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

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EuroDrying’2017
19-20-21 June 2017 - Liège
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*

Please visit [http://www6.rennes.inra.fr/stlo_eng](http://www6.rennes.inra.fr/stlo_eng)
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**

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**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 condensation and temperature increase at particle surface:
- Glass transition of amorphous components

Random collisions between particles:
- Formation of liquid and viscous bridges

Water evaporation during a drying step:
- Bridges solidification

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** (≈ 50% dry matter, amorphous state) and **proteins** (≈ 35% dry matter).

- Steam-jet agglomeration is used in order to obtain **instant powders** with improved rehydration properties.

*Schematic of powder reconstitution (Forny, 2009)*
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


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

Steam flow rate ($\dot{m}_S$) = Steam/Powder ratio

Steam

Drying time

Dried agglomerates

Agglomerates (0.4 mm < d ≤ 4 mm)

Oversize (d > 4 mm)

Fines (d < 0.4 mm)

Agglomeration

1

Drying

2

Sieving

3
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:

Statistical analysis after standardization of the data \(\rightarrow\) **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

**Mechanical strength**
Uniaxial compression test

**Responses**

**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

<table>
<thead>
<tr>
<th>Raw material</th>
<th>Pilot plant agglomerate</th>
<th>Industrial agglomerate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Skim milk powder</td>
<td><strong>Porous</strong> structure</td>
<td>Dense structure</td>
</tr>
<tr>
<td></td>
<td><strong>Irregular</strong> shape</td>
<td>Spherical shape</td>
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<tr>
<td></td>
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<td>Regular surface</td>
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Influence of the process parameters

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:

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Significant influence of the steam/powder ratio

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.
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

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

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

Significant differences are indicated (\( p < 0.05 \) (*), \( p < 0.01 \) (**), \( p < 0.001 \) (***)).
Influence of the process parameters

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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)?
A steam-jet agglomeration pilot plant was developed.

Possible to study the influence of the process parameters on the agglomerates properties:

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

### Conclusions

#### Agglomeration

- the steam control the extent of agglomeration:
  - size
  - shape

#### Drying

- the time is important for storage evolution:
  - water content
  - mechanical strength
This pilot plant will allow to study:

- the agglomeration mechanisms \(\rightarrow\) hydrotextural diagram (solid volume fraction vs water content).

*Hydro-textural diagram to describe the agglomeration mechanisms (Barkouti 2012)*
This pilot plant will allow to study:

- 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).
THANK YOU FOR YOUR ATTENTION