

## SKIM MILK AGGLOMERATES GROWTH DURING FLUIDIZED BED WET AGGLOMERATION AND DRYING

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**Abstract:** Fluidized bed agglomeration of skim milk powder by spraying water was used to produce dry agglomerates with improved instant properties. The growth of agglomerates was studied following the evolution of the particle size distribution during agglomeration under different conditions of processing. During the first minutes, initial particles (180µm) associated to form two size populations (250 and 400 µm). The smallest one disappeared quickly while the largest one increased in size until 650-700 µm at the end of experiment (30-40 min). The tested conditions corresponding to a lower final particle moisture content (6.8-7.0 % d.b.) generated the smaller agglomerates (650-640 µm).

**Keywords:** Agglomeration, Fluidized bed, Milk powder, Growth mechanisms

### INTRODUCTION

Many ingredients in the food industry are supplied in powdered form. It allows a longer shelf-life of the ingredient, due to reduced water content, and easier and more economic transport, due to reduced mass. Besides maintaining the ingredient functionality until its utilization, food powders must also be easy to handle. Especially, when used in wet formulation, the powder must have good mixing and instant properties. Fluidized bed agglomeration of individual particles (100-200µm) is often used to produce large and porous dry agglomerates (800µm-1.5mm) with improved instant properties.

Initial particles, fluidized in rising hot air, are sprayed at their surface with a liquid (binder solution or water) to render them locally sticky. Collisions between fluidized wet "sticky" particles allow

adhesion with the formation of either liquid or sinter bridges. The simultaneous drying by the hot fluidization air leads to the consolidation of the new structure formed. Agglomerates grow progressively due to the repetition of these different steps (wetting, collision, adhesion, drying, mixing). Fluidized bed agglomeration is therefore a complex process in which many parameters interact influencing the structure and the properties of the agglomerates obtained.

Milk particles are complex mixtures of carbohydrates, proteins, minerals and fats. During fluidized bed agglomeration with water spraying in hot air, amorphous carbohydrates are likely to undergo glass transition due to the plasticizing effect of water wetting locally the particles. If the surface viscosity becomes locally lower than 10<sup>8</sup> Pa.s, the critical viscosity corresponding to sticky particles, sinter bridges may be formed when particles collide

(Palzer, 2011). At the same time, fats, known to be over-represented in the first layers of the particle surface, even in skim milk (Kim et al., 2009), may melt and also contribute to adhesion between colliding particles.

The aim of this study was to investigate the influence of some process parameters on the growth of milk powder agglomerates in order to understand mechanisms involved during wet agglomeration in fluidized bed and their impact on the physical and handling properties of agglomerates.

## MATERIALS AND METHODS

Agglomeration trials were performed in a pilot scale batch fluidized bed granulator (UniGlatt, Glatt, Ge). Skim milk powder (carbohydrate 53% w/w dry matter, protein 37%, minerals 9%, fat <1%), provided by Lactalis (France), had a median diameter of 180 $\mu$ m (sieved between 100 and 250 $\mu$ m). The conditions tested are summarized in Table 1. In all cases, the ratio between the total mass of water sprayed and the mass of milk particle in the bed was 55/100 leading to different spraying times. The hot air flow (75 kg/h) was constant and the powder bed height was not controlled.

Table 1. Operating conditions for the agglomeration trials (total sprayed water 55g/100g particle,  $T_b$  48°C, air flow rate 75kg.h<sup>-1</sup>).

n°	Mass load (g)	Water flow rate (g.min <sup>-1</sup> )	Spraying duration (min)	Sprayed water (g)
1	300	5.5	30	165
2	300	4.4	38	165
3	400	5.5	40	219

In each experiment, the milk powder was first pre-heated (12-20 min) in the fluidizing hot air to reach a stable bed temperature of 55 $\pm$ 1°C. Then distilled water (20°C) was top sprayed (bi-fluid nozzle) producing a decrease of the bed temperature. To keep a constant fluidized bed temperature  $T_b$  of 48 $\pm$ 1°C in any case, the inlet air temperature was adjusted along the trials.

An additional experiment was carried out with skim milk powder fluidized in hot air (48 °C) without water pulverization to check some effect of heating in the agglomeration process (ie. melting of some surface components).

During the trials, samples (approx. 4g) were regularly taken in the bottom of the fluidized bed at preset times with a perforated tube. The final agglomerated powder was immediately recovered into hermetic jars (without cooling stage).

Particle size distribution was obtained by manual sieving (2g, 2min) and powder water content was measured by oven drying (105°C, 1g, 24h). Final powder wettability was estimated by measuring the time required for 5g of powder to completely sink in 100mL of water (20°C). Bulk and tapped densities were obtained measuring the volume occupied by a given mass of powder in a 25mL test tube before and after 1250 falls (density tester, Varian, Fr). Flowability was estimated measuring the time required for 10g of powder to flow out of a calibrated funnel (AFNOR NFB 35032).

## RESULTS AND DISCUSSION

When fluidizing skim milk particles in hot air without spraying water, no evolution of the particle size distribution was observed. The agglomeration process therefore required spraying water on the particle surface to render it sticky. And, in the studied conditions, the growth of skim milk agglomerates may be attributed mainly to carbohydrates.

### *Properties of final powder*

Fluidized bed agglomeration allowed increasing the median diameter of the milk powder from 180 $\mu$ m to about 700 $\mu$ m (Table 2). Compared to the initial powder requiring several hours to sink into water, all the agglomerated powders had a very good wettability, and were nearly instant. This was probably due to their larger size and more porous structure (Figure 1) also leading to a significant decrease of their bulk and tapped density (factor 2). Flowability of the initial milk powder was very good and was not modified after agglomeration despite the irregular shape of agglomerates. Powder water content was increased from 4.9 to about 7% but the water activity was still low, ensuring powder stability.

Table 2. Properties of the initial powder and of the final powder for the three trials.

Powder	Initial	1	2	3
$d_{50}$ ( $\mu$ m)	180	700	650	640
Wettability (s)	> 1h	< 4s		
$\rho_{bulk}$ (kg.m <sup>-3</sup> )	500	220		
$\rho_{tapped}$ (kg.m <sup>-3</sup> )	590	250		
Flowability	Very good			
X (%)	4.9	7.3	6.8	7.0
$a_w$	0.30	0.35	0.32	0.36

### *Agglomerates growth*

The evolution of the median diameter  $d_{50}$  and of the water content increase of the samples is shown in Figure 2. The median diameter and the particle water content increased rapidly, and then a slow down was

observed after about 10 minutes. Comparing with trial 1, the decrease of the water flow rate (trial 2) or the increase of the milk mass load (trial 3) generated smaller agglomerates that also corresponded to a lower increase of the moisture content of the powder.

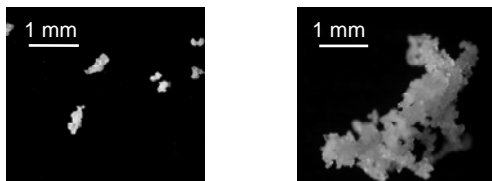


Figure 1. Initial particles and final agglomerate (optical microscope).

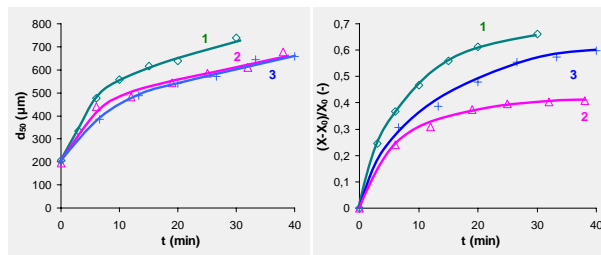


Figure 2. Evolution of  $d_{50}$  and of the water content increase during agglomeration.

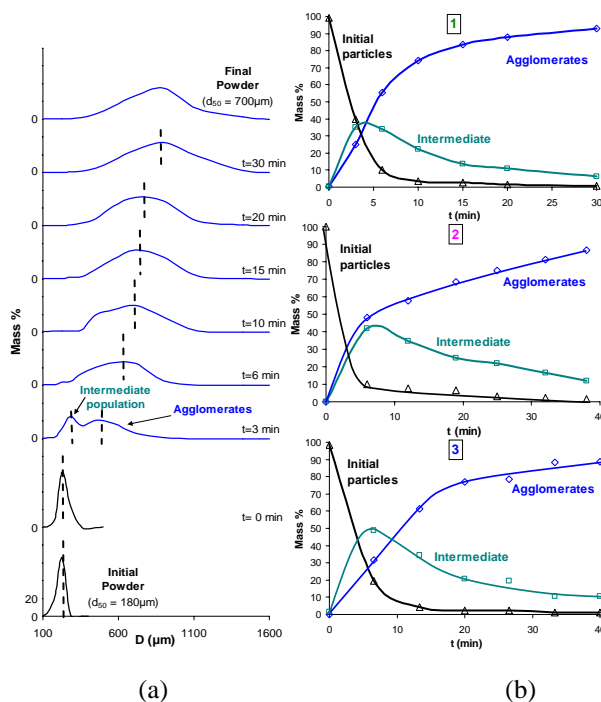


Figure 3. Typical evolution of particle size distribution (trial 1) (a) and evolution of the 3 populations in the 3 trials (b).

The evolution of the particle size distribution showed that an intermediate population (250  $\mu\text{m}$ ) appeared at the beginning of the process (3 min, Fig.3) and disappeared progressively. At the same time, the main population of agglomerates grew from an initial

size of about 400  $\mu\text{m}$  to a final  $d_{50}$  of 640-700  $\mu\text{m}$  (Table 2). This is consistent with the hypothesis that agglomeration occurs in stages, with first the association of initial particles into small initial structures from which larger agglomerates are progressively built. The size and compactness of the final structures and the kinetics of agglomerates growth may depend on the operating conditions.

## CONCLUSION

Fluidized bed agglomeration of skim milk particles allowed improving the powder wettability. The final particle size and the moisture content depended on the agglomeration conditions. From the evolution of the particle size distribution, it can be stated that agglomeration could occur in two stages: initial particles are first associated into intermediate structures which, in a second stage, form progressively larger agglomerates. Consequences of this mechanism on the agglomerates structure and properties must now be investigated.

## NOMENCLATURE

$a_w$	water activity	
$d_{50}$	median mass diameter	$\mu\text{m}$
$D$	mean diameter of size class	$\mu\text{m}$
$t$	time	min
$T_b$	air temperature in fluidized bed	$^{\circ}\text{C}$
$X$	water content in 100g dry matter	%
$X_0$	water content at $t=0$ of spraying	%
$\rho$	density	$\text{kg}\cdot\text{m}^{-3}$

## ACKNOWLEDGEMENTS

This study is part of the PhD work of A. Barkouti in the scope of “Reactive Powder” project funded by ANR (French National Agency for Research). It was performed in collaboration with Juan A. Carcel from the Universitat Politècnica de València (Spain) during a stay in Agroparistech, Massy, thanks to the economic support of Ministerio de Educación of Spain (ref. PR201-0333).

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