

# THE IMPACT OF THE SIZE DISTRIBUTIONS OF THE NATIVE WHEAT POWDERS ON THEIR STRUCTURATION BEHAVIOR DURING WET AGGLOMERATION

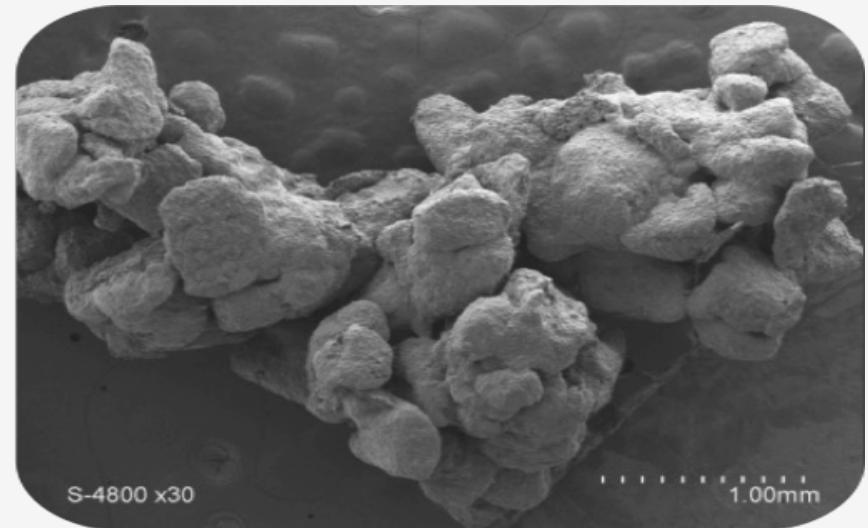
**Bettina Bellocq**

Under the supervision of  
**Pr. Bernard CUQ, Dr. Thierry RUIZ and Dr. Agnès DURI**

UMR IATE – INRA Montpellier – France

**8<sup>th</sup> International Granulation Workshop**

Sheffield (U.K.) - 28<sup>th</sup> June 2017



# PLAN

- 1 INTRODUCTION - WET AGGLOMERATION**
- 2 NATIVE WHEAT POWDERS AND METHODS**
- 3 RESULTS - IMPACT OF DIAMETER AND SPAN**
- 4 DISCUSSION - AGGLOMERATION MECHANISMS**
- 5 CONCLUSION**

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## 1.1. WET AGGLOMERATION OF FOOD POWDERS

**Agglomeration** is largely used to improve the powders **properties and behaviour**:

- *Reduction in dust production.*
- *Enhancement in flowability.*
- *Increase in bulk density.*
- *Reduction in segregation.*

During the agglomeration process...  
...the **native small particles** are gathered to form **larger assemblies** with specific **porous structures**, called the **agglomerates**.



*Agglomerated food powders*

- *Cocoa beverage powders*
- *Instant soluble coffee*
- *Culinary powders*
- *Flavors powders*
- *Protein powders*
- *Infant formulas*
- *Couscous grains*
- *Dairy powders*
- *Milk powders*
- *Bakery mixes*
- *Starch*



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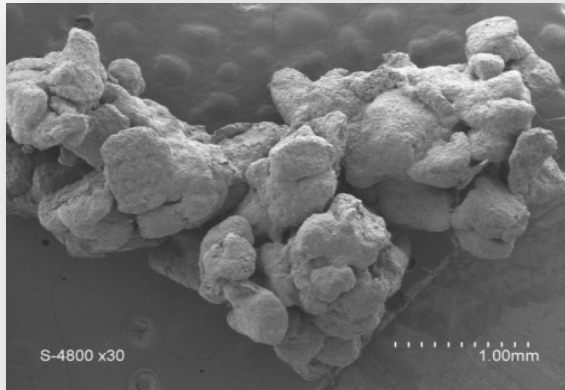
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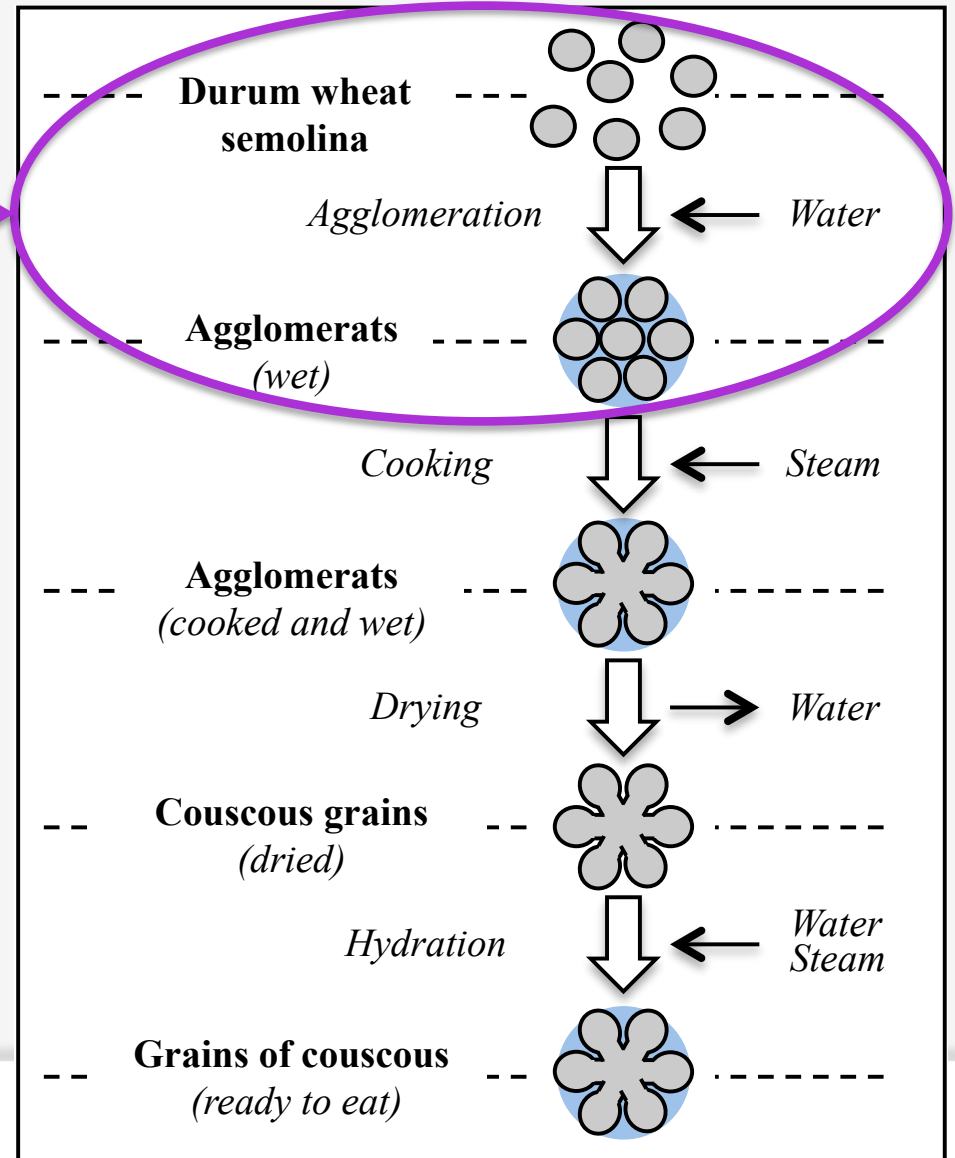
# 1.1. WET AGGLOMERATION OF FOOD POWDERS

Couscous grains are made by the succession of **3 unit operations**. The polydispersity of the size causes the high ratio of out of scope (up to 60%) of wet agglomerates.

Agglomeration is a **key unit operation** in the couscous production.



WET AGGLOMERATES OF COUSCOUS

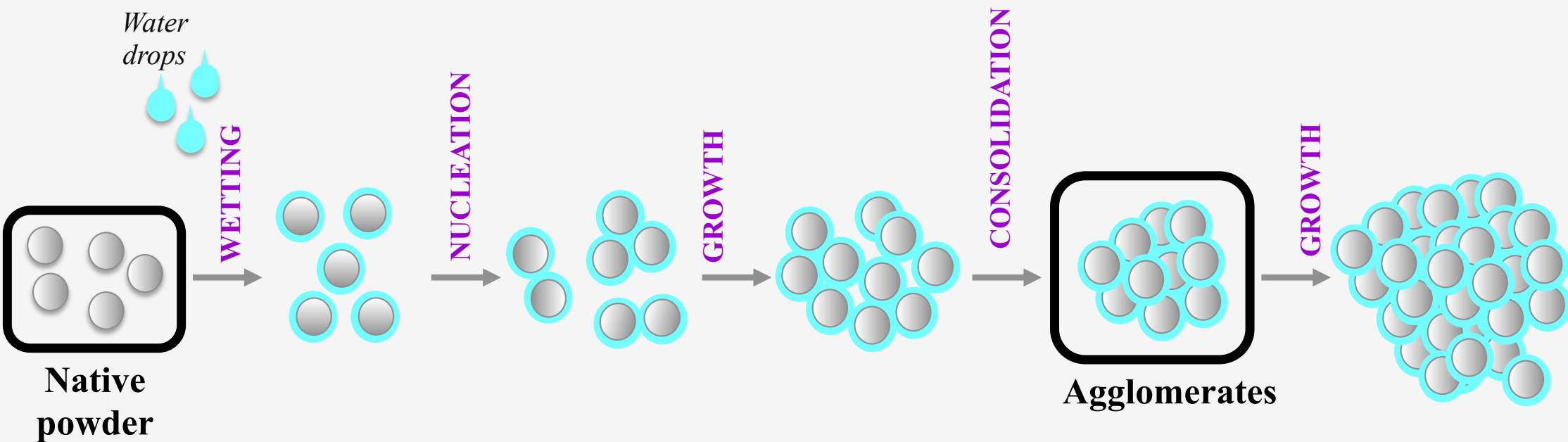


## 1.2. WET AGGLOMERATION - MECHANISMS

Wet agglomeration process is classically described as a combination of successive mechanisms at different rates :

- **Wetting and nucleation.**
- **Growth and consolidation.**
- *Breakage, erosion, rupture*

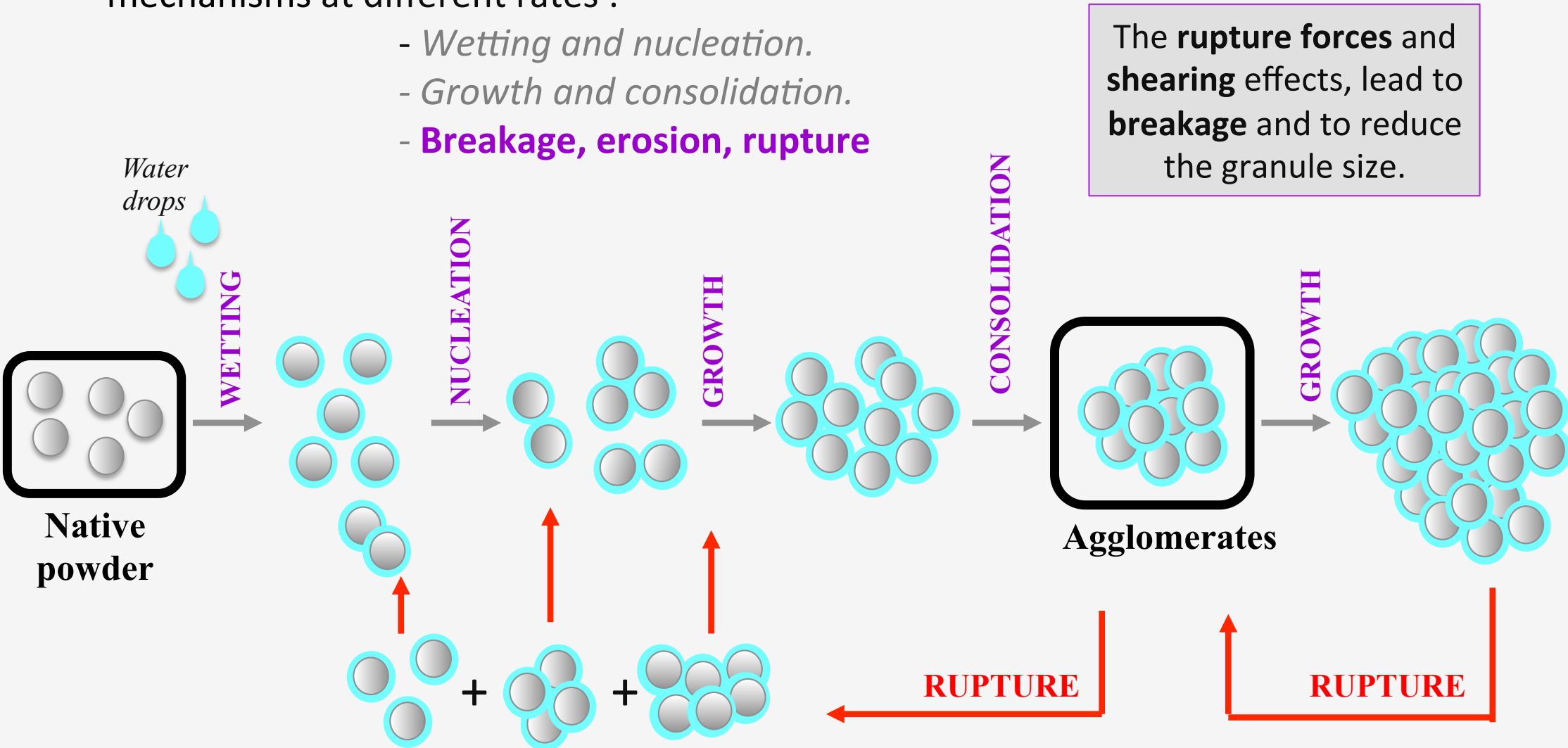
The **cohesion forces** generate **interactions** between particles, and promote granules **growth**.



## 1.2. WET AGGLOMERATION - MECHANISMS

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The wet agglomeration process is a **balance** between **growth** and **breakage**



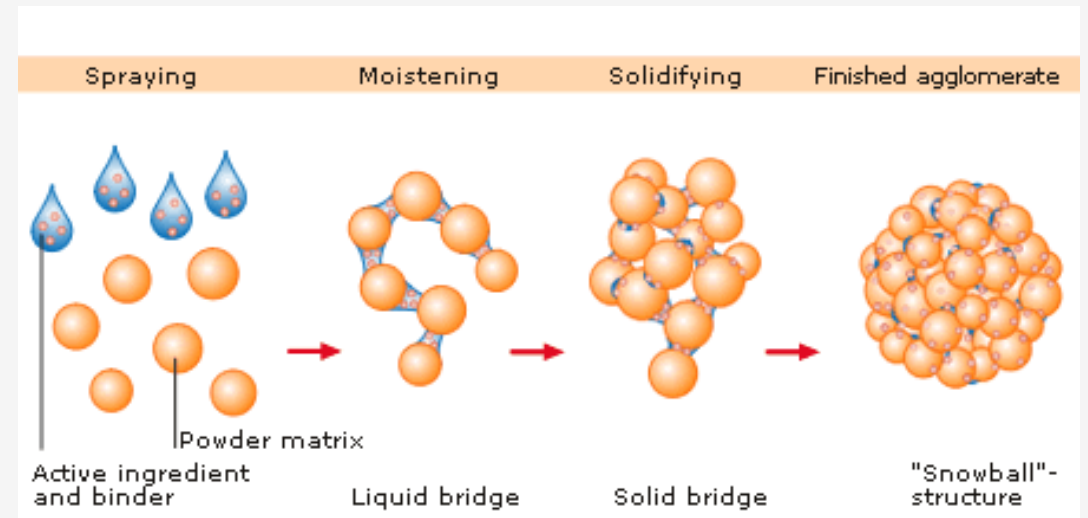
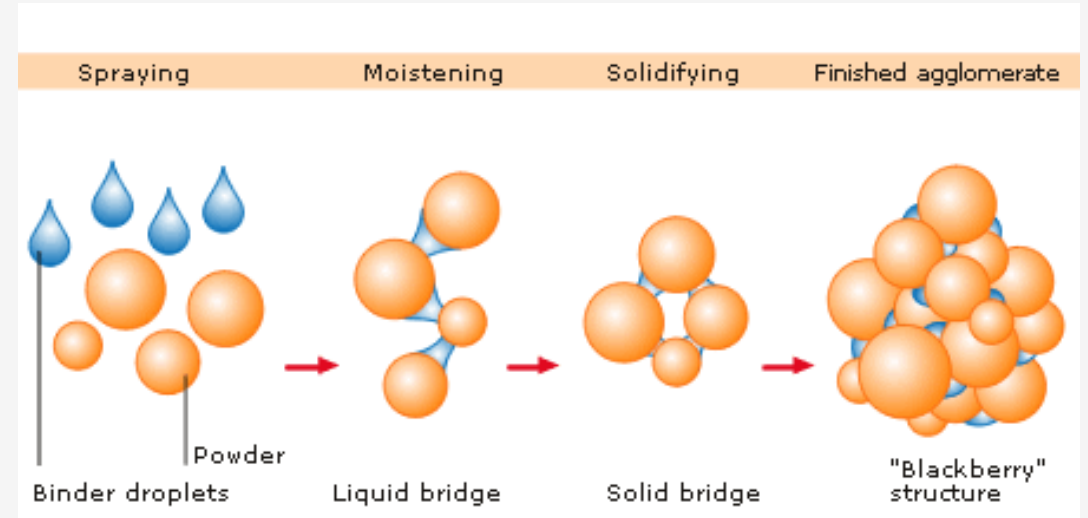
## 1.2. WET AGGLOMERATION - MECHANISMS

During the agglomeration process, a **liquid binder (water)** is **sprayed** over an **agitated powder bed**.

It generates **attractive interactions** and **links** between the native particles and...

... Promotes the **spatial arrangement of the native particles** with the binder.

The native particle characteristics, like the **particle size**, control the agglomeration **mechanisms** and the **end properties** of the products.



<http://www.cjtech.co.kr/Process%20Principles%20Agglomeration%20Granulation.htm>

## 1.3. HYDROTEXTURAL APPROACH

**Hydrotextural approach** is used to describe agglomeration mechanisms based on changes in **compactness** and **diameter** of the agglomerates as a function to the **water content**.

Hydrotextural diagram is limited by the **saturation curve** until which agglomerates are completely filled by water.

Agglomerates were analysed by measuring :

**Size**

**Water content**

**Compactness**

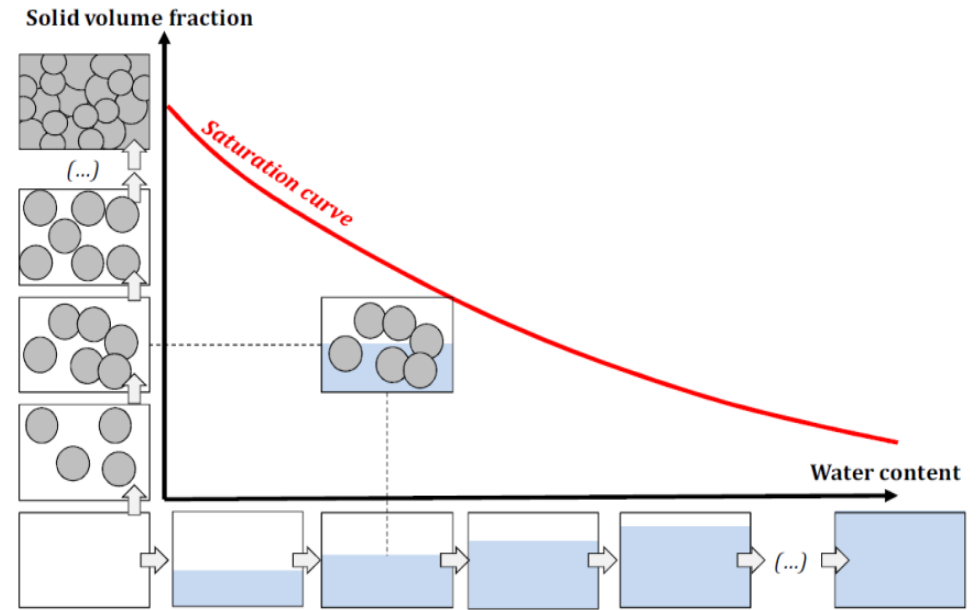
**Saturation degree**

= Median diameter

= *Mass of water / Mass of dry product*

= *Volume of solid / Total Volume*

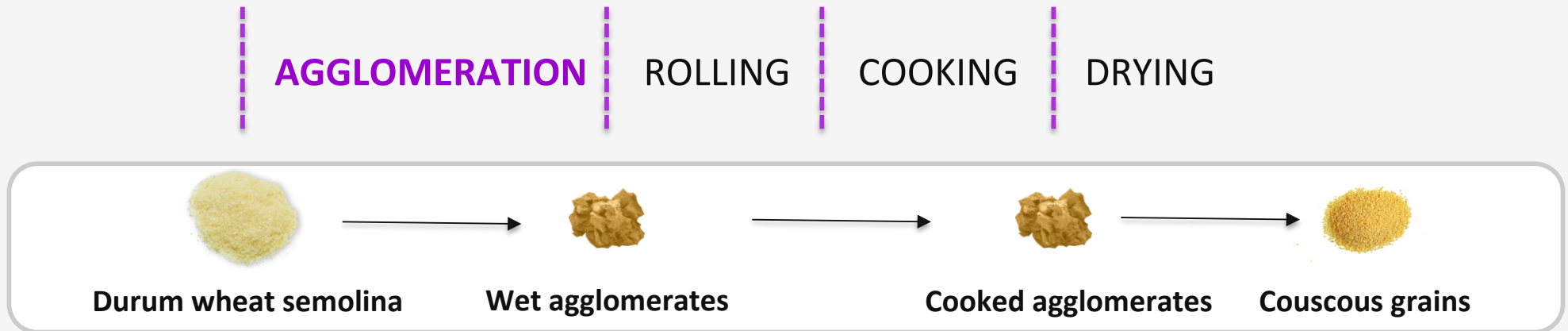
= *Volume of liquid / Volume of void*



Schema of the hydrotextural diagram (Ruiz et al., 2005)

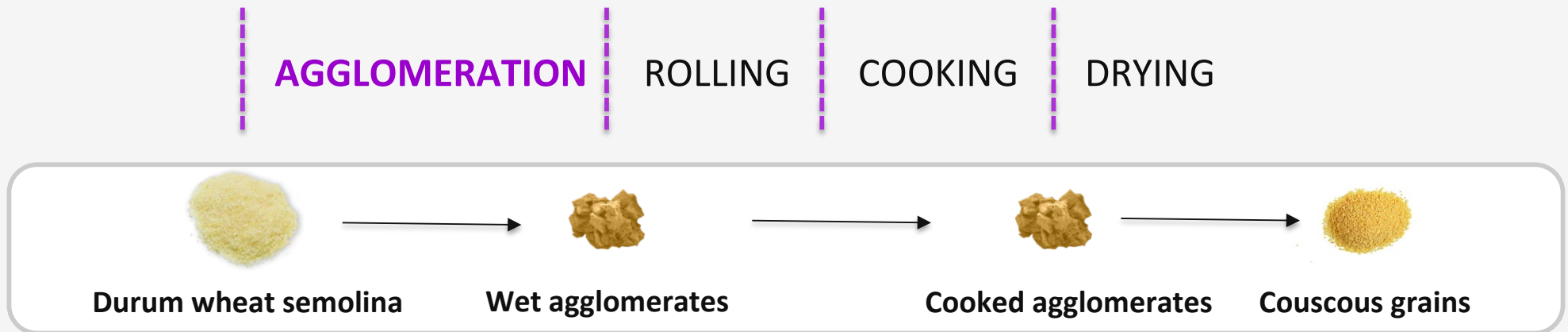
## 1.4. OBJECTIVES

No study on the impact of the particle size of native semolina during the agglomeration in the process of the couscous grain has been conducted yet.



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### OUR OBJECTIVES WERE :

- To describe the agglomeration operation of the couscous grains by analysing experimental investigations.
- To understand how the particle size distribution of semolina impact the agglomeration operation of the couscous grains.

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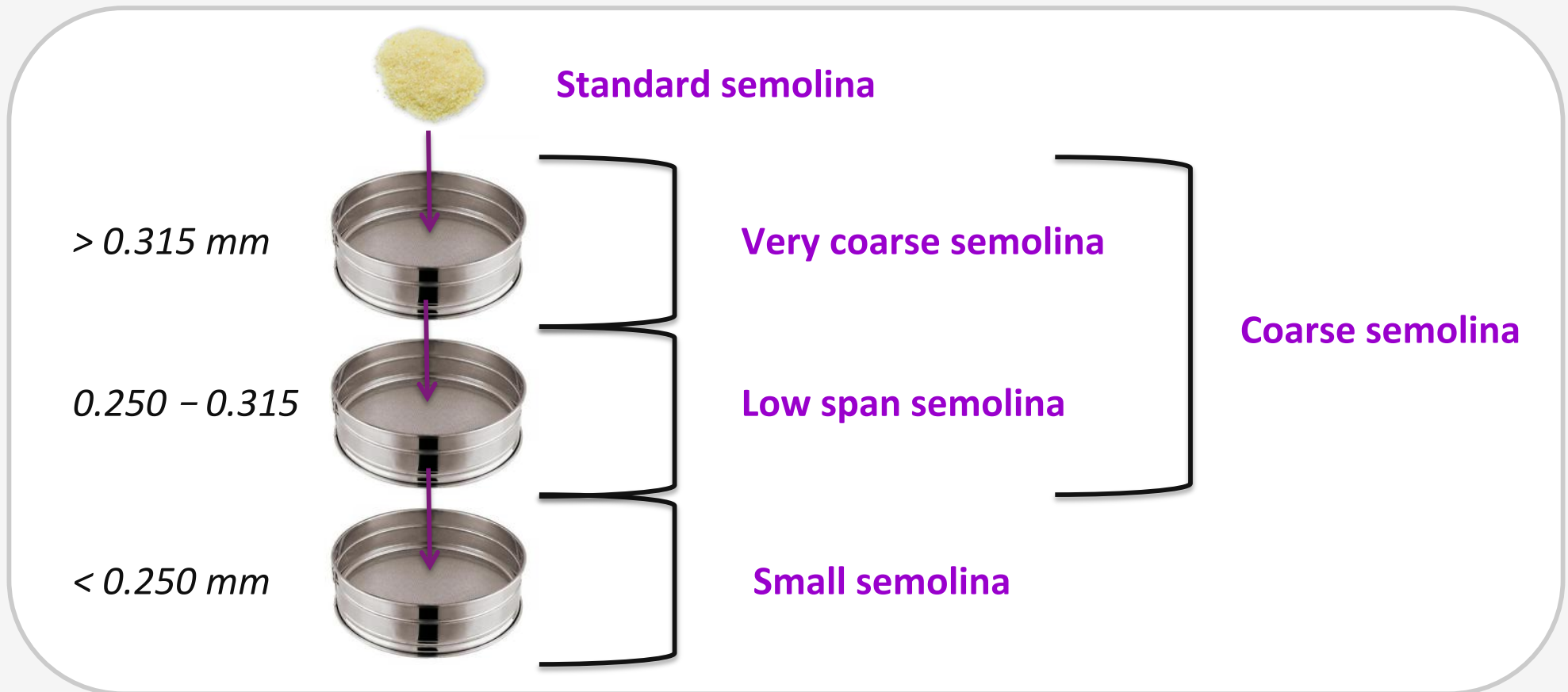
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CONCLUSION



## 2.1. NATIVE WHEAT POWDERS

Durum wheat semolina of industrial quality was used as “standard semolina”.



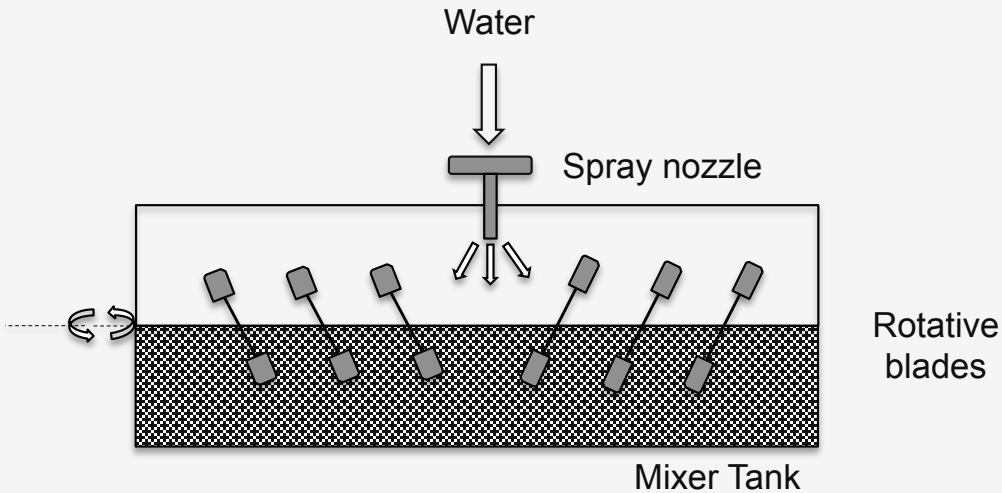
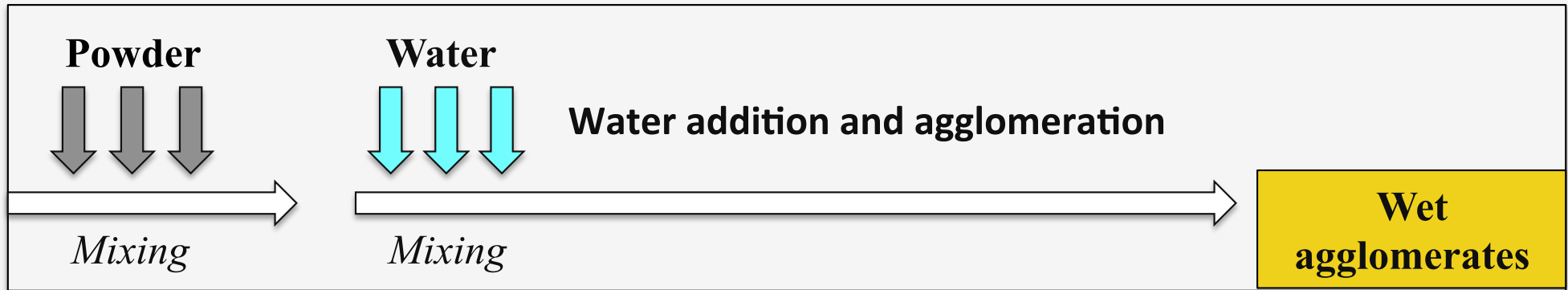
**4 fractions of semolina** with different particle size distributions were obtained by sieving the standard semolina.

## 2.1. NATIVE WHEAT POWDERS

The selected semolina allow studying the effect of particle size distributions:  $d_{50}$  and span, with **almost similar values of the physico-chemical properties.**

	Standard	Small	Low span	Coarse	Very coarse	Methods
$d_{50}$ ( $\mu\text{m}$ )	<b>287</b>	<b>129</b>	282	<b>339</b>	<b>393</b>	Laser granulometry
Particle size span (-)	1.51	1.58	<b>0.92</b>	1.02	0.77	Laser granulometry
Water content (%)	17.30	15.70	16.00	13.90	16.10	Method 44-15A AACC
Protein content(%)	12.4	13.02	12.66	12.39	12.35	Method 050 (AFNOR
True density $\rho_s^*$ ( $\text{g}/\text{cm}^3$ )	1.42	1.46	1.45	1.45	1.45	Helium pycnometry

## 2.2. DIRECT WET AGGLOMERATION PROCESSES



### Horizontal mixer

- **Low shear** conditions
- Two horizontal **shaft axes**
- Laboratory scale
- Constant mixing rate (100 rpm-15min)



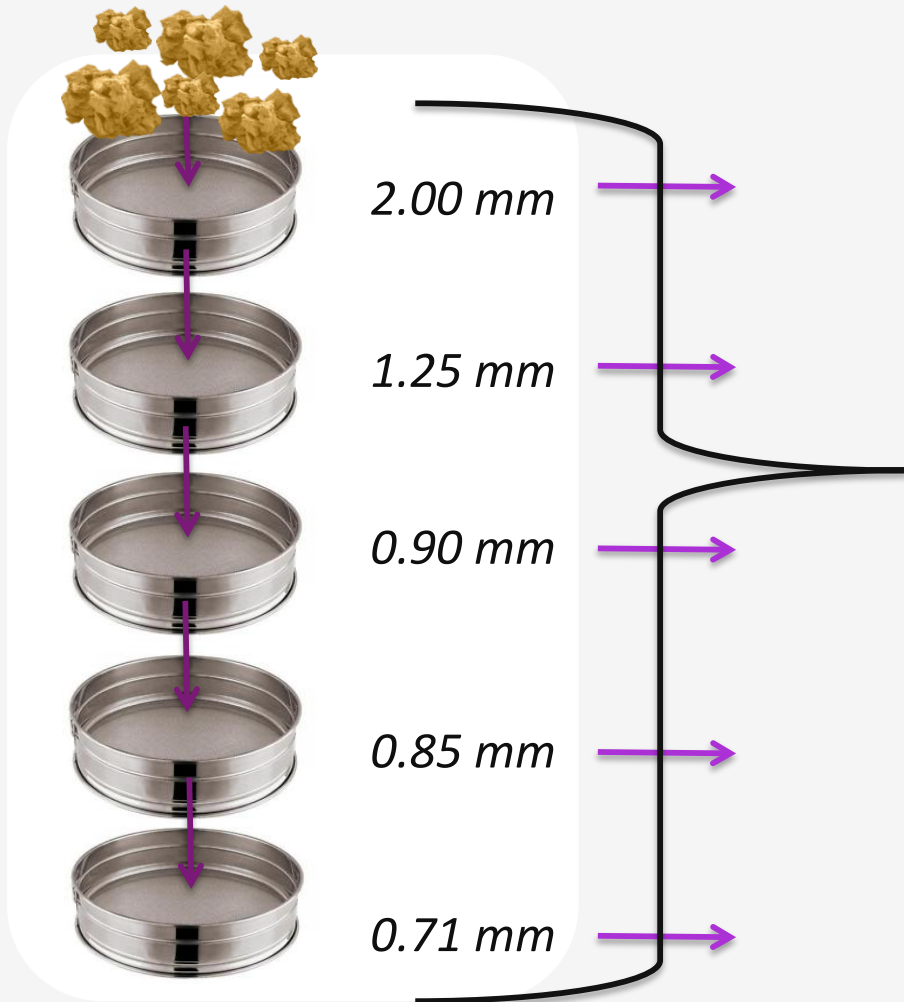
### Single fluid nozzle

- $d_g = 240 \mu\text{m} \rightarrow$  Change in the ratio  $d_{50}/d_g$
- Flow rate =  $2 \text{ g}\cdot\text{sec}^{-1}$
- Final water content =  $0.45 \text{ g/g}$  dry matter
- Constant final mass =  $1000 \text{ g}$  (semolina + water)

## 2.3. AGGLOMERATES CHARACTERISTICS

To describe the agglomeration mechanisms and the product characteristics, we evaluated the size distribution and the agglomerates characteristics **on each sieve**.

**Size distribution** by sieving: *d50, span, yield per sieves*



**Distribution of water content** by weighing after oven drying at 105°C for 24h

**Distribution of compactness** hydrostatic balance (Precisa serie 321 LX 120+ density kit)



Hydrostatic balance

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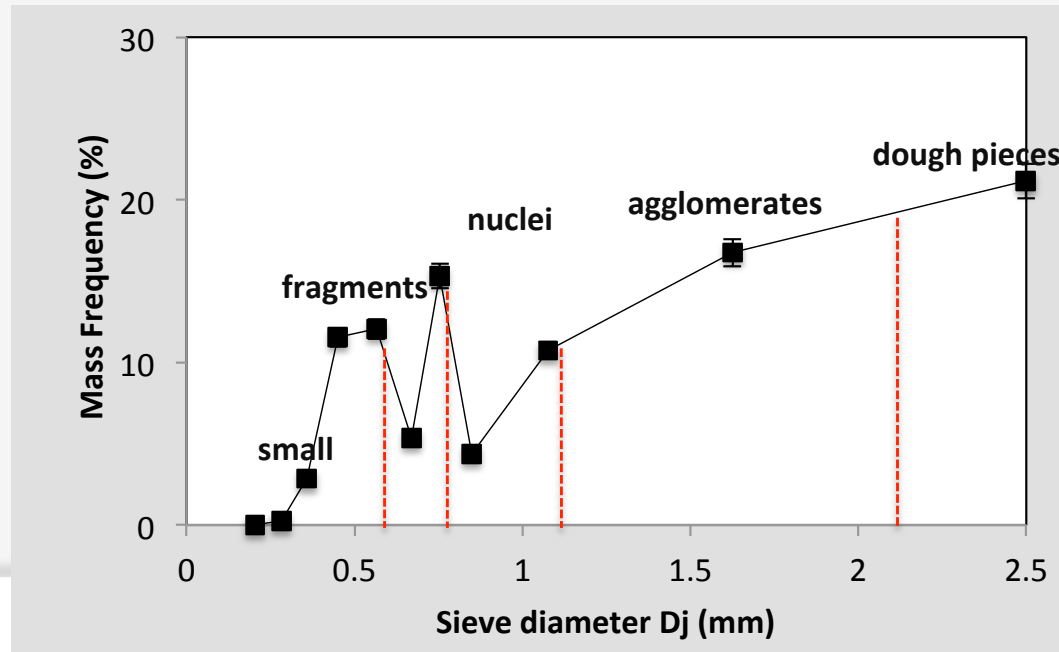
CONCLUSION



### 3.1. AGGLOMERATION OF STANDARD SEMOLINA

**Large dispersion** in size was observed. Specific shape and hydrotextural characteristics allow distinguishing **5 types of structures** according to their diameter:

- **Small** (0.3 – 0.5 mm) = native particles of semolina
- **Fragments** (0.5 – 0.6 mm) = mechanical erosion of larger structures
- **Nuclei** (0.6 – 1.0 mm) = primary association of semolina particles
- **Agglomerates** (1.0 – 2.0 mm) = association of fragments and/or nuclei
- **Dough pieces** (>2.0 mm) = association of agglomerates which passes the percolation state



### 3.1. AGGLOMERATION OF STANDARD SEMOLINA

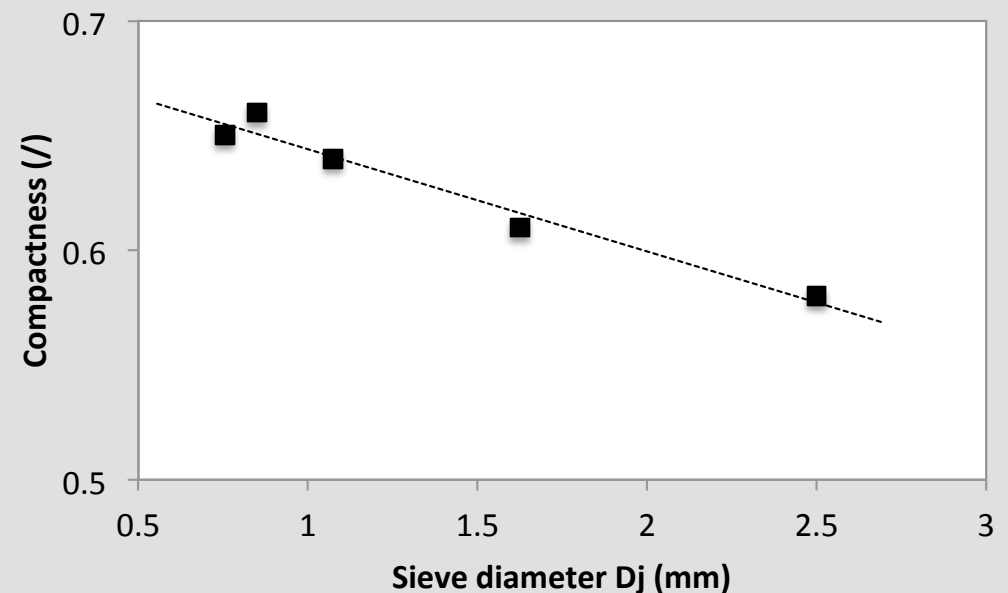
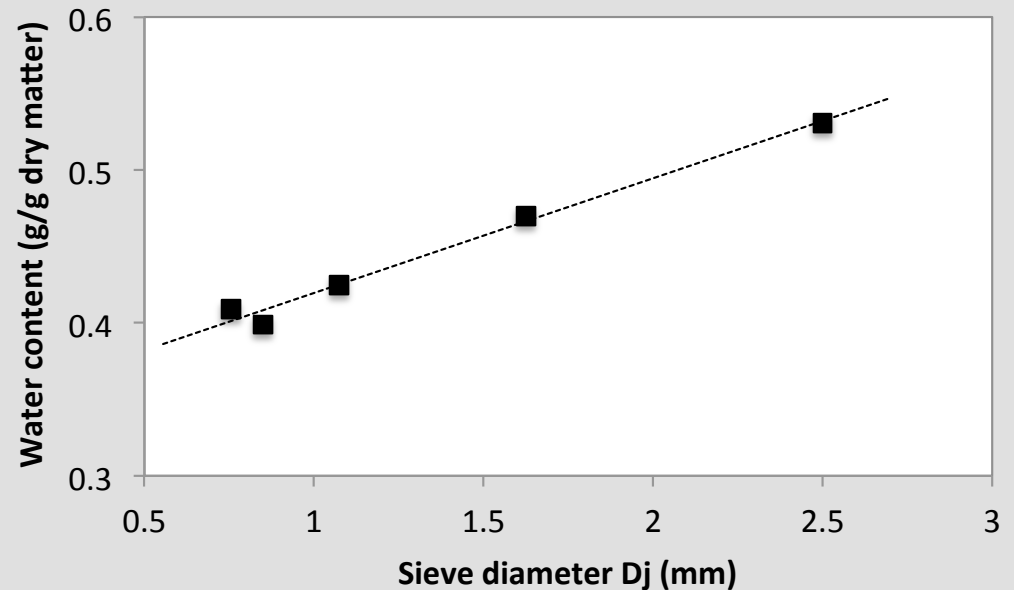
Agglomeration growth of semolina to produce couscous grains leads to:

- Increasing water content
- Decreasing compactness

according to an **increase in the median diameter** of the structures.

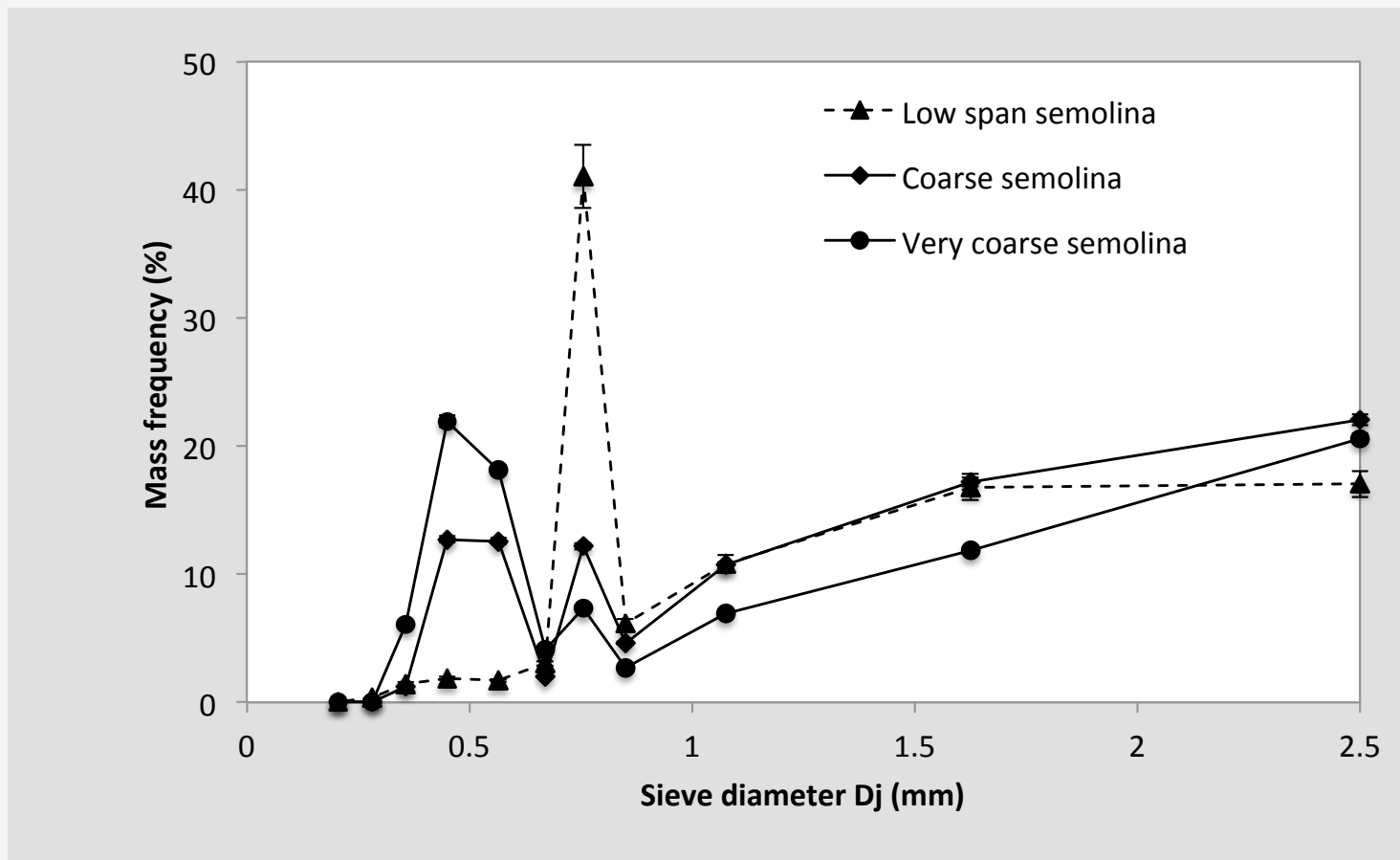
Evolution of the size distribution of the agglomerated structures with compactness shows a **continuous growth process associated with the expansion of their internal structure.**

Mechanisms deal with (i) a **classical growth** and (ii) a **fragmentation of dough pieces.**



## 3.2. IMPACT OF THE DIAMETER

**5 types of structures** are presents in the mixer after the agglomeration. Ratio of each structure is different with a change in the  $d_{50}$  of the size distributions of semolina.



The **median diameter** of the native semolina strongly impact the agglomeration yield

### 3.2. IMPACT OF THE DIAMETER

An increase of the d50 of the semolina leads to:

→ **increasing the dough pieces, the small and the fragments.**

	d50 ( $\mu\text{m}$ )	Small (%)	Fragments (%)	Nuclei (%)	Agglomerates (%)	Dough pieces (%)
Low span semolina	282	3.7	1.7	41.0	27.6	17.0
Coarse semolina	339	13.9	12.6	12.2	27.9	22.1
Very coarse semolina	393	28.0	18.1	7.4	18.8	20.6

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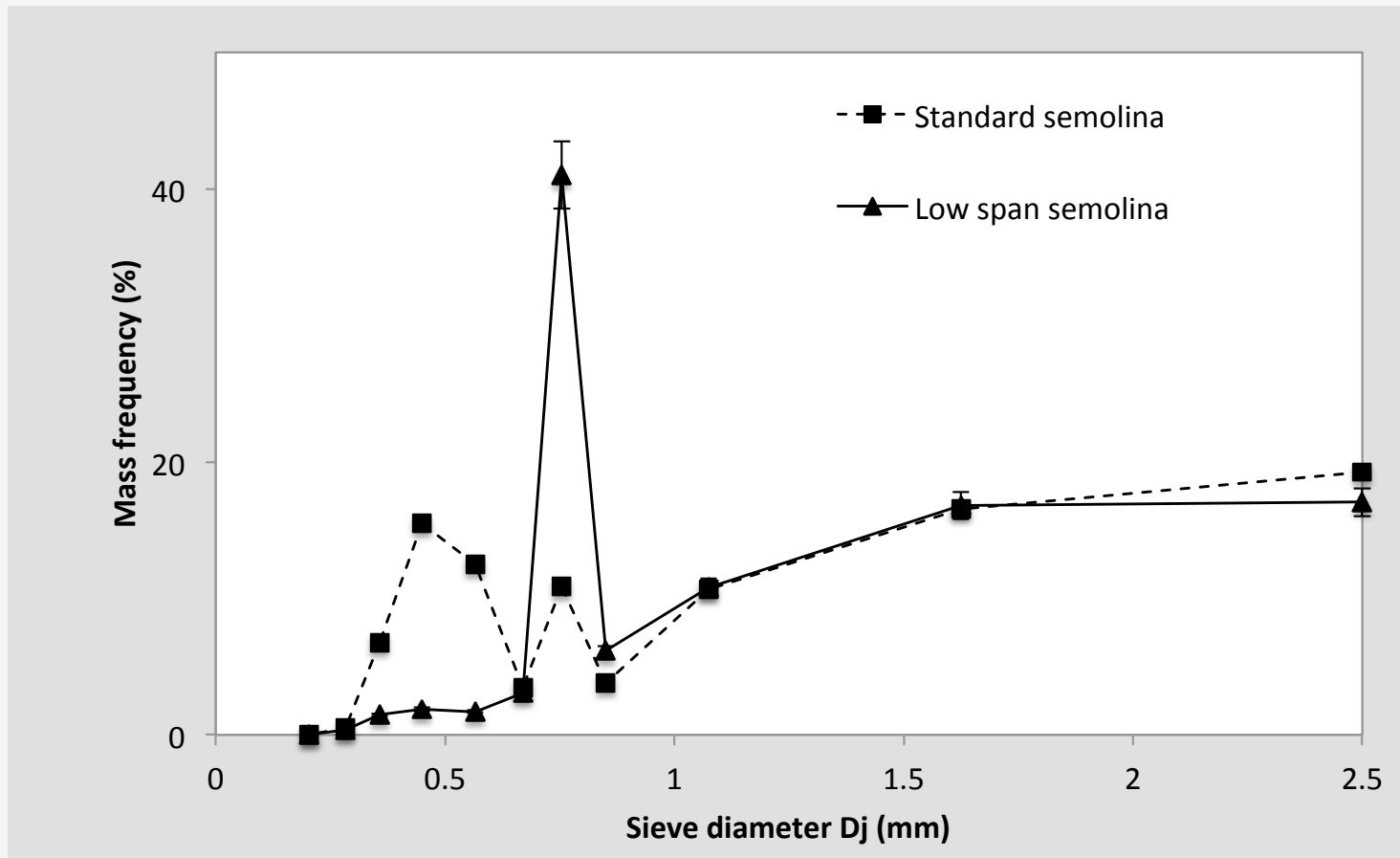
→ **decreasing the nuclei and the agglomerates.**

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### 3.3. IMPACT OF THE SPAN

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The **span** of the native semolina strongly **impact the agglomeration yield**

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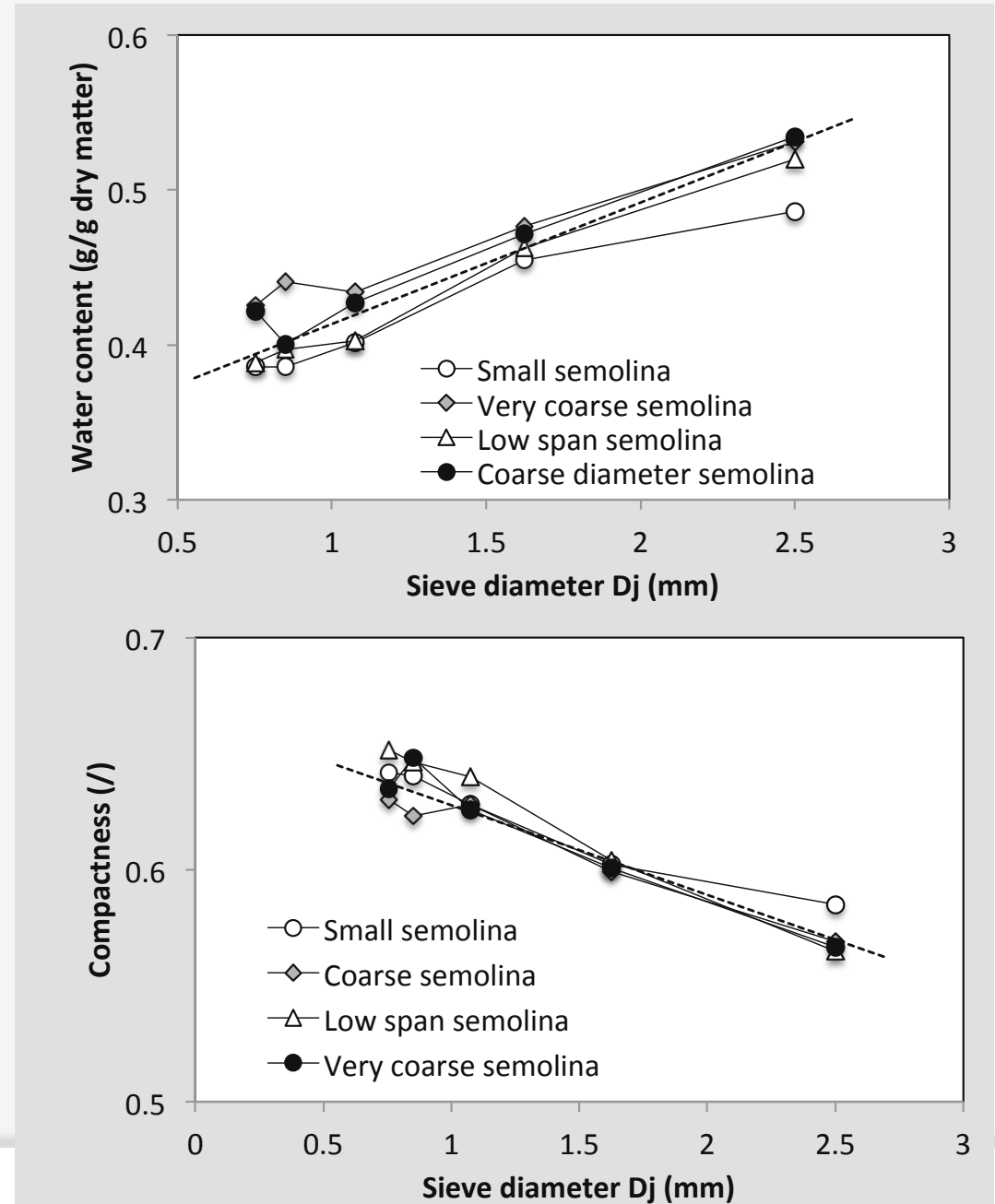
→ **decreasing the nuclei.**

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### 3.4. AGGLOMERATES CHARACTERISTICS

Same trends were observed concerning the evolution of water content and compactness according to the diameter of the structure...

...The median diameter and the span do **not impact the compactness and the water content** of each structure according to their median diameters.



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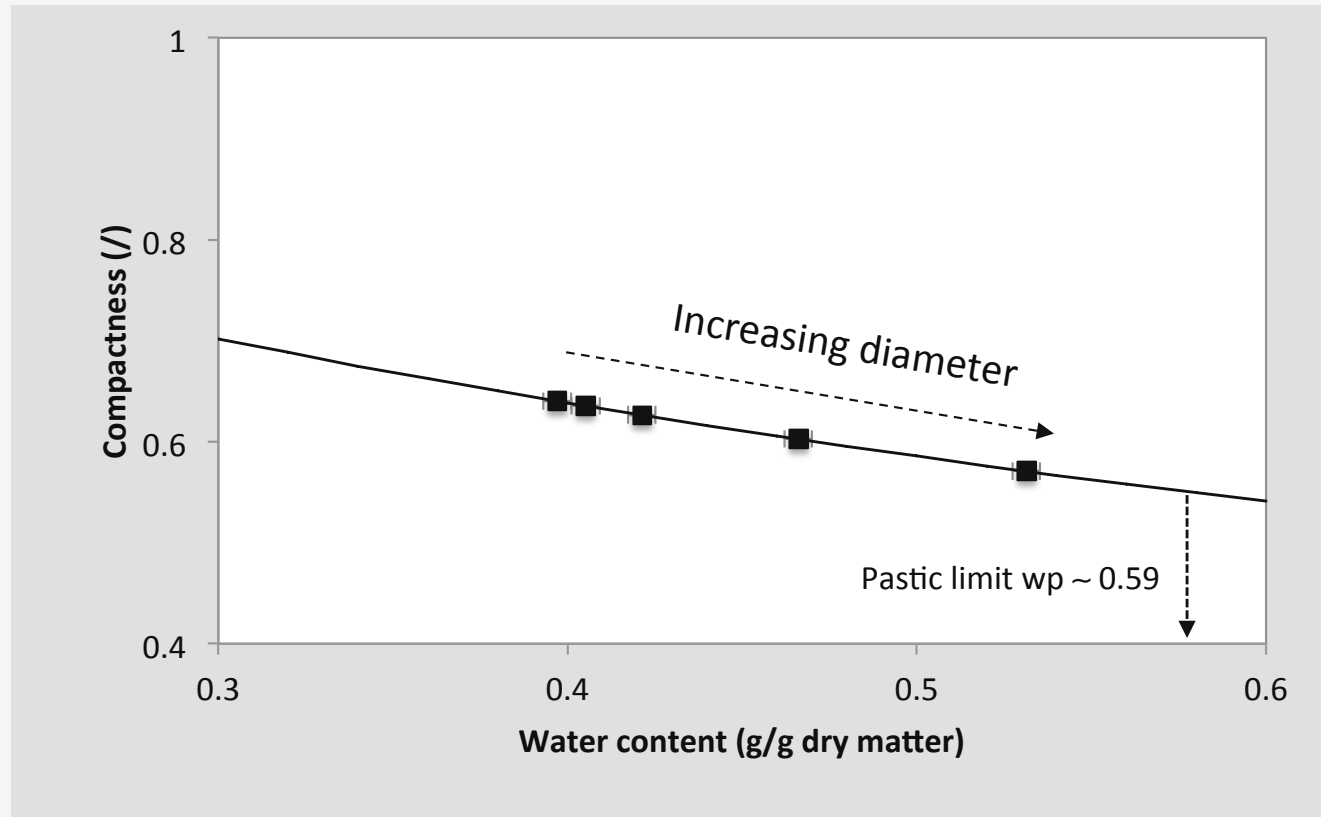
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CONCLUSION



## 4.1. HYDROTEXTURAL ANALYSIS

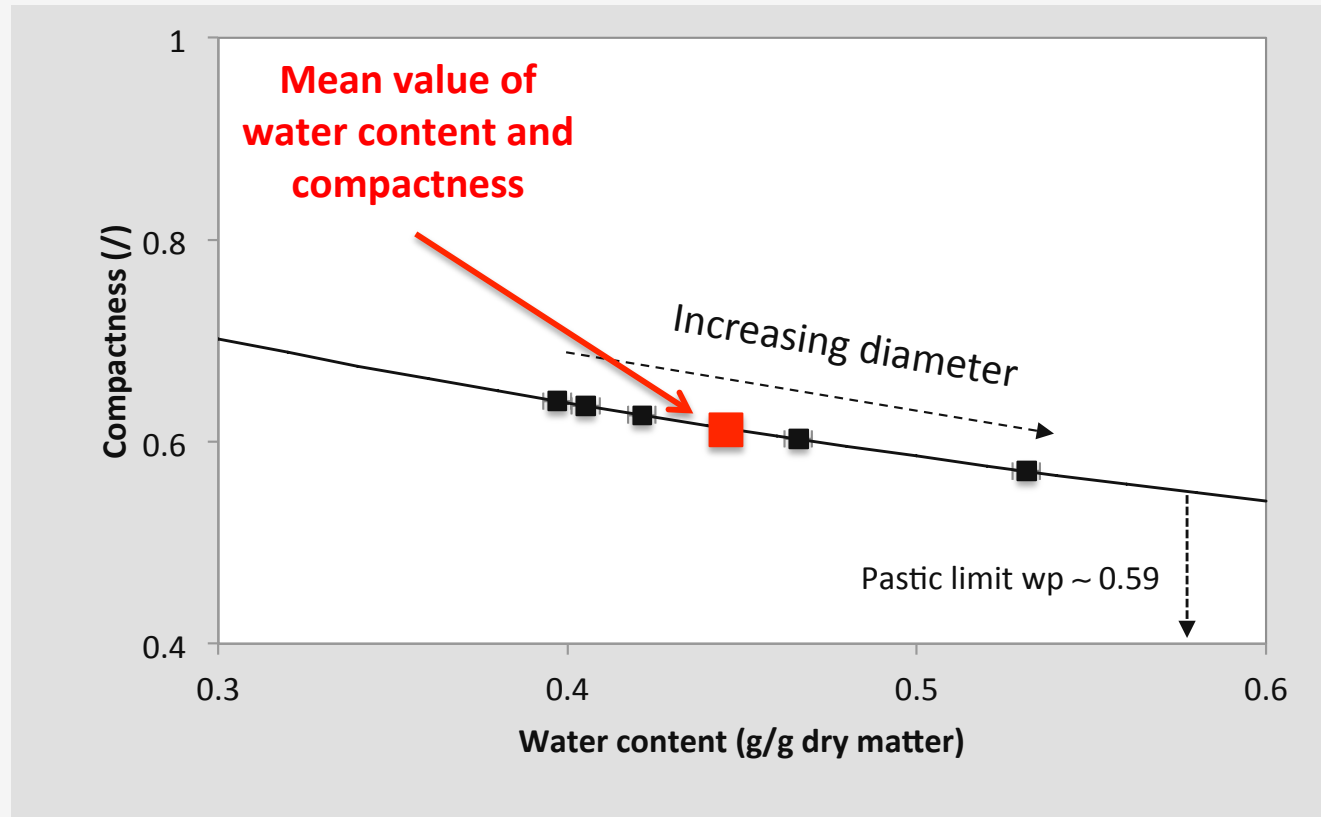
We have represented on the diagram **the values** of **water content** and **compactness** of the **different structures** for the standard semolina.



The structures on each sieve follow the saturation curve.

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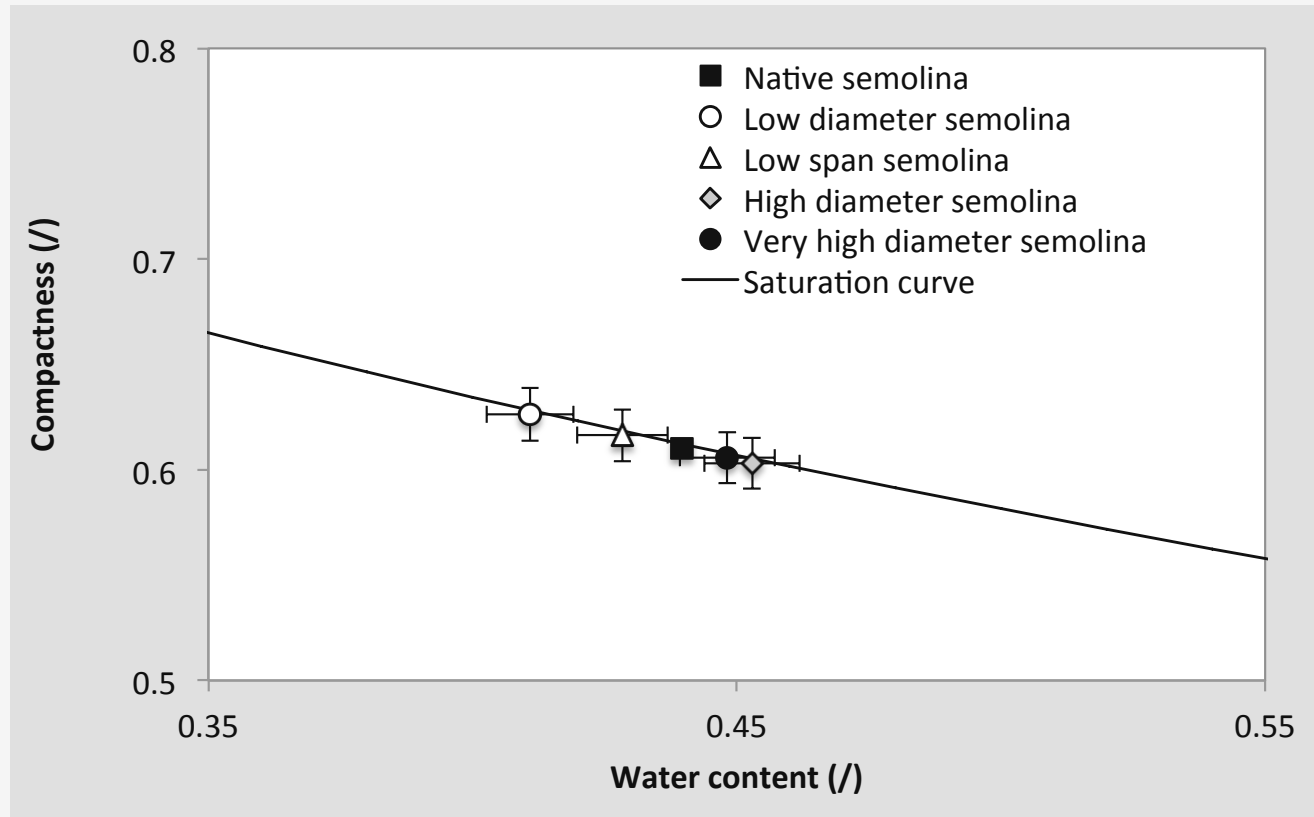
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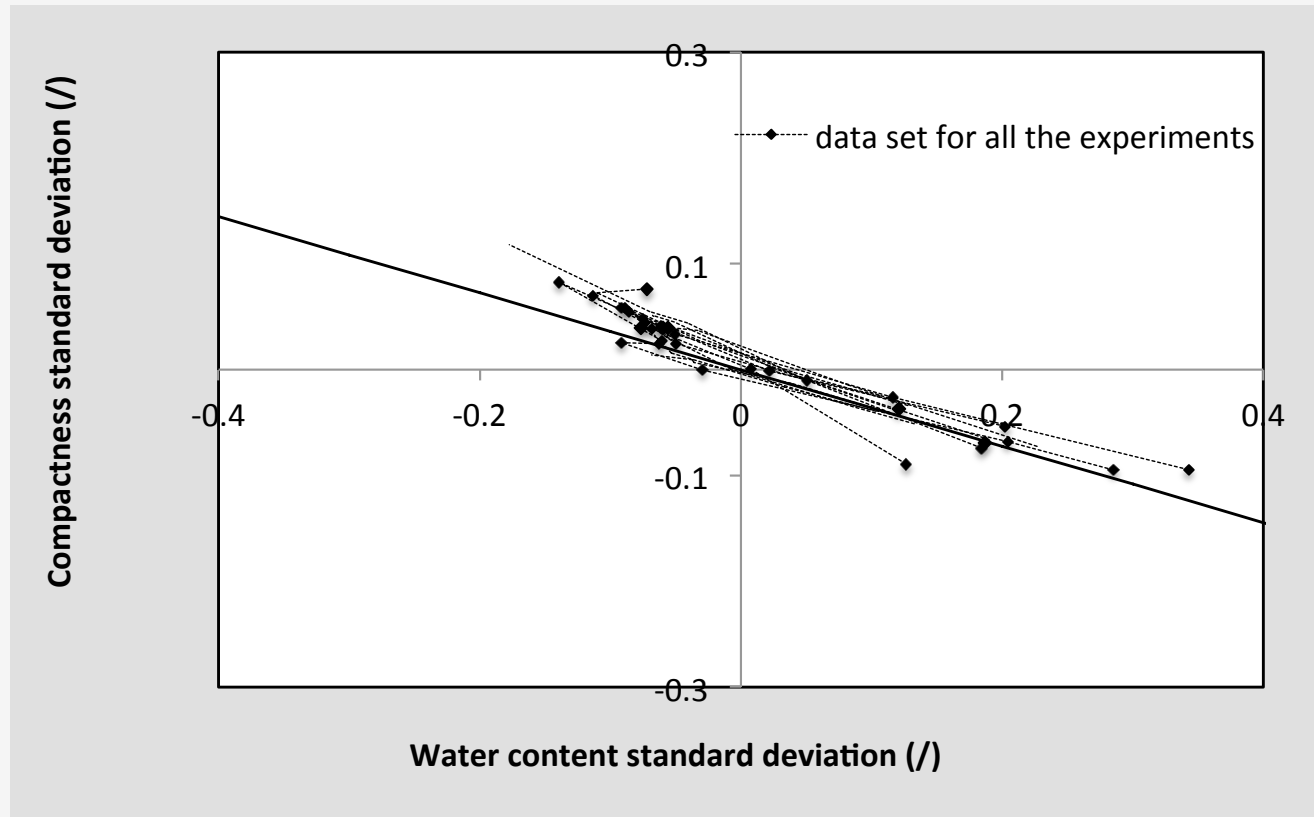
We have represented on the diagram **the mean values** of **water content** and **compactness** of the structures for all the **different size distributions** of semolina.



The size distributions of semolina do not change the hydrotextural properties of the structure: they are **all saturated by water**.

## 4.1. HYDROTEXTURAL ANALYSIS

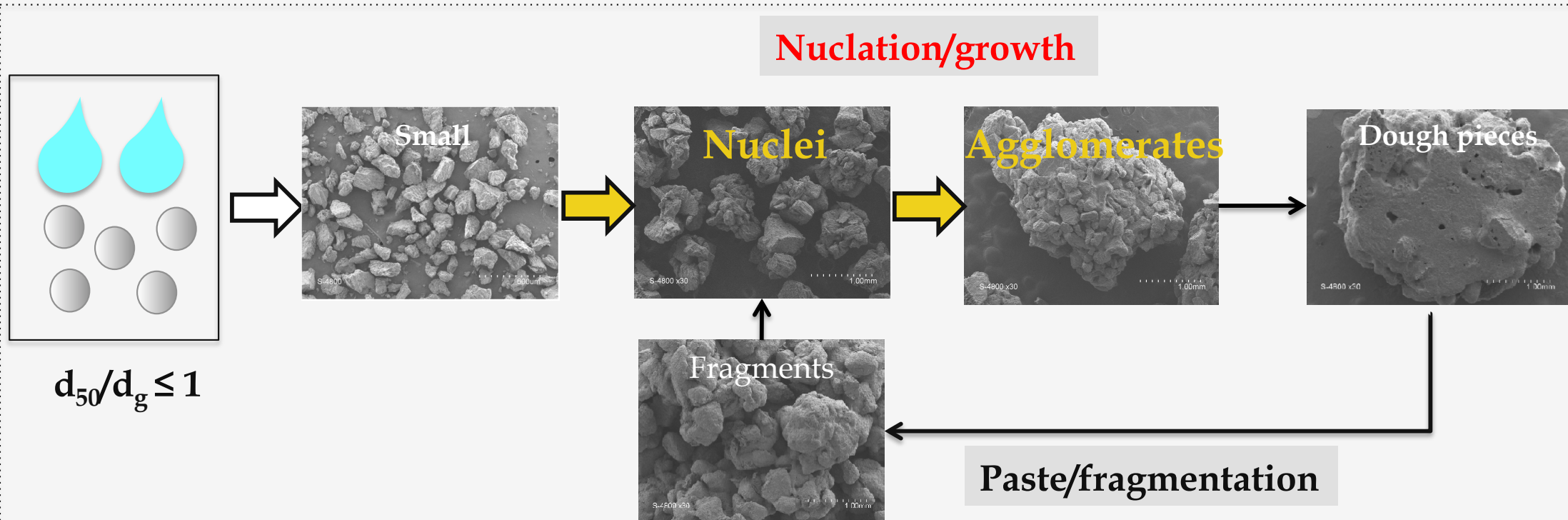
**Fluctuations** of the water content and the compactness are **correlated** around their respective mean values for all the different size distributions of semolina.



The growth of the agglomerates respects an **association by the same hydro-textural categories.**

## 4.2. AGGLOMERATION MECHANISMS

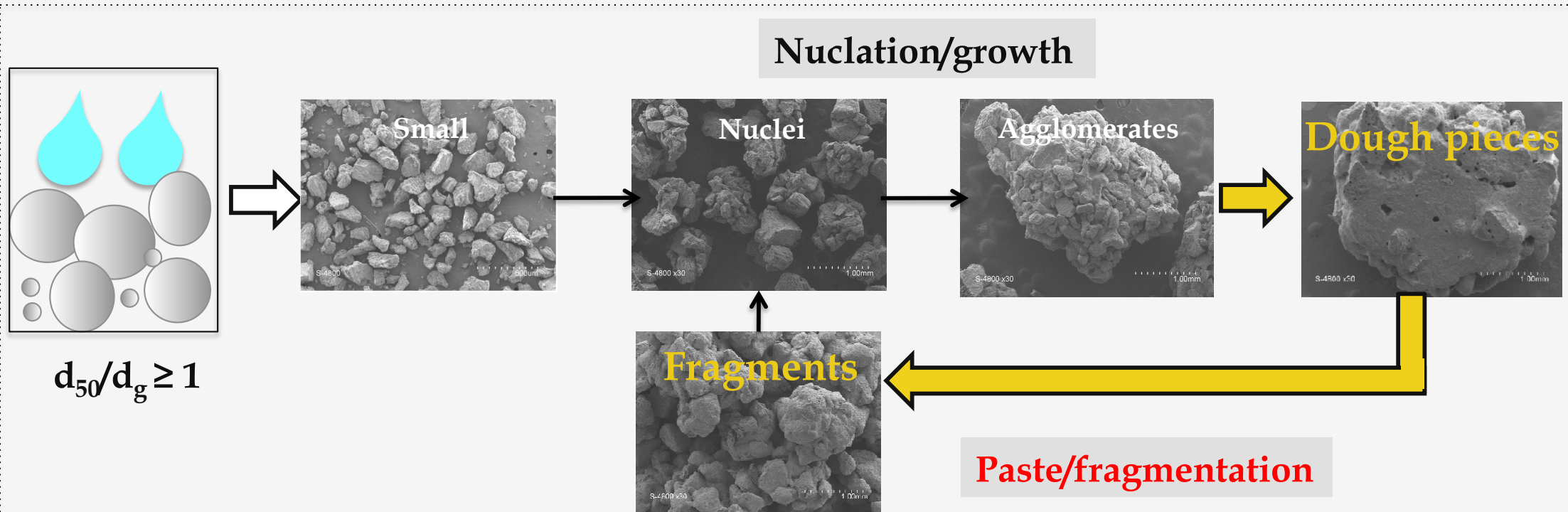
Agglomeration mechanisms was studied regarding the ratio of the median diameter of the semolina particles on the median diameter of the droplets of water ( $d_{50}/d_g$ ):



$d_{50}/d_g \leq 1$  or lower span  $\rightarrow$  Mechanism of **nucleation/growth** is favoured.

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$d_{50}/d_g \geq 1$  or higher span  $\rightarrow$  Mechanism of **paste/fragmentation** is favoured.



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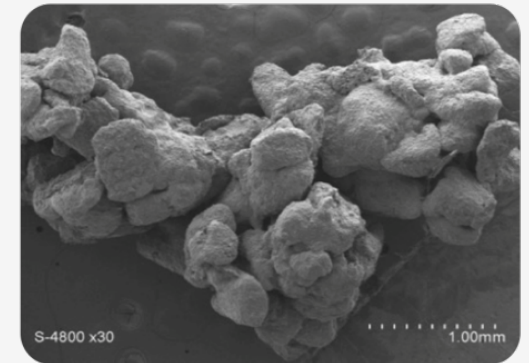
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**CONCLUSION**



# CONCLUSION

- The **size distributions** ( $d_{50}$  and span) of the semolina particles has a **significant influence on the yield** of the agglomeration.
- Continuous growth process is associated with the **expansion of the internal structure**.
- These mechanisms deal with :
  - (i) **Classical growth** of associated particles to nuclei, then nuclei to agglomerates, and percolation to local paste state (dough pieces)
  - (ii) **Fragmentation of dough pieces** into a specific population of small saturated clusters, which are able to interact with nuclei.
- As a function to **the ratio of  $d_{50}/d_g$**  one **major mode is favoured**:
  - $d_{50}/d_g \leq 1$  the nucleation/growing is favoured.
  - $d_{50}/d_g \geq 1$  the paste/fragmentation is favoured



# Thank you for your attention !

## QUESTIONS ?

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