

Steam-jet agglomeration of skim-milk powders : influence of the process parameters

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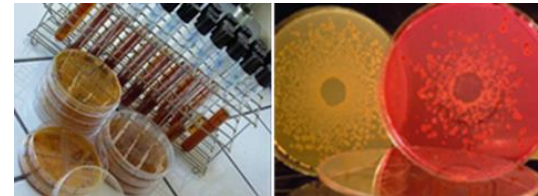
A multidisciplinary and multiscale approach, reinforced by two high-calibre facilities:



Dairy Platform



Biological Resource Centre



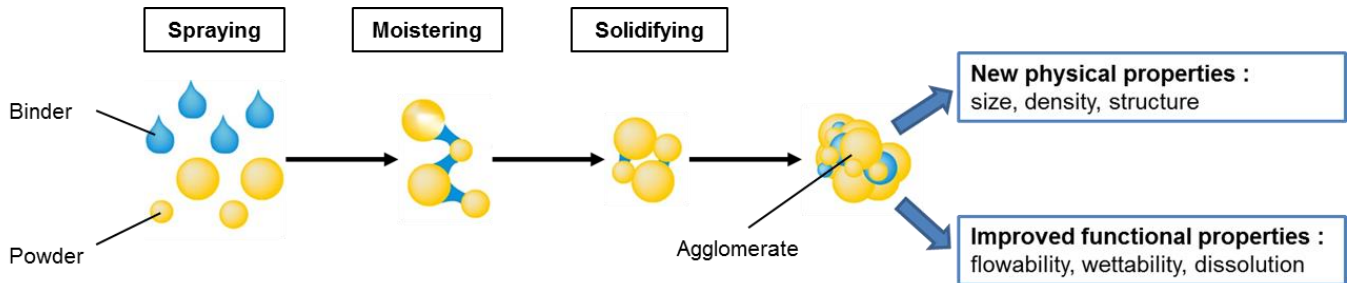
- ❑ **Structuration / destructuration mechanisms of food matrix:**
from structural characterisation to digestion
- ❑ **Dairy processing and cheese making:**
toward sustainable dairy systems
- ❑ **Microbial interaction:**
food matrix and host cell



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Agglomeration processes

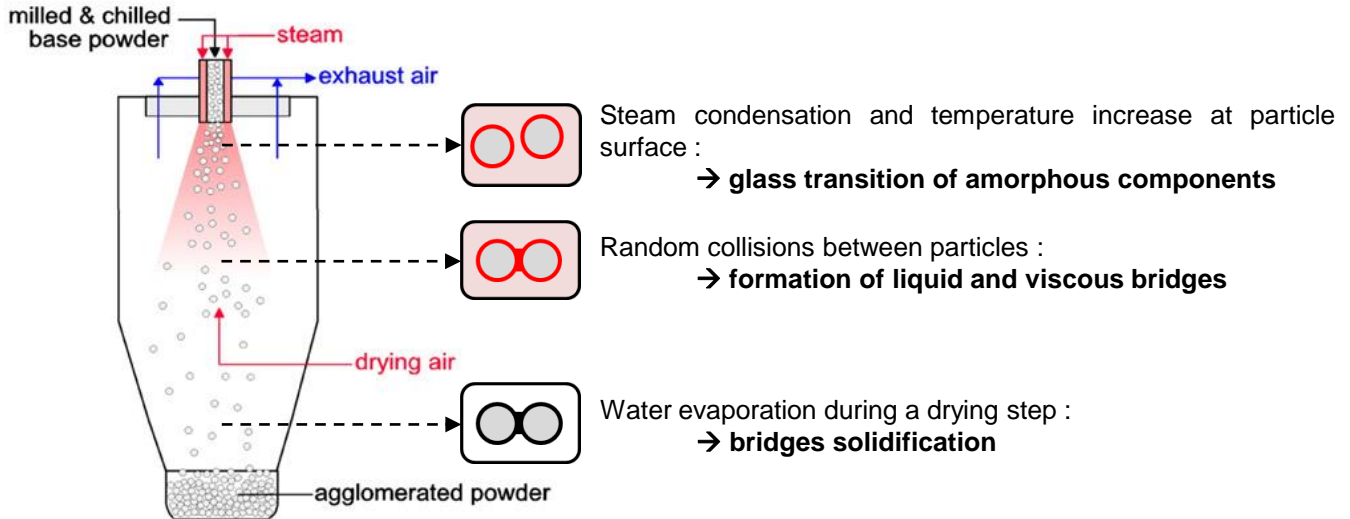
- ❑ Processes consisting in combining **fine primary particles** to form **larger ones** with modified properties.
- ❑ Different technologies depending on the **powder types** and the **target properties** :
 - Dry agglomeration processes : use of **pressure**
 - Wet agglomeration processes : use of a **binder**



Wet agglomeration processes principles (adapted from Glatt)

Steam-jet agglomeration

- ❑ 3 main technologies are used for wet agglomeration : fluidized bed, high shear mixer and **steam-jet**.



Steam-jet agglomeration process (Palzer, 2011).

➔ Production of agglomerates with **high porosity** and **high dissolution rate**

Skim-milk powders agglomeration

- ❑ Skim-milk powders are mainly composed of **lactose** ($\approx 50\%$ dry matter, amorphous state) and **proteins** ($\approx 35\%$ dry matter).
- ❑ Steam-jet agglomeration is used in order to obtain **instant powders** with improved rehydration properties.

↗ *size, mass, porosity*

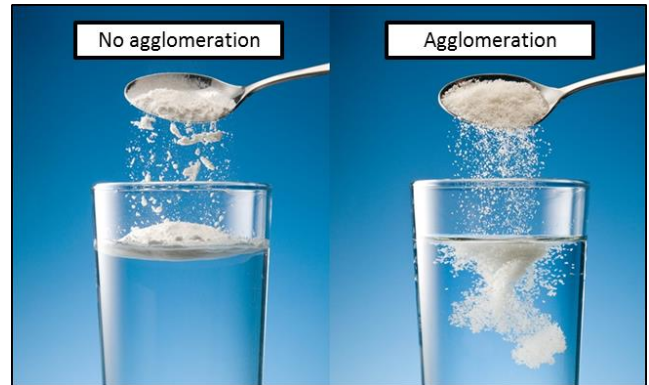
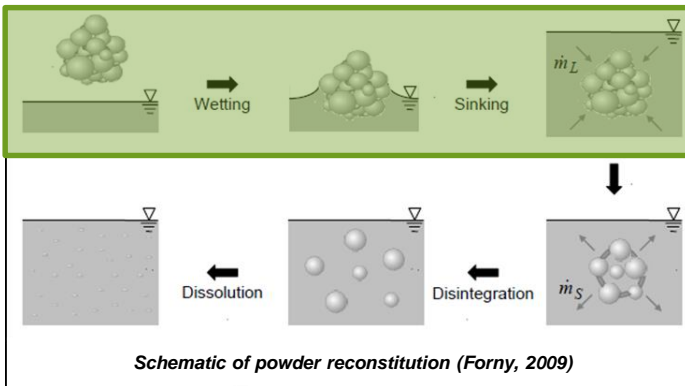


Image from sternmaid.de

Objectives

- ❑ Quality control of industrial products remains **experimental** and **empirical** :
 - Steam-jet agglomeration → **black box** process (closed, fast, random collisions)
 - Instant properties → **multi-factorial** causes (structure, composition, physicochemical state)

- ❑ **Lack of knowledge** → scientific publications 1997 – 2017 :
 - Granulation & Agglomeration = 622 papers
 - Granulation & Agglomeration + steam = 9 papers
 - Granulation & Agglomeration + steam + milk = **1 paper**

Martins P.C. (2008). Influence of a lipid phase on steam jet agglomeration of maltodextrin powders. Powder Technology, 185, 258 – 266.

→ How can we study and identify the key process parameters that should be controlled ?

Steam-jet agglomeration pilot plant

Powder feed rate (\dot{m}_p)

Native particles

Feeder

$$\frac{\dot{m}_s}{\dot{m}_p} = \text{Steam/Powder ratio}$$

Steam flow rate (\dot{m}_s)

Steam

Agglomeration zone

Wet agglomerates

Drying time

Oven

Drawer

Dried agglomerates

Drawer

Dried agglomerates

Oversize
(4 mm < d)

Agglomerates
(0.4 mm < d < 4 mm)

Fines
(d < 0.4 mm)

Agglomeration

1

Drying

2

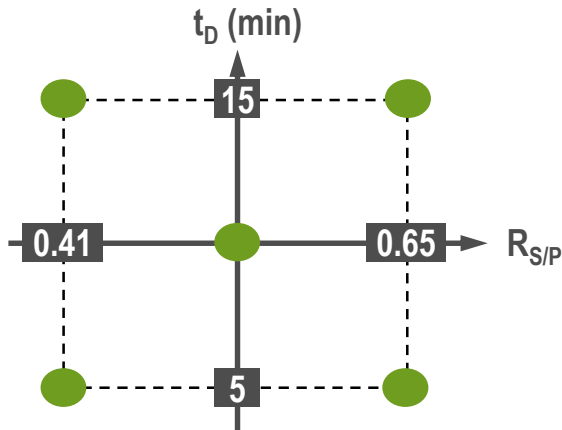
Sieving

3



Experimental strategy

- ❑ 2 process parameters were studied : the **steam/powder ratio ($R_{S/P}$)** and **the drying time (t_D)**.
- ❑ 1 **factorial design** of experiment was performed in triplicate :



Steam flow rate (kg.h ⁻¹)	Powder feed rate (kg.h ⁻¹)	$R_{S/P}$
1.3	3.2	0.41
1.7	3.2	0.53
2.1	3.2	0.65

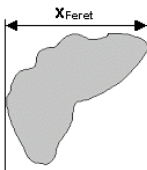
- ❑ Statistical analysis after standardization of the data → **linear model** with interaction :

$$\text{Response} = a R_{S/P} + b t_D + c (R_{S/P} \times t_D) + \text{constant}$$

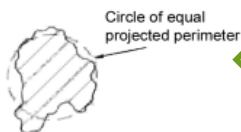
Characterization of the agglomerates

Water content
Water evaporation at 102°C during 5h

Feret diameter and circularity
Image analysis

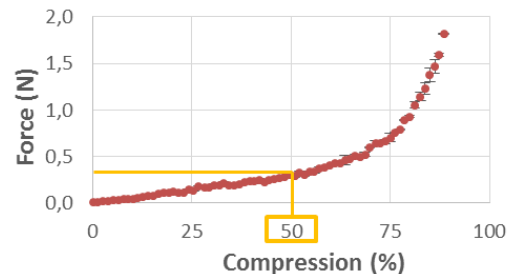


Circularity Factor

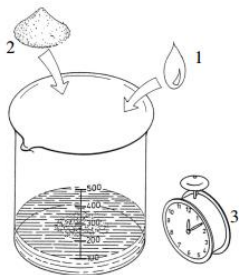


Responses

Mechanical strength
Uniaxial compression test



Wetting time
Time for 5g of sample to completely sink
into 100ml of water at 20°C
(< 20sec for instant powders)



Wetting time measurement (Westergaard, 1994).

Structure of the pilot plant agglomerates



Raw material :
Skim milk powder

Pilot plant agglomerate :

- **Porous** structure
- **Irregular** shape

Industrial agglomerate :

- Dense structure
- Spherical shape
- Regular surface

Influence of the process parameters

$$\text{Response} = a R_{S/P} + b t_D + c (R_{S/P} \times t_D) + \text{constant}$$

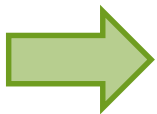
a, b, c coefficients values of the linear model :

Responses	$R_{S/P}$	t_D	$R_{S/P} \times t_D$	R^2
Circularity	-0.718 *	0.173	-0.078	0.528
Feret diameter (mm)	0.797 **	0.230	0.101	0.740
Mechanical strength (N)	0.420 *	-0.794 **	0.056	0.741
Water content (g.100 g ⁻¹)	0.355 *	-0.913 ***	-0.100	0.907
Wetting time (s)	0.674 ***	0.480 **	0.339 **	0.901

Significant influence of the steam/powder ratio

Significant differences are indicated ($p < 0.05$ (), $p < 0.01$ (**), $p < 0.001$ (***)).*

➔ More **liquid bridges** comes with more **steam available**, leading to **larger** and **less spherical** agglomerates.



During the agglomeration step, the ratio control the **extent of agglomeration** in the studied range of values.

Influence of the process parameters

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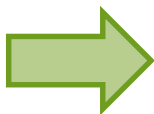
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Preponderant effect of the drying time

Significant differences are indicated ($p < 0.05$ (), $p < 0.01$ (**), $p < 0.001$ (***)).*

➔ Increasing the **drying time** leads to the formation of **dry** and **brittle** agglomerates.



The drying step is crucial for the **storage evolution** to prevent **microbial growth, caking or breakage**.

Influence of the process parameters

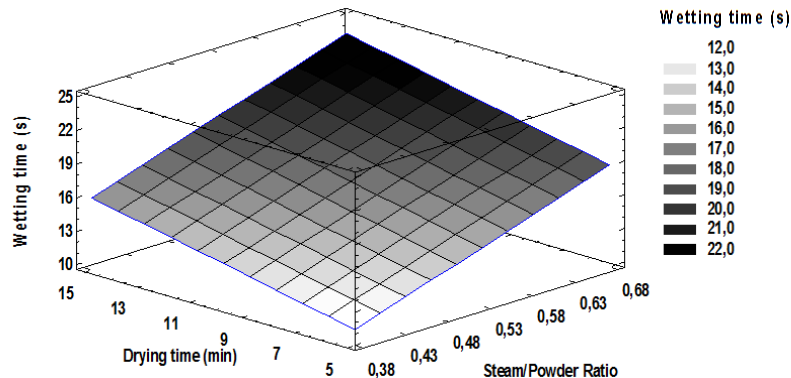
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Strongest influence of the ratio, maximal at long drying time because of the interaction.

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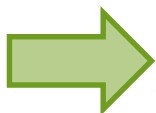
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➔ Instant properties are influenced by both **agglomeration** and **drying** steps ➔ difficult to identify a **key process parameter** to control.



Is the influence of the process parameters due to :

- **Structural modifications** (size, density, porosity)?
- **Physicochemical state** of the dairy components (lactose crystallization, protein denaturation)?

Conclusions

- ❑ A steam-jet agglomeration **pilot plant** was developed.
- ❑ Possible to study the influence of the **process** parameters on the agglomerates **properties** :

Agglomeration

→ the steam control the **extent of agglomeration** :

- size
- shape

Drying

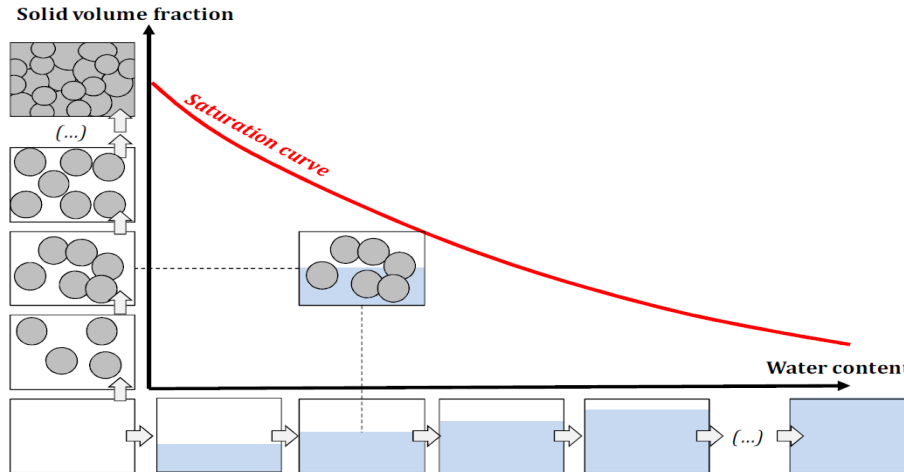
→ the time is important for **storage evolution** :

- water content
- mechanical strength

- ❑ **Interaction** between the two process parameters → further studies needed to understand the instant properties **mechanisms**.

Perspectives

- ❑ This pilot plant will allow to study :
 - the agglomeration **mechanisms** → **hydrotextural diagram** (solid volume fraction vs water content).



Hydro-textural diagram to describe the agglomeration mechanisms (Barkouti 2012)

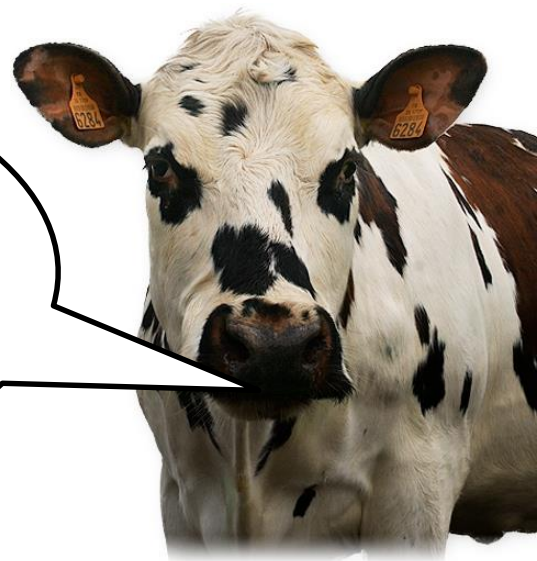
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 - the agglomeration **mechanisms** → **hydrotextural diagram** (solid volume fraction vs water content).
 - the **interactions** between the **raw material** properties, the **process** parameters and the **product** properties.
 - the **correlations** between the agglomerates properties to understand the **mechanisms of the instant properties** (multivariate data analysis).

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*THANK
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YOUR ATTENTION*



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