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AGGLOMERATION OF A FINE AND HYDROPHOBIC POWDER BY SPRAY DRYING

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INTRODUCTION

Agglomeration has wide applications in the food industry since the formation of larger and permanent aggregates of finely divided solids allows better flow characteristics and an improved control of solubility [1, 2]. In the case of cocoa, the main reason for agglomeration is to enhance its dispersion rate for instant beverages. 'Instant' means the agglomerated cocoa powder can be dispersed in water or in milk by stirring the product in the liquid for a short time [3, 4, 5].
The low dispersibility of cocoa powder in liquid is due to its physical properties (small particle size diameter, high cohesivity) and its composition (10-12% cocoa butter content). A previous study [6] has shown that cocoa dispersion kinetics can be improved by increasing the slowest step of the dispersion, the powder wetting by the liquid. Among several possibilities for achieving this increasing the grain size and porosity by agglomeration appears to have many advantages.
Several techniques have been used to produce instant cocoa powders which require additives such as binders and surfactants [7, 8] or more recently, a continuous thermal agglomerator [9]. And alternative is spray drying, a technique widely used in the food and pharmaceutical fields to dry heat-sensitive materials. In this work, spray drying has been investigated as a potential technique to obtain agglomerated cocoa powder from finely dispersed cocoa powder.

MATERIALS AND METHODS

CHARACTERIZATION OF AGGLOMERATED AND NON-AGGLOMERATED COCOA POWDERS

The cocoa powder used here was provided by Cargill Ltda., Brazil. It is a low fat cocoa powder (10-12% of cocoa butter) and it is insoluble in water.The non-agglomerated raw powder and the resultant spray dried granules are both characterized in the same way. The mean diameter was measured using a Malvern laser diffraction instrument with edible oil as a dispersant phase to separate the granules without breaking them. The true density was measured with a Micromeritics Accupyc helium pycnometer. The aerated (ρ_a) and packed (ρ_p) densities were measured with a Hosokawa powder tester. These two densities were used to calculate the following indexes : the Carr index (I_C) that is representative of the powder flowability and the Hausner index (H_R) that corresponds to a measure of the cohesivity between the grains or the granules :

$$I_C = \frac{\rho_p - \rho_a}{\rho_p}$$

$$H_R = \frac{\rho_p}{\rho_a}$$

The intra-particular porosity of the grains and the intra-granular porosity of agglomerated granules were measured with a Micromeritics mercury porosimeter using a maximum pressure of 413 MPa. The inter-particular or inter-granular porosities were calculated from these porosities and the measured values of true and aerated densities. The morphology of the grains and the granules were observed with a scanning electronic microscope (S.E.M.).

AGGLOMERATION BY SPRAY DRYING

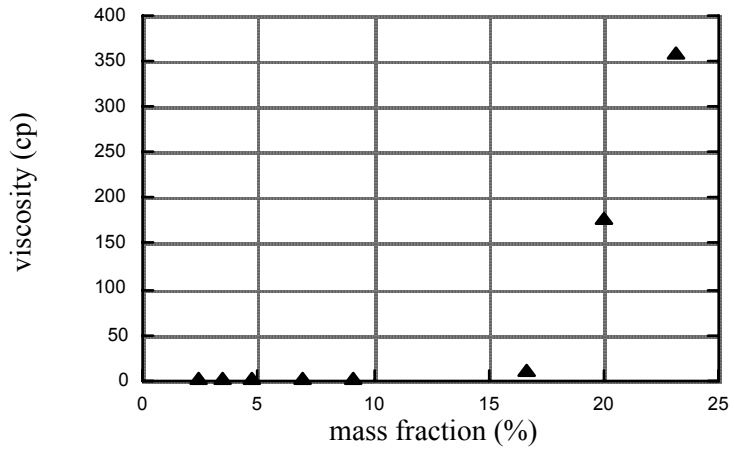


Figure 1. Dynamic viscosity vs. mass volume of cocoa dispersions in demineralized water at 20°C

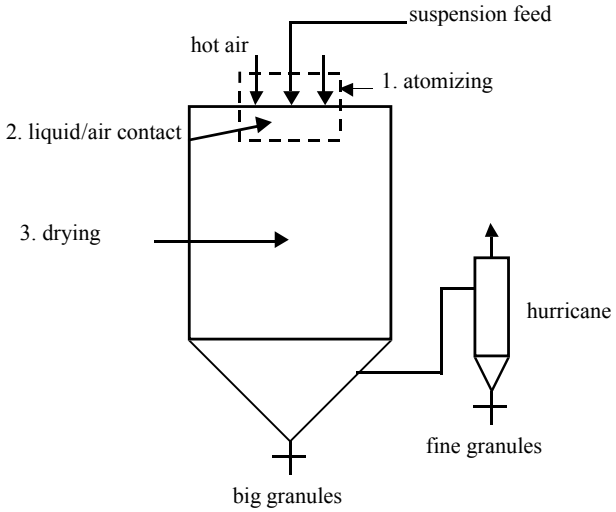


Figure 2. Experimental spray dryer

Suspensions of cocoa powder in demineralized water were prepared with different solid concentrations and their dynamic viscosities were measured with a Brookfield DVII viscometer in a cell controlled at 20°C. The aim was to identify the suitable solid

concentration in aqueous suspension for the agglomeration tests since, the initial solid concentration in the feed and the resultant suspension viscosity can strongly influence the physical characteristics of the final dried particles from a spray drying processes [10]. From the data obtained (Figure 1), a mass fraction of 20% was chosen for the spray drying agglomeration tests because the viscosity of a 20% mass fraction suspension is still low enough not to hinder droplet formation.
A rotary atomizer APV Nordic Anhydro PSD52 with a drying capacity of 7 kg water per hour was used in the experiments (Figure 2). The speed of rotation of the atomizer was 30 000 rpm. The outlet temperature was 90°C, and the inlet temperature and the suspension flow were chosen to keep the outlet temperature constant. The large granules of the agglomerated powder were collected at the bottom of the drying chamber and the fine granules were carried by the air flow and collected in a cyclone. The two size population of granules were obtained and called "large spray dried granules" and "fine spray dried granules" and they were characterized in the same way as for the non-agglomerated cocoa powder.

RESULTS AND DISCUSSION

DIAMETERS AND SIZE DISTRIBUTIONS

The characteristic diameters and the span of the size distribution for the powder and the granules are given in Table 1. It can be seen that the spray drying is a good method for agglomerating cocoa powder as the mean diameter changes from 15.7 μm to 40.8 μm for the large granules and to 24.8 μm for the fine ones. We can also note that there is a wide size distribution for the non-agglomerated cocoa powder (span ~ 3), but narower for the agglomerated granules. Agglomeration by spray drying reduces the amount of fine particles..

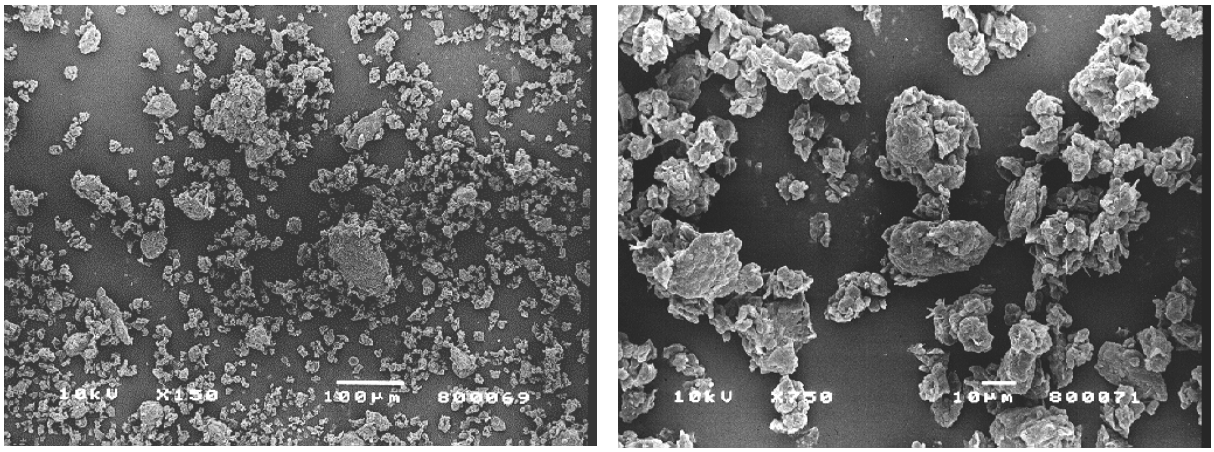
Material	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	Span $(d_{90}-d_{10})/d_{50}$
Non agglomerated cocoa powder	5.9	15.7	51.4	2.9
Large spray dried granules	23.2	40.8	65.0	1.0
Fine spray dried granules	7.3	24.8	46.2	1.6

Table 1. Mean diameters and span measured by a laser diffraction in edible oil

PARTICLE AND GRANULE MORPHOLOGY

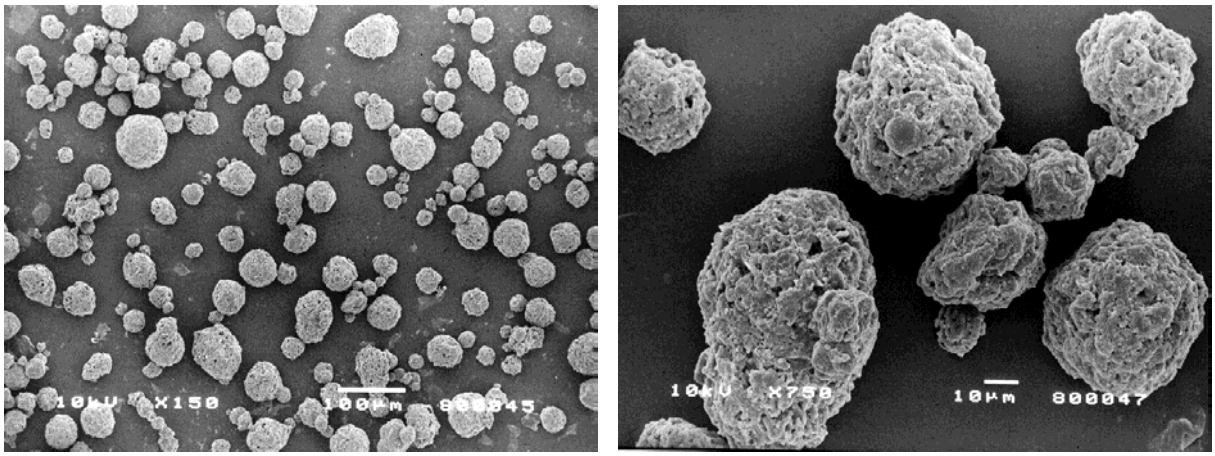
Figures 3 and 4 show the differences between non-agglomerated cocoa grain powder and the spray dried granules. It can be seen that the non-agglomerated cocoa grains are irregular, and the spray dried granules are predominantly spherical and porous (Table 3). Their rounded shape can be attributed to drying,by atomisation. The suspension is broken into small liquid droplets by the rotary atomiser, and these spherical droplets are instantly dried by the co-current flow of hot air . The solids contained in these droplets remains aggregated, the original particles being still identifiable (Figure 3c and 3d), and the liquid volume initially

between the powder grains is substituted by air volume, that induces a porosity in the formed agglomerates.



(a) (b)

Figure 3. S.E.M. for raw cocoa powder, magnitude 150X and 750X



(a) (b)

Figure 4. S.E.M. for spray dried granule powder, magnitude 150X and 750X

FLOWABILITY AND COHESIVITY

The non-agglomerated cocoa powder is very cohesive probably because of its irregular slab shaped particles. These irregularities produce an entanglement of the grains, and then fragile agglomerates. The agglomerates produced by spray drying are spherical and solid interactions between granules are less important than for the grains leading to a lower cohesivity for granules than for grains and consequently improved flowability (Table 2). Due to its physical characteristics, the agglomerated powder is easier to handle.

Material	ρ_{true} ($g.cm^{-3}$)	$\rho_{aerated}$ ($g.cm^{-3}$)	ρ_{packed} ($g.cm^{-3}$)	I_C (%)	Flowability	H_R (-)	Cohesivity
Non-agglomerated powder	1.45	0.28	0.54	47.9	Very bad	2.1	Very strong
Large spray dried	1.45	0.31	0.54	41.5	Medium	1.4	Strong

granules							
Fine spray dried granules	1.45	0.30	0.54	44.8	Bad	1.8	Very strong

Table 2. Densities and mechanical properties of cocoa powders

POROSITIES

The non-agglomerated cocoa powder is characterized by an inter-particular porosity, that can be calculated from its true and aerated densities. However, the spray dried cocoa granules present an intra-granular porosity, equivalent to the inter-particular porosity of non-agglomerated powder. The results are shown in Table 3.

Material	Intra-particular porosity (%)	Intra-granular porosity (%)	Inter-granular porosity (%)	Sum of porosities (%)
Non-agglomerated powder	0	no	83.0	83.0
Large spray dried granules	0	17.5	60.8	78.3
Fine spray dried granules	0	17.2	62.2	79.4

Table 3. Porosites for cocoa grains and granules

CONCLUSIONS

Spray drying appears to be an attractive method for the preparation of cocoa powder agglomerates which seems to come close the desired properties for rapid dispersing beverages. Briefly, the technique brings about modifications to the raw cocoa powder, by increasing the mean diameter of the original grains, inducing an intra-granular porosity and decreasing the cohesivity between the resulting cocoa granules. The spray drying process thus modifies some physical properties of a cocoa powder that are the important characteristics in the dispersion kinetics of a powder in a liquid. Quantification of the dispersion kinetics is presently under evaluation.

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