Diagrams in Fig. 2. A considerable size increase in the granules formed was shown in the temperature range tested, the effect being equal to 22.80 μm for an increase from 90 to 100 °C (Table 4). A certain amount of agglomeration may possibly have occurred during the drying step, especially in the first drying stages, when the still moist material would have had greater chances of contact due to rotation of the dryer, or could even have led to the formation of more resistant solid bridges due to the higher temperatures, favoring a decrease in the rate of breakage of the dry granules. The moisture content of

Table 4
Effects of the process variables on the characteristics of the cocoa beverage powders formulated with crystal sugar (first design) and granulated sugar (second design).

Variables	D_p (μm)	U (% w.b.)	W (s)	I (%)	Color		
					L^*	a^*	b^*
Crystal sugar							
Mean	464.21*	1.37*	1.27*	7.47*	48.27*	11.72*	14.42*
P_v (× 10² kPa)	25.35*	−0.11	0.14	0.21	−1.24*	0.01	−0.03
T_a (°C)	22.80*	−0.22*	−0.07	0.39*	−0.14	−0.02	−0.09
V_s (g/min)	−73.83*	−0.15	−0.22	−0.09	2.11*	−0.15	0.20
R_d (rpm)	27.25*	0.10	−0.11	0.08	−0.52*	0.02	−0.12
Granulated sugar							
Mean	501.55*	1.70*	–	7.90*	48.03*	12.02*	14.84*
P_v (× 10² kPa)	11.72	0.24*	–	−0.03	−0.51	0.15	0.33*
T_a (°C)	22.37	−0.07	–	−0.20	0.28	−0.02	0.13
V_s (g/min)	−63.15*	0.04	–	0.08	0.55	−0.09	0.09
R_d (rpm)	1.64	0.18*	–	−0.12	0.75	0.05	0.19

* $p < 0.1$, (corresponds to 90% confidence level).

the cocoa beverage powder decreased with increase in temperature of the rotary dryer, as expected during a drying process, the negative effect being 0.22% (Table 4). There was also a slight increase in product insolubility with increase in dryer temperature, the effect being 0.39% (Table 4). Solid bridges probably formed between the insoluble compounds of the cocoa beverage powder in the drying range studied, especially between the cocoa solids and the crystal sugar, which would be more resistant and more difficult to dissolve.

An increase in the solids feed rate from 400 to 700 g/min led to a decrease in the mean particle diameter of 73.83 μm (Table 4 and Fig. 2). When the amount of steam is maintained constant, steam being the material responsible for the formation of the liquid inter-particle bridges in the agglomeration, and the powder flow rate is increased, the formation of granules of smaller diameter is to be expected. The increase in solids feed rate also led to an increase in luminosity of the cocoa beverage powder, with an effect equal to 2.11.

On the other hand, an increase in dryer rotation from 12 to 52 rpm led to the production of larger granules, by 27.25 μm, probably due to the amount of shock between the solids, increasing the possibility of agglomeration of the moist particles in the first section of the dryer. There was also a slight decrease in luminosity of the product, the effect being equal to 0.52.

In the formulation with granulated sugar, the results showed the same tendencies obtained with the crystal sugar. The increase in solids feed rate from 400 to 700 g/min led to a decrease in the mean particle diameter of the granules, with an effect equal to 63.15 μm (Table 4 and Fig. 2). On the other hand, an increase in dryer rotation (12–52 rpm) led to a slight increase in product moisture content (positive effect of 0.18%), possibly due to the reduced residence time of the granules in the dryer. The variable of process vapor pressure was statistically significant ($p \leqslant 0.10$) for the moisture content (positive effect of 0.24%) and for the color parameter b (positive effect of 0.33) as shown in Table 4 and Fig. 2. The color parameter b indicates the presence of the color yellow, and thus this color was intensified in the cocoa beverage powder when increasing the vapor pressure from 1.0×10^2 to 1.8×10^2 kPa.

The granulometry of the sugar used in formulating the agglomerates showed a marked influence on wettability (Tables 2 and 3). It can be clearly seen that in the range studied, when granulated sugar was used, none of the operational variables (vapor pressure, rotary dryer temperature, solids feed rate and dryer rotation) evaluated in the agglomeration process, were statistically significant (Fig. 2). Nevertheless, the standard cocoa beverage powder formulated with granulated sugar and not submitted to agglomeration, presented a value for wettability of 11.79 s. This indicates that with the conditions of the lower levels (−1), these variables were already sufficient for the wettability to be instantaneous (Table 3). On the other hand, it would not be possible to study levels lower than these variables, since those presented in the present study were within the operational limits of the steam agglomerator used.

3.2. Influence of steam agglomeration on the physical properties and reconstitution of the powders

Steam agglomeration resulted in an increase in quality of the cocoa beverage powders. As compared to the control (product without agglomeration) the process led to an increase in mean particle size, this being more pronounced in the cocoa beverage powder formulated with granulated sugar. As shown in Tables 2 and 3, the mean particle diameter increased from 396.35 μm (control) to 546.02 μm (product formulated with granulated sugar), and from 438.12 μm (control) to 512.65 μm (product formulated with crystal sugar). In the case of granulated sugar, due to the greater surface area of soluble solids exposed to the ligating agent (steam) and greater dissolution rate of amorphous sugar, a sugar solution formed on the surface of the particles, increasing the stickiness and hence granulation. Kowalska and Lenard (2005), working with the agglomeration of cocoa products, showed that a sucrose solution coating led to a greater increase in diameter and improvements in the instantization properties. However coating with cocoa or maltodextrin, which are more hydrophobic materials, led to an increase in diameter without the concomitant decrease in wettability. Omobuwajo et al. (2000) also showed the influence of particle size of the sugar used in the thermal agglomeration of products formulated with cocoa and sugar. Products with a more attractive color and easier to dissolve were obtained using sugar with a smaller diameter (230–370 μm).

A decrease in moisture content was observed in the agglomerated products after the drying stage, as compared to the initial product (control) (Tables 2 and 3). The low moisture content of these powders is highly favorable, since it confers adequate fluidity on the product, as well as decreasing the possibility of caking during storage. The initial moisture content of the cocoa beverage powders was 3.65% w.b. (formulated with crystal sugar) and 3.53% w.b. (formulated with granulated sugar), and after steam agglomeration and drying, these values changed to 1.06% w.b. and 1.45% w.b., respectively. Shittu and Lawal (2007) determined the moisture content of 10 commercial brands of cocoa beverage powders and found values varying from 0.8% w.b. to 3.6% w.b., similar to those found in the present study.

With respect to the characteristics of reconstitution in liquids, there was a marked decrease in the wetting times, increasing the facility with which liquid penetrated into the porous matrix of the granule by capillarity, formed due to the agglomeration of

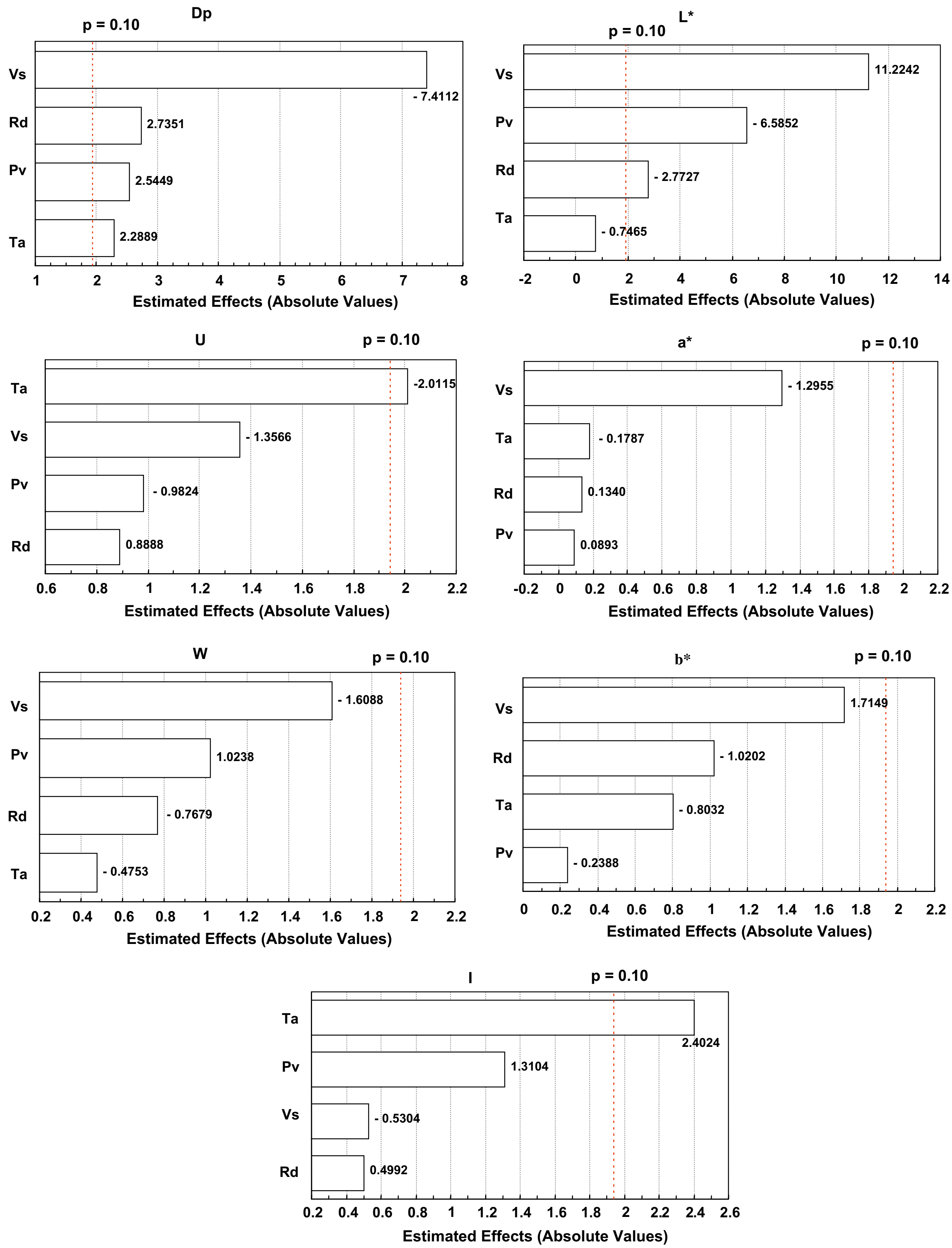

Fig. 2. Pareto diagrams showing the significance of the effects for the responses of mean particle diameter (D_p), moisture content (U), wettability (W), insolubility (I) and color ($L^*a^*b^*$), for the first fractionated experimental design (cocoa beverage powder formulated with crystal sugar).

the solids with steam. This effect was more evident with the cocoa beverage powders formulated with granulated sugar, the wetting times decreasing from 11.79 to 0 s (instantaneous product), as shown in Table 3. With respect to the product formulated with crystal sugar, the times decreased from 19.63 s (control) to 1.07 s (Table 2). Shittu and Lawal (2007) measured the wetting times for commercial cocoa beverage powders and found values in the range from 7.44 to 21.7 s. According to these authors the increase

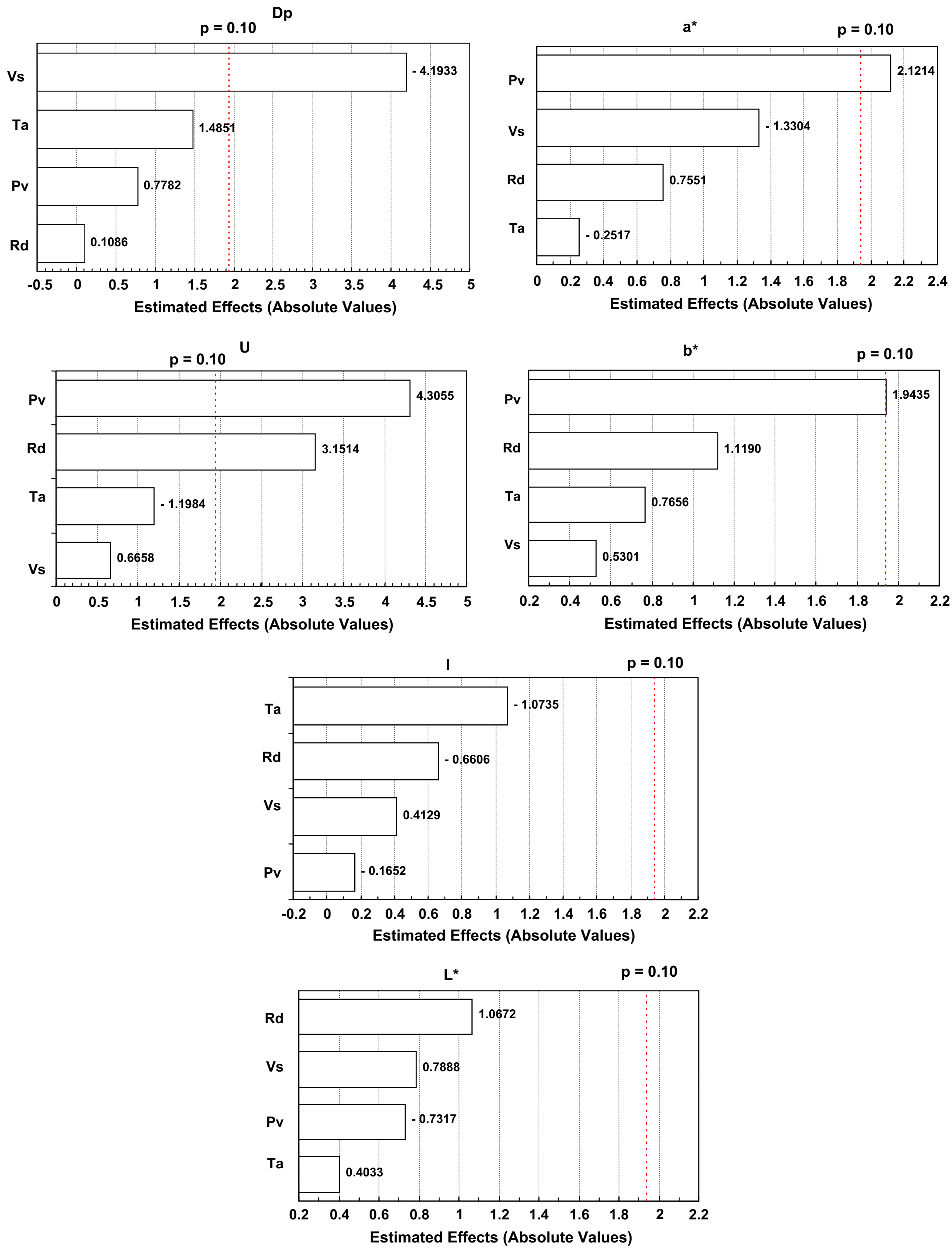

Fig. 3. Pareto diagrams showing the significance of the effects for the responses of mean particle diameter (D_p), moisture content (U), insolubility (I) and color (L^* a^* b^*), for the second fractionated experimental design (cocoa beverage powder formulated with granulated sugar).

in particle size of beverages made with cocoa powder showed negative relationships with respect to wettability. In the present study it was possible to observe that the increase in mean particle diameter due the agglomeration process caused a decrease in wetting times, agreeing with the results obtained by Shittu and Lawal (2007). Schubert (1980) showed that particle sizes below 200 μm should be avoided in mixtures of sugar and cocoa in order to obtain better wettability, whereas particle sizes above 400 μm are recom-

Table 5
Evaluation of the influence of drying temperature on the physical properties and reconstitution properties of the cocoa beverage powder formulated with granulated sugar.

Drying temperature (°C)	D_p (μm)	U (% w.b.)	I (%)	Color		
				L^*	a^*	b^*
40	471.89 ± 3.87[a*]	1.70 ± 0.08[a*]	7.86 ± 0.26[ab*]	41.59 ± 0.17[a*]	9.15 ± 0.14[a*]	10.62 ± 0.16[a*]
60	465.28 ± 3.76[a]	1.61 ± 0.06[b]	7.87 ± 0.20[ab]	41.67 ± 0.15[a]	9.12 ± 0.12[a]	10.44 ± 0.19[a]
80	457.63 ± 3.40[b]	1.55 ± 0.08[c]	7.64 ± 0.23[b]	42.33 ± 0.18[a]	9.16 ± 0.14[a]	10.46 ± 0.20[a]
100	468.51 ± 3.34[a]	1.41 ± 0.08[d]	8.09 ± 0.24[a]	42.59 ± 0.16[a]	9.17 ± 0.15[a]	10.39 ± 0.18[a]

Fixed process variables: $P_v = 1.0 \times 10^2$ kPa; V_s = 400 g/min; R_d = 12 rpm.
[*] Values in the same column with different letters are statistically different ($p \leqslant 0.10$) according to Tukey's test.

mended in order to obtain good instantizing properties. The cocoa beverage powders obtained in the present study showed particle sizes equal or greater than 400 μm, it is the case of the product formulated with granulated sugar and not agglomerated, and the wettabilities were relatively low, the highest value obtained for wettability being 19.63 s, for the non-agglomerated product formulated with crystal sugar. The presence of soybean lecithin, a surface active reagent, certainly influenced the decrease in wetting times (Vissotto et al., 2006), since this compound is constituted of one lipophilic portion and another hydrophilic portion, in addition to contributing to a slight pre-agglomeration of the powdered products.

The insolubility remained the same in the case of the hot chocolate formulated with crystal sugar (control = 7.69%; agglomerated product = from 7.00% to 7.90%), and showed a slight increase for that formulated with granulated sugar (control = 7.04%; agglomerated product = from 7.44% to 8.22%), as can be seen in Tables 2 and 3, respectively. The formation of solid bridges between the sugar and cocoa solids in the product formulated with granulated sugar made dissolution of the granules more difficult and therefore it took longer, reflecting in the increase in the insoluble compound content. According to Takeiti et al. (2008) the increase in mechanical resistance of steam agglomerated maltodextrin granules resulted in a decrease in their instantizing properties. Shittu and Lawal (2007) stated that solubility was a highly relevant property for cocoa beverage powders with respect to their consumption characteristics, although for these products complete dissolution does not apply since the cocoa contains a significant amount of insoluble compounds. Of the insoluble cocoa compounds, 18.6% correspond to protein and 21% to fats (Omobuwajo et al., 2000). Shittu and Lawal (2007) showed that sugar is the most important factor influencing the solubility of cocoa beverage powders, since this is the most soluble constituent of this type of product. In the present study the sugar content of the formulations was not altered, only its granulometry, and thus this study contributed in the sense of demonstrating that the sugar particle size had an impact on the solubility of the agglomerated cocoa beverage powders.

With respect to the color, the agglomerated cocoa beverage powders showed a decrease in luminosity (parameter L^*), an increase in the red hues (parameter a^*) and an intensification of the yellow color (b^*) as compared to the respective non-agglomerated products. It is possible that the Maillard reaction occurred during the agglomeration process, which is characterized by a combination of reducing sugars with amino-acids, both present in the cocoa beverage powders, under the effect of heat, resulting in darkening of the product. No reports were found in the literature about alterations in the color of cocoa beverage powders during steam agglomeration.

3.3. Product quality at milder and economic conditions of the process

Further trials were conducted considering the results obtained in the fractional designs. Granulated sugar was used as it showed better results. It was also verified that product wettabilities were high for all condition studied, so the most economic conditions for steam agglomeration were the lowest possible vapor pressure and dryer rotation ($P_v = 1.0 \times 10^2$ kPa; R_d = 12 rpm and also lower drying temperatures (40, 60 and 80 °C) than those studied in the earlier experimental plans. Solids feed rate was fixed at low level (V_s = 400 g/min) that corresponds to higher agglomeration rate and high particle diameter. Table 5 shows the results obtained: moisture content – U, insolubility – I, mean particle diameter – D_p and color (L^* a^* b^*).

The use of drying temperatures of 40, 60 and 100 °C resulted in granules with mean diameters which did not differ from each other at a significance level of 10% by Tukey's test (Table 5). However, at a temperature of 80 °C, the mean diameter obtained was 457.63 μm, smaller and significantly different (at a significance level of 10%) than those obtained at the other drying temperatures. Since cocoa beverage powders are mixtures and each constituent is present in the formulation in different proportions, one could raise the hypothesis of a phase transition of the product at temperatures close to 80 °C. It is known that the phase transition temperature of sucrose is at 62 °C and that of the maltodextrin 20 D.E. used in the formulation studied, at 141 °C (Bhandari and Howes, 1999). It should be mentioned that the proportions of sucrose and maltodextrin present in the cocoa beverage powders studied, were, respectively, 80% and 12%.

A decrease in moisture content of the granules with increase in drying temperature of the rotary dryer of the agglomerator was also shown, the moisture content being equal to 1.70% w.b. at a drying temperature of 40 °C, and 1.41% w.b. at 100 °C. It should be mentioned that a significant difference was observed by Tukey's test, at a level of 10%, between all the moisture contents obtained at the drying temperatures evaluated.

On the other hand, the insolubilities of the agglomerates dried at the temperatures of 40 and 60 °C were equal to each other (Table 5). A significant difference existed between the insolubility of the powder dried at 80 °C ($I = 7.64 \pm 0.23$%) and that dried at 100 °C ($I = 8.09 \pm 0.24$%). This probably occurred due to the formation of more resistant granules at drying temperatures of about 100 °C; the dissolution time of the solids in the liquid being insufficient for the total dissolution of the granules.

No difference was observed for the color parameters (L^* a^* b^*) of the cocoa beverage powders dried at the different temperatures evaluated (Table 5). There was a difference in color between the non-agglomerated and agglomerated products, but no statistically significant difference at the 10% level of significance between the agglomerated products dried at the different temperatures with respect to this parameter of evaluation.

4. Conclusions

It was concluded that for the cocoa formulated with crystal sugar, increases in the vapor pressure and in the rotary dryer temperature and rotation resulted in an increase in granule size. An increase in solids feed rate led to a significant reduction in the mean size of the agglomerates obtained, and an increase in dryer temperature resulted in an increase in product insolubility due to the formation of more resistant solid bridges that were more difficult to dissolve, formed between the insoluble compounds (cocoa solids) and the sugar crystals. For the cocoa beverage powder formulated with granulated sugar, the process variable with the greatest impact on the product was the solids feed rate, its most pronounced effect being in the decrease in size of the granules formed as a function of the increase in the amount of powder fed into the agglomerator.

The process of steam agglomeration led to an increment in general product quality, with an increase in particle diameter and decrease in the wetting times, these effects being observed principally for the cocoa beverage powder formulated with granulated sugar, due to the greater surface area exposed to the steam. It also resulted in changes in the color parameters, with a decrease in luminosity (L^*) and increases in the red (a^*) and yellow (b^*) hues for both products studied, formulated with crystal and granulated sugar.

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