AGGLOMERATION OF COCOA POWDER MIXTURES – INFLUENCE OF PROCESS CONDITIONS ON PHYSICAL PROPERTIES OF THE AGGLOMERATES

AGLOMERACIJA KAKAO MJEŠAVINA – UTJECAJ UVJETA PROCESA NA FIZIKALNA SVOJSTVA AGLOMERATA

Maja BENKOVIĆ, Ingrid BAUMAN

University of Zagreb, Faculty of Food Technology and Biotechnology, Pierottijeva 6, 10000 Zagreb

e-mail: mbenkovic@pbf.hr

Abstract

Nowadays agglomerated cocoa drink powder represents a very appealing beverage for children as well as for adult customers. It is produced by mixing basic ingredients (cocoa powder, sugar, vitamin and lecithin, which is followed by agglomeration. Agglomeration is necessary, because non-agglomerated cocoa powders exhibit very poor flow and reconstitution properties. This research showed the influence of process conditions and composition of the cocoa powder mixtures on the physical properties of the agglomerated final product. Eight mixtures were prepared, four containing cocoa powder with 10-12% fat and four containing cocoa powder with 16-18% fat. Three different kinds of sugars (sucrose, glucose and trehalose) and a sweetener (erythritol) were added to cocoa powder while preparing the mixture. Agglomeration was conducted using STREA 1™ fluid bed agglomerator at a constant air temperature (60°C), with a constant addition of water soluble lecithin (Metarin®) with varying duration of the process and varying amount of added water. Bulk density (Engelsmann jolting volumeter), particle size (Malvern Mastersizer 2000) and colour measurements (Minolta colorimeter; L = brightness, a = redness, b = yellowness) were conducted prior to and post agglomeration, while agglomerate hardness was estimated by conducting a compression test with a 30 mm probe on TA.HDPlus Texture Analyser. Percentage of added water had a significant effect on colour, bulk density and the duration of the agglomeration process. This effect was more significant with mixtures made with cocoa powder containing 10-12% fat. As for mixtures with 16-18% fat, a significant influence of water addition on agglomerate median diameter was found. Agglomeration time and the percentage of added water showed dependence towards the composition of the mixture. This research showed that, in order to agglomerate powder mixtures successfully, parameters such as mixture composition, particle size, percentage of added water and lecithin, drying temperature, process duration and ambient conditions should be well coordinated and controlled to get agglomerates with optimal quality.

Keywords: *agglomeration, cocoa powder, process conditions*

Sažetak

Aglomerirani kakao napitci danas predstavljaju vrlo popularno piće kod djece i kod odraslih konzumenata. Takvi se napitci proizvode miješanjem osnovnih sastojaka (kakao praha, šećera, vitamina i lecitina) te nakon toga slijedi aglomeracija. Aglomeracija kakao mješavina je potrebna jer neaglomerirane mješavine karakteriziraju izrazito loša rekonstitucijska svojstva i svojstva tečenja. U ovom je istraživanju prikazan utjecaj procesnih parametara i sastava kakao mješavina na fizikalna svojstva aglomeriranog gotovog proizvoda. Priređeno je osam mješavina, četiri koje su sadržavale kakao prah sa 10 – 12% masti i četiri koje su sadržavale kakao prah sa 16-18% masti. Tri različite vrste šećera (saharoza, glukoza i trehaloza) te sladilo (eritritol) su dodani kakao prahu tijekom pripreme mješavina. Aglomeracija je provedena pri konstantnoj temperaturi zraka (60°C), sa konstantnim dodatkom lecitina topljivog u vodi (Metarin®) te sa promjenjivim vremenom trajanja procesa i količinom dodane vode.

Nasipna gustoća, veličina čestica i boja (L = sjaj, a = crvena boja, b = žuta boja) izmjereni su prije i poslije aglomeracije, dok je čvrstoća aglomerata određena kompresijskim testom pomoću 30 mm sonde na TA.HDPlus analizatoru teksture. Postotak dodane vode pokazao je značajan utjecaj na boju, nasipnu gustoću i vrijeme trajanja procesa aglomeracije. Utjecaj postotka dodane vode bio je značajnije izražen kod mješavina izrađenih od kakao praha sa 10 – 12% masti. Kod mješavina sa 16 – 18% masti postotak dodane vode značajno je utjecao na srednji promjer čestica aglomerata. Vrijeme aglomeracije i postotak dodane vode također su povezani sa sastavom mješavine. Ovo je istraživanje pokazalo da, ukoliko se proces aglomeracije uspješno želi provesti, parametri kao što su sastav mješavine, veličina čestica, postotak dodane vode i lecitina, temperatura sušenja, trajanje procesa i uvjeti okoliša moraju biti dobro usklađeni i kontrolirani da bi se dobili aglomerati optimalnih svojstava.

Ključne riječi: *aglomeracija, kakao prah, procesni parametri*

INTRODUCTION

Cocoa is a very popular food flavor in the world nowadays. It is available in a wide variety of colors and flavors and used in numerous applications. A good quality cocoa powder is relatively free flowing, stable and uniform in color and flavor, of good microbiological quality, and easy to handle by the user *(de Muijnck, 2005)*. Besides the above mentioned, a range of other characteristics define the powder, and have and important impact on the end product in which the cocoa is used. These are pH, fineness, fat content, alkalinity, wettability, moisture absorption, solubility and density *(de Muijnck, 2005).* It is a well known fact that sole cocoa powders show poor reconstitution properties, such as dispersibility, wettability and solubility. This can be improved by adding other components to the powder (e.g. sugars, vitamin mixtures, milk powder) and instantizing the powder with the addition of lecithin. The third way to improve dispersibility is to agglomerate cocoa, sugar and other particles. During this process, sugar crystals are moistened with water or steam, (lecithinated) cocoa and other solid particles adhere to the wet sugar crystals, and the agglomerated particles are dried *(Anonymous, 1999).* Various techniques can be used to agglomerate cocoa beverage powders: steam agglomeration *(Visotto et al, 2010)*, fluid bed agglomeration *(Kowalska and Lenart, 2005)* and thermal agglomeration *(Omobuwajo et al., 2000).* High shear mixers can also be used for wet agglomeration of cocoa powder beverages *(Vu et al., 2003).* Agglomeration improves reconstitution properties and it is therefore important that the degree of agglomeration and the compactness of the powder can be controlled during the production process *(Benković and Bauman, 2009).*

Method used in this research was fluid bed agglomeration, because of the possibility of fine dispersion of Metarin solution and the ability to spray dry the agglomerates immediately after the agglomeration process. Fluid bed agglomeration can be classified as one-pot system, because the elementary steps of the process occur in the same chamber. Fluidization and bulk mixing are provided by the upward air flow, while fine droplets of liquid (solvent with or without a binder) are distributed by a nozzle positioned above the fluidized bed *(Turchiuli et al., 2005).* In this research, Metarin® solution was used as a binder, since cocoa particles exhibit poor solubility due to fats mostly positioned on the particle surface. Agglomeration occurs when wet particles collide to each other and a liquid bridge is formed between them. When subsequent drying occurs, the solvent evaporates and a solid bridge arises due to the solidification of the binder. If the wetting, colliding and drying steps are repeated, fluidized particles grow through agglomeration with each other *(Turchiuli et al., 2005).*

The objective of this work was to determine the influence of process conditions and mixture composition on the agglomerate properties of cocoa powder mixtures with two different fat contents. Each mixture also contained sugars and sweeteners with different particle sizes (from crystal to powder).

MATERIALS AND METHODS

Mixture formulation

The basic recipe for the mixtures was 30% (w/w) of cocoa powder and 70% (w/w) sugar. Sugars were added according to relative sweetness. Four mixtures were made with cocoa powder containing 10-12% fat and four were made with cocoa powder containing 16-18% fat. Composition of the mixtures is shown in Table 1. All the components were mixed together in a Turbula mixer (Willy A. Bachofen Maschienenfabrik, Muttenz, Switzerland) for 15 minutes to obtain a homogenous blend.

***Table 1**. Mixture composition (mass percentages of the components)*

Sample	Cocoa (%)	Sugar (%)
1	30	Sucrose (70%)
2	30	Sucrose (60%) + Trehalose (10%)
3	30	Sucrose (35%) + Eritrythol (35%)
4	30	Sucrose (35%) + Glucose (35%)

Particle size

Particle size distribution was measured using laser diffraction technique. All the samples were analyzed on a Malvern 2000 particle size instrument equipped with Scirocco 2000 dry dispersion unit. Feed rate and pressure were adjusted according to laser obscuration at about 1 bar and 50% feed. Particle size distributions were determined for the mixtures (before agglomeration) and for agglomerates (after agglomeration).

Bulk density

Loose and tapped bulk density were measured using a jolting volumeter (STAV 2003, J. Engelsmann AG, Ludwigshafen). The cocoa powder mixture was weighed, poured into a glass container, tapped 10 times to obtain loose bulk density value and then tapped 90 more times to obtain tapped bulk density value *(Haugaard Sørensen et al., 1978).* The volume of the powder in the column was recorded and loose and tapped bulk densities were calculated. Hausner ratio was then calculated by dividing loose bulk density value with tapped bulk density value. All the measurements were done in triplicate. Bulk densities and Hausner ratios were determined for the mixtures as well as for the agglomerates.

Color

Colour of the samples was measured in a Minolta colorimeter, using 6 cm diameter and 3.8 cm height cells and a 0.5 in. diaphragm. Reflection spectra were registered and colour parameters for 10 ° vision angle and D65 illuminant (L^*: brightness, a^*: redness, b^*: yellowness) were calculated. Measurements were done in triplicate for the mixtures, as well as for the agglomerates.

Agglomeration

Agglomeration of the mixtures was carried out using STREA 1™ fluid bed agglomerator/spray drier/ coater (Gea Niro, Denmark) (Figure 1a). Mixtures were poured into a chamber where they were fluidized by upward hot air flow. Metarin®, water soluble lecithin was mixed with water and sprayed over the mixture in the fluidization chamber (Figure 1b) until the final concentration of Metarin® in the end product was 0.8% (w/w). If the amount of water added with Metarin® turned out to be insufficient to agglomerate the mixture, more water was added, without Metarin®. Duration of the agglomeration process was recorded for every mixture. After agglomeration, agglomerates were spray dried (Figure 1c) at a temperature of 60°C, left to cool down and tested for physical properties.

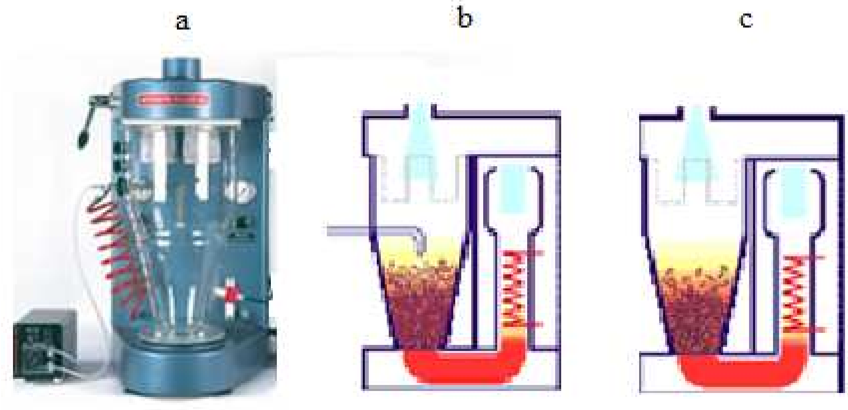

***Figure 1**. STREA 1^{TM} fluidized bed agglomerator/spray dryer/coater*
(http://www.niroinc.com/pharma_systems/laboratory_fluid_bed_STREA1.asp)

Agglomerate hardness

Agglomerate hardness was estimated by a compression test, using TA.HDPlus Texture Analyser equipped with a 30 mm cylinder probe. Strips of adhesive tape were cut to a length of approximately 150 mm and the sample was poured gently over the adhesive side of the tape. After pouring the tape was gently taped to ensure an even covering of the surface and the excess granules were tipped off. After probe calibration, the tape was placed under the probe. The probe compressed the sample to 0.2 mm above the machine base at which point the maximum force was recorded as the hardness of the sample. The compression test was repeated three times on different regions of the tape.

Statistical analysis, with the significance level p = 0.05, was performed using Statistica 8 software (Statsoft, USA).

RESULTS AND DISCUSSION

Nowadays, agglomeration is one of the most important processes in the cocoa powder drink production. Cocoa powder particles have a layer of fat positioned on the surface, which makes it hard to dissolve such particles. Therefore, sugars and other components are added to cocoa drink powder mixtures to improve the reconstitution properties. Also, agglomeration is a well know method for improving instant properties of cocoa powders, as well as other food powders which exhibit poor dispersibility, wettability or solubility. The aim of this research was to determine which properties and in what way are they influenced by different process parameters during the agglomeration process.

Comparison of median diameter, loose and tapped bulk density of the mixtures and the agglomerates are shown in Table 2.

Table 2. *Median diameter, loose and tapped bulk density of the mixtures and the agglomerates (results are expressed as mean (n=3) ± standard deviation)*

Sample	Median diameter [µm]		Loose bulk density [kg m^{-3}]		Tapped bulk density[kg m^{-3}]	
	Mixtures	Agglomerates	Mixtures	Agglomerates	Mixtures	Agglomerates
Cocoa powder 10-12% fat						
1	527.76 ± 5.33	550.44±6.91	740.80±8.72	800.71±22.72	881.90±12.34	830.09±16.67
2	422.22 ± 9.11	465.97±8.29	774.42±14.03	738.56±5.54	900.00±15.12	774.16±11.02
3	511.73 ± 6.34	462.63±3.52	738.85±17.11	751.75±10.38	853.78±9.36	791.32±8.63
4	220.45 ± 2.46	381.64±6.95	710.40±12.72	536.34±6.12	807.27±22.44	550.57±8.22
Cocoa powder 16-18% fat						
1	489.78 ± 12.40	501.73±28.19	875.45±12.22	731.03±9.44	963.00±15.16	785.18±7.62
2	414.29 ± 8.27	507.26±5.79	815.61±14.65	798.86±17.63	928.10±14.39	843.24±9.35
3	521.15 ± 6.32	541.99±13.00	832.10±10.34	733.02±10.24	915.31±19.68	766.04±10.11
4	202.78 ± 3.24	379.60±25.26	718.40±5.29	558.75±7.56	844.12±14.32	583.92±5.53

It can be seen in Table 2 that bulk density of the agglomerates was lower than the bulk density of the mixtures. An exception can be seen for samples 1 and 3 made with cocoa powder containing 10-12% fat, although this seems like a rather illogical observation, since agglomerates have a much more porous structure than mixtures. A rational explanation could be that some sugar crystals have broken down into smaller particles due to high attrition forces and particle collisions in the fluidization chamber. Sample 3 (10-12% fat) mixture exhibited bigger median diameter than the agglomerate, probably also due to sugar crystal breakage. It is also important to emphasize that the difference between loose and tapped bulk density was much bigger for the mixtures than for the agglomerates, and these differences were more expressed with cocoa powder mixtures with 16-18% fat.

Loose and tapped bulk density values were used to calculate the Hausner ratio. Comparison of the Hausner ratio for mixtures and agglomerates with 10-12% fat and 16-18% fat are shown in Fig. 2.

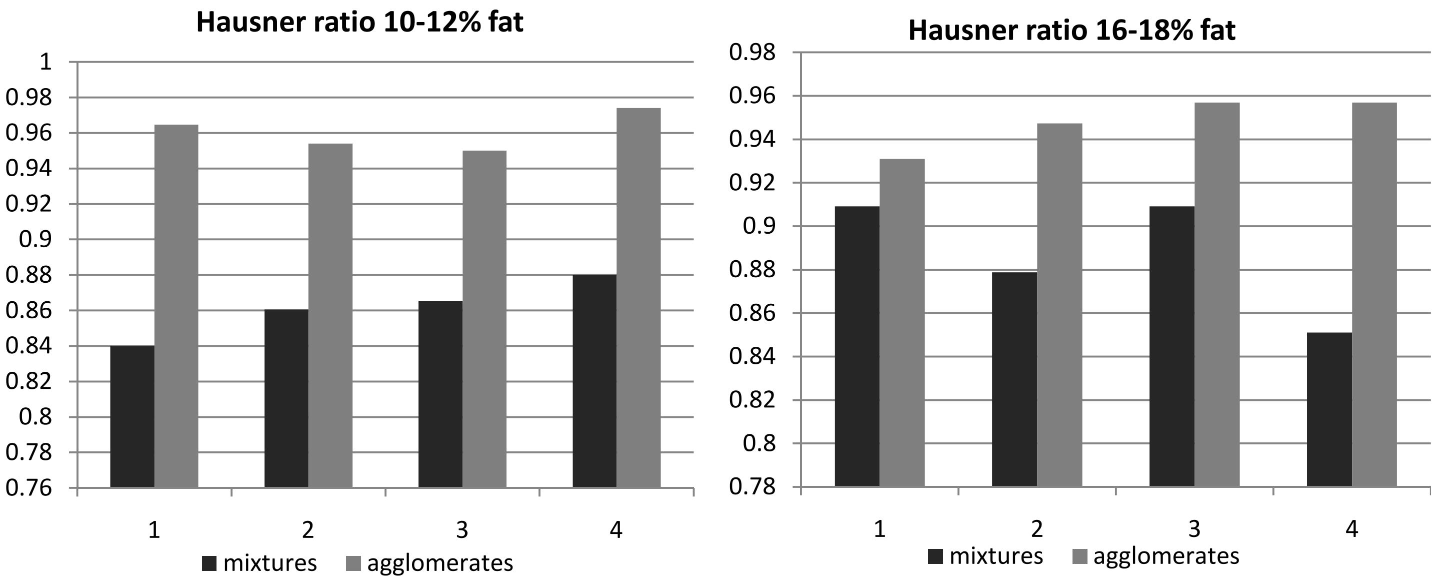

Figure 2. *Comparison of Hausner ratio for mixtures and agglomerates*

a) Mixtures and agglomerates made with cocoa powder containing 10-12% fat
b) Mixtures and agglomerates made with cocoa powder containing 16-18% fat

Agglomerates showed higher Hausner ratio values than the mixtures. The differences between mixture and agglomerate Hausner ratios were higher for the mixtures with 10-12% fat. According to this ratio, agglomerates and mixtures show low compressibility and good flowability. Even though Hausner ratio is an indicator of powder flowability, more sophisticated methods are used nowadays to characterize the powder flowability, for example powder rheometer which can give information on behavior of the powder during flow and its susceptibility to caking and cohesion *(Ghosal et al., 2010).*

Cocoa is available in a wide variety of colors, ranging from light yellowish brown to deep red brown to very dark blackish brown *(Anonymous, 1999; de Muijnck, 2005).* In general, the color of cocoa powder is influenced by the optical effect in which the fat on the solid particles affects the light absorption. The higher the fat content, the darker the color will appear to be *(de Muijnck, 2005).* Addition of sugars to cocoa powder caused changes in color of the mixture in the first stage of cocoa drink powder production. After the agglomeration phase, changes in color were also detected and the results are shown in Table 3.

Table 3.*Color changes of the mixtures during the agglomeration process*

Cocoa powder 10-12% fat						
Sample	Mixtures			Agglomerates		
	L	a	b	L	a	b
1	50.33±0.11	13.08±0.02	24.19±0.05	48.61±0.14	10.49±0.02	15.87±0.01
2	50.75±0.07	12.72±0.01	23.66±0.03	48.11±0.10	10.09±0.02	15.33±0.02
3	50.23±0.07	13.19±0.02	24.63±0.03	47.91±0.11	9.82±0.03	14.75±0.02
4	53.11±0.05	12.38±0.02	23.24±0.04	45.50±0.08	9.39±0.02	12.82±0.01
Cocoa powder 16-18% fat						
1	45.58±0.09	14.28±0.03	24.24±0.04	49.27±0.12	10.25±0.01	16.59±0.06
2	46.88±0.09	13.70±0.03	23.25±0.04	48.19±0.09	11.26±0.01	16.86±0.02
3	45.49±0.10	14.74±0.01	25.12±0.02	46.83±0.06	10.55±0.02	15.48±0.03
4	50.10±0.17	13.01±0.03	22.42±0.02	48.27±0.05	9.73±0.01	14.34±0.03

As can be seen in Table 3, cocoa powder mixtures with 10-12% fat showed markably higher brightness (L-values) than the mixtures made with cocoa powder containing 16-18% fat. After the agglomeration process L, a and b values dropped significantly for mixtures made with 10-12% fat cocoa powder and sample 4 made with 16-18% cocoa powder. Samples 1, 2 and 3 showed a rise in brightness after agglomeration. The biggest drop was detected in b values (yellowness) for all the samples. Changes in L, a and b values indicated a color change during the exposure of the cocoa powder to heat. These results are in accordance with *de Muijnck (2005),* stating that when cocoa powder is subjected to temperature fluctuations, discoloration will occur owing to the change in crystalline size or form of the cocoa butter.

Agglomeration and instantization were conducted with the addition of Metarin® at a constant concentration of 0.8% (w/w) in the end product. Drying temperature was 60% °C and the water was added by spraying only if the amount of added Metarin® solution showed to be insufficient to agglomerate the mixture. Process conditions during the agglomeration process and the agglomerate hardness is shown in Table 4.

Table 4. *Agglomeration process conditions and agglomerate hardness*

Cocoa powder 10-12% fat				
Sample	Time [min]	Metarin® [% w/w]	Water [% w/w]	Hardness [N]
1	10	0.8	0	65.18 ± 18.51
2	5	0.8	0	144.197 ± 8.60
3	20	0.8	0	91.80 ± 12.95
4	50	0.8	27.5	216.89 ± 43.24
Cocoa powder 16-18% fat				
1	10	0.8	0	199.65 ± 19.61
2	20	0.8	0	144.64 ± 20.66
3	35	0.8	0	113.95 ± 2.85
4	60	0.8	27.5	181.27 ± 8.91

Statistical analysis showed that the amount of added water had a significant ($p < 0.05$) influence on the duration of the agglomeration process. If more water was added, the agglomeration process lasted longer. During the visual inspection of the agglomeration chamber, segregation was visible for samples 1 – 3 (both % of fat), since sucrose, trehalose and erithrythol added to the mixtures were crystalline and there was a big difference in bulk density and particle size of sugars and cocoa powder. However, presence of crystalline sugar appeared to have speeded up the agglomeration process, because the sugar crystals posed as very good agglomeration core particles. Samples 1 - 3 (both % of fat) had a surplus of crystalline particles and the agglomeration was visible even before the addition of the binder (Metarin®) solution. Sample 4 (both % of fat) had a surplus of small particles, the duration of the agglomeration process was longer and it required extra water addition. Statistical analysis also revealed a significant influence of water addition on bulk density of the agglomerates, with the bulk density being lower as the amount of added water increased. Water addition and the duration of the process, especially the exposure of the powder to the higher drying temperature (60 °C) for a longer time period, also had a significant influence on the color of the agglomerates. Agglomerate hardness was determined by a compression test, but the statistical analysis showed no significant influences of the process conditions on agglomerate hardness. More samples with different percentages of added water and process duration should be prepared in our future research to determine the influence of these parameters on agglomerate hardness.

CONCLUSIONS

Since the agglomeration process represents an important part of cocoa drink powder production, the intention of this work was to show how process conditions and mixture composition affect the agglomeration process and quality of the agglomerates. A significant influence of the amount of water added during the agglomeration was found on duration of the agglomeration process. If more water is added, the agglomeration process lasts for a longer period of time. The amount of water added also had a significant influence on bulk density and the color of the agglomerates. Fluid bed agglomeration caused significant changes in color of the mixtures and the agglomerates – L (brightness), a (redness) and b (yellowness) values drop during the agglomeration for most of the samples. Presence of sugar in crystal form speeded up the agglomeration process, because these crystals acted as good agglomeration cores. However, big difference in bulk density and particle size between sugar crystals and cocoa powder caused segregation during the agglomeration process.

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