



**HAL**  
open science

## Drying research From physical and biological mechanisms to breakthrough innovation

Luca Lanotte, Romain Jeantet

► **To cite this version:**

Luca Lanotte, Romain Jeantet. Drying research From physical and biological mechanisms to breakthrough innovation. STLOpendays, Institut National de Recherche Agronomique (INRA). UMR UMR INRA / AgroCampus Rennes : Science et Technologie du Lait et de l'uf (1253)., Mar 2019, Rennes, France. hal-02097218

**HAL Id: hal-02097218**

**<https://hal.science/hal-02097218>**

Submitted on 5 Jun 2020

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

AGRO  
CAMPUS  
OUEST



# Drying research

*From physical and biological mechanisms to breakthrough innovation*



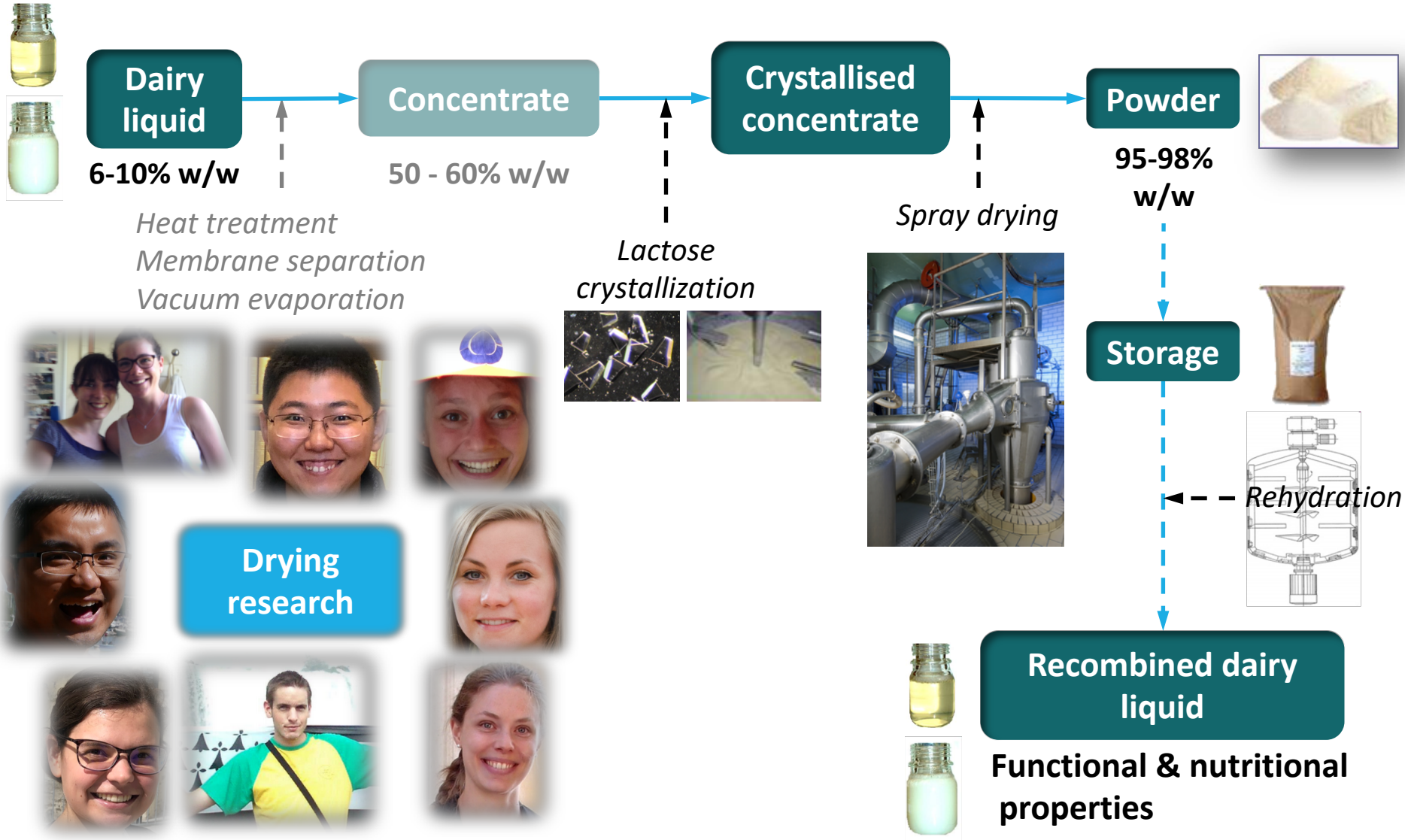
STL Open Days  
19-21 March 2019



Luca Lanotte & Romain Jeantet



# Concentration, drying and rehydration of dairy products



# Context , Research topics , Strategy

- ❑ Massive growth of Infant milk formula (IMF) / protein isolate market
- ❑ Controlled properties, incorporation of live probiotics
- ❑ Environmental and costs concern

Understand the particle formation mechanisms

Identify the key parameters that rule the powder properties and evolution

Explore new technological concepts for technological breakthrough innovation

Consider simplified & controlled models (dairy colloids, bacteria)

Observe the phenomenon at different scales to make the study possible

Combine disciplines: from biology to soft matter and chemical engineering

Partnership to feed the scientific questions and identify technological outputs

**1.**

***Exploring particle formation to control IMF properties***

**2.**

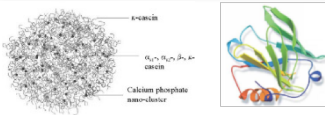
***Triggering the protection mechanisms to maximize survival of bacteria***

**3.**

***Redesigning the process for the production of whey/permeate powders***

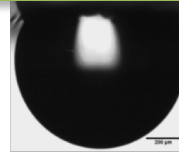
# Strategy - Study of the drying process on multi-scales

## Milk colloids



- Whey protein (WP)
- Micellar casein (MC)
- Single or mixed together

## Single droplet 3D / 2D



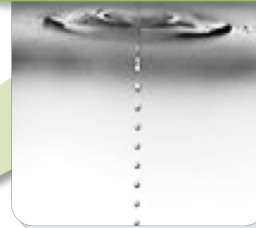
### PARTICLE FORMATION

- **Control and study** the drying kinetics
- **Record *in situ*** deformation

## Coupling methods

- **Mass balance**
- **Microscopy**
- **Rheology**

## Mono-disperse droplets



### PARTICLES PROPERTIES

- **Control** drying history
- **Get** homogenous samples
- **Analyze** physical properties

## Spraying cone of droplets



### SCALE UP

- **Validate** on an industrial scale
- **Control** powder properties

# Protein signature in the course of drying

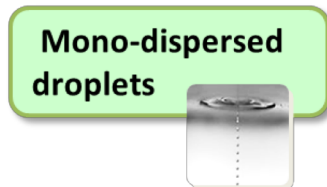
*Langmuir* 29 (2013) 15606 - 15613

*Drying Technol* 32 (2014) 1540 - 1551

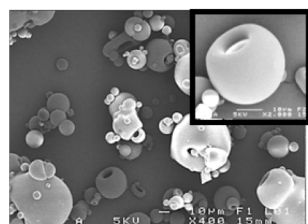
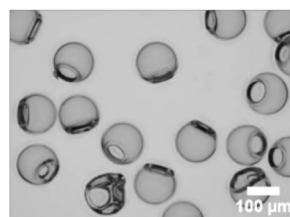
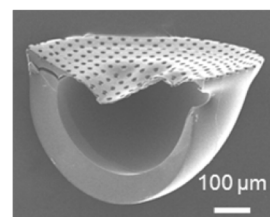
*Food Hydrocolloids* 48 (2015) 8 - 16

*Food Hydrocolloids* 52 (2016) 161 -166

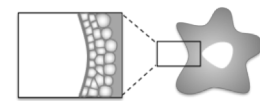
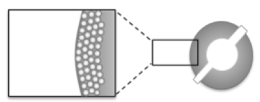
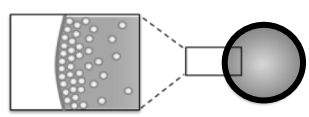
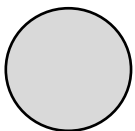
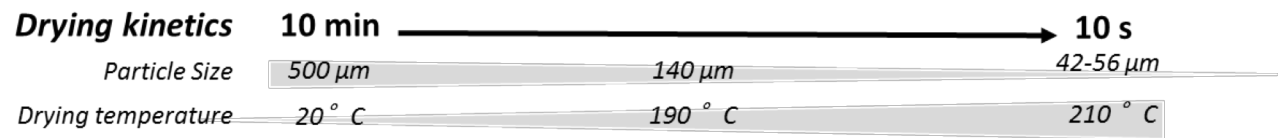
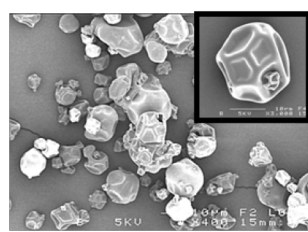
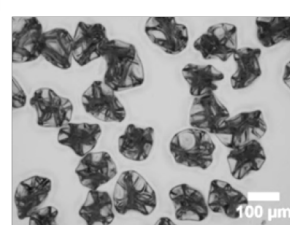
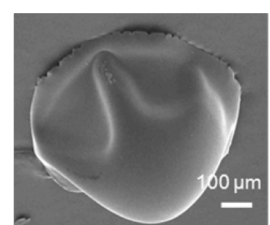
**Specific signatures of WP and MC proteins governing the particle formation / shape and properties, regardless of the drying kinetics**



**Whey Protein**



**Micellar Casein**



**Powder properties**

**1. Homogeneous kinetics & shrinkage**

**2. Skin formation: sol/gel transition**

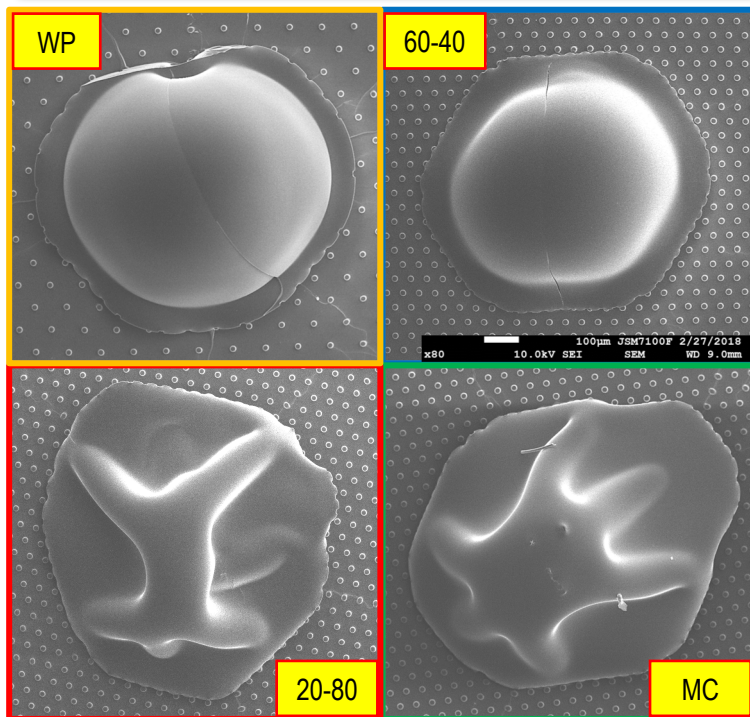
**3. Mechanical stress = f (properties)**

*Brittle (WP)*

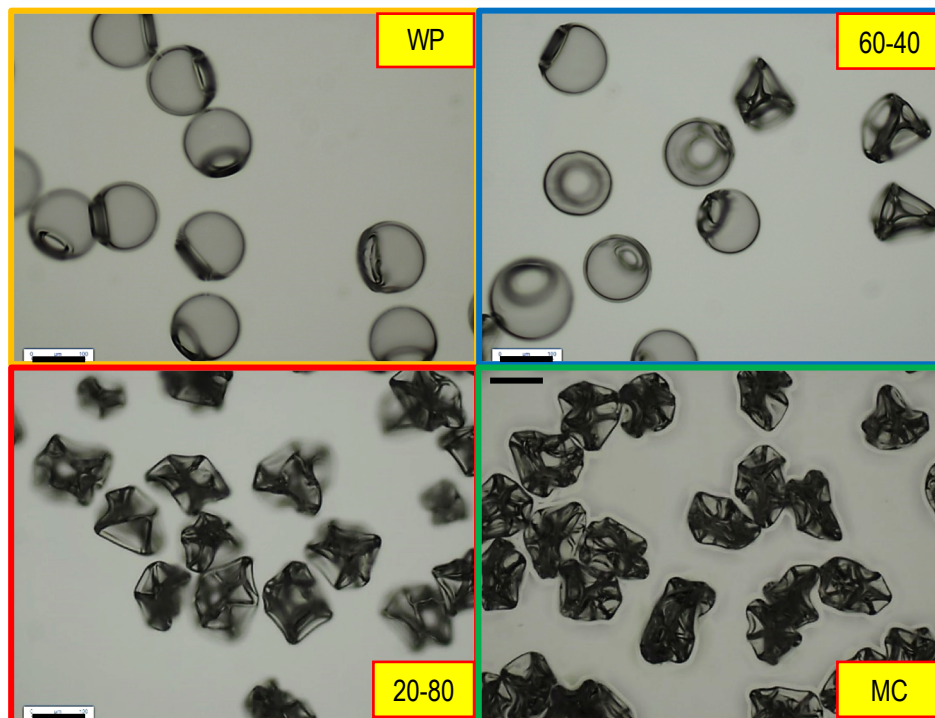
*Ductile (MC)*

# Protein signature : the case of protein mixes

## Single pendant droplet Scanning Electron Microscopy (SEM)



## Flying monodisperse droplets Optical Microscopy

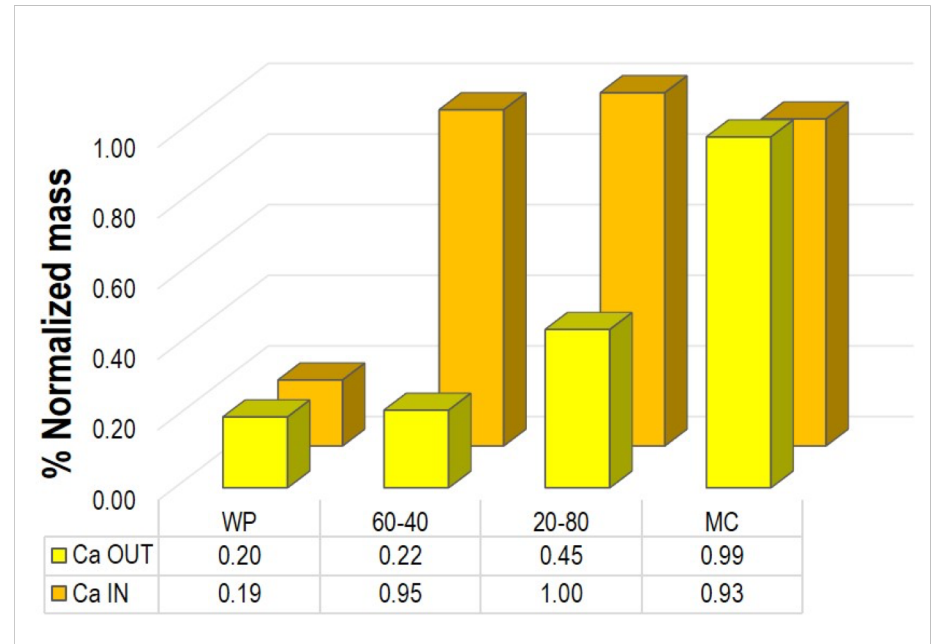
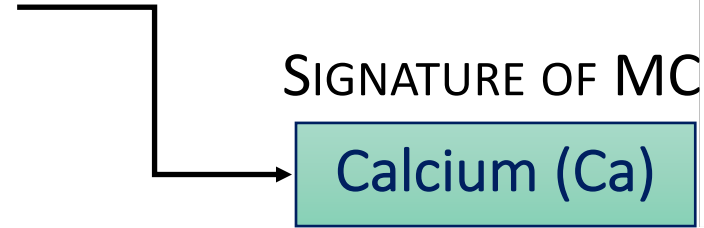
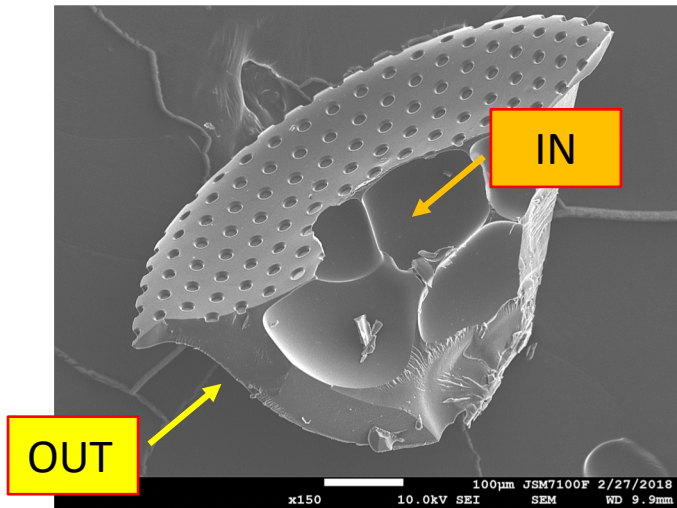


In this case of protein mixes, the major protein rules the transition and final shape of the droplet / particle



# Is the skin composition representative of the bulk?

Elemental analysis by SEM (EDS)

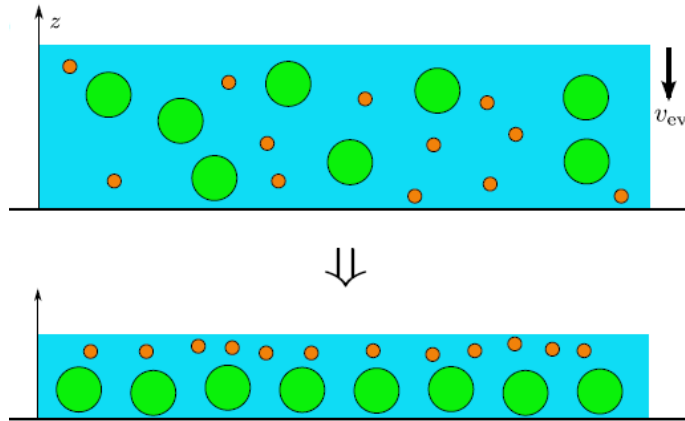


**Over-representation of WP on the external part of the dry skin in WP/MC mixes: how to explain such behavior?**

*Colloids and Surfaces A 553 (2018) 20-27*



# Drying-induced colloid behavior



**Small on top theory**

FILM FORMATION IN BINARY COLLOIDAL MIXTURES

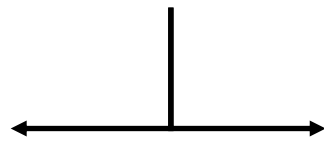
Due to osmotic pressure, smaller colloids accumulate on the external part of the skin/gelled layer passing through its interstitial pores

Zhou J. *et al.*, PRL 118, 108002 (2017).  
Fortini A. *et al.*, PRL 116, 118301 (2016).

## Characterization of the sol-gel transition in dairy mixes

Control of the nutritional properties of the powders (rehydration)

*Industrial application*



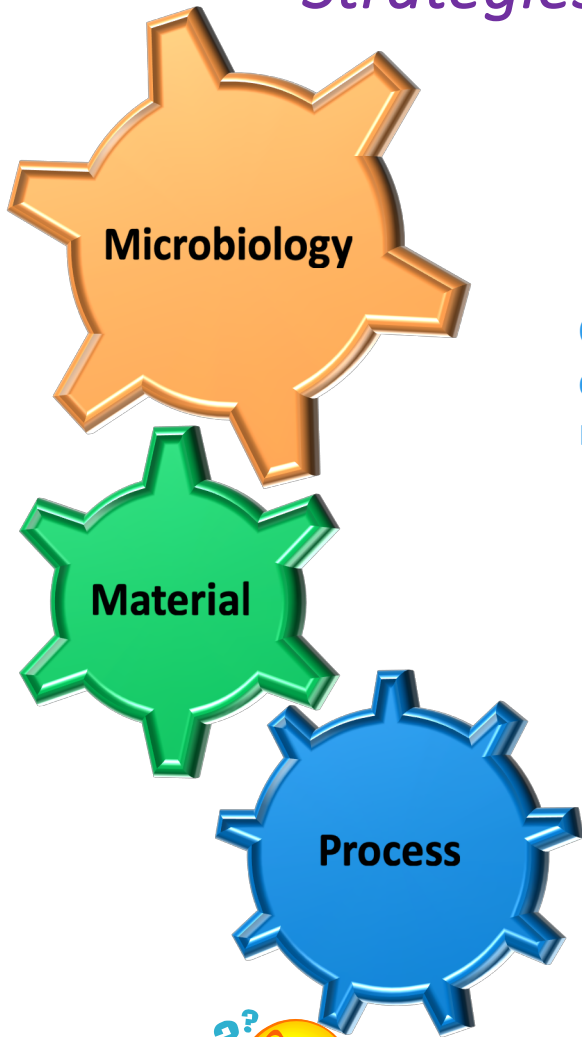
Understanding the drying mechanisms in colloidal polydisperse systems

*Physics*

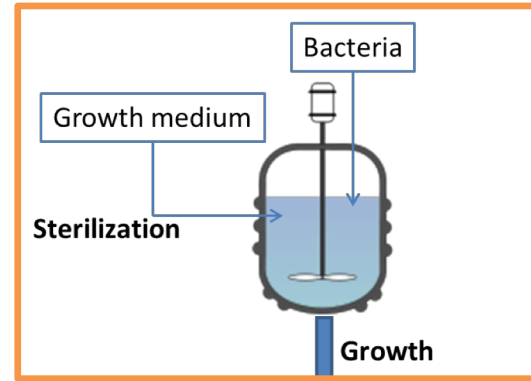
2.

***Triggering the protection mechanisms  
to maximize survival of bacteria***

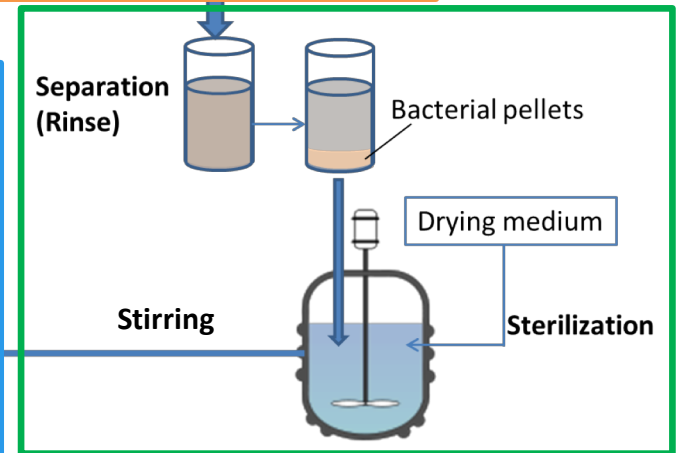
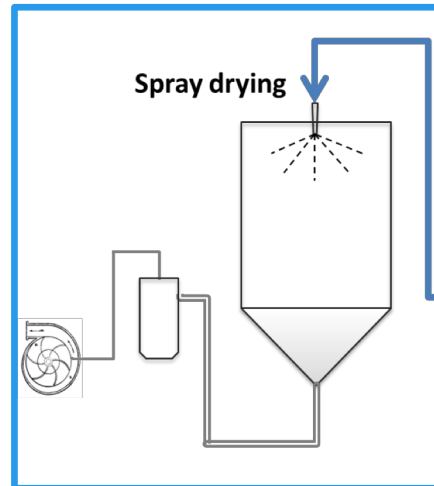
# Strategies in probiotics drying research



Optimize/moderate drying conditions: multistage, belt, etc.



Resistant strain selection, Cross-protection via sublethal stresses, etc.

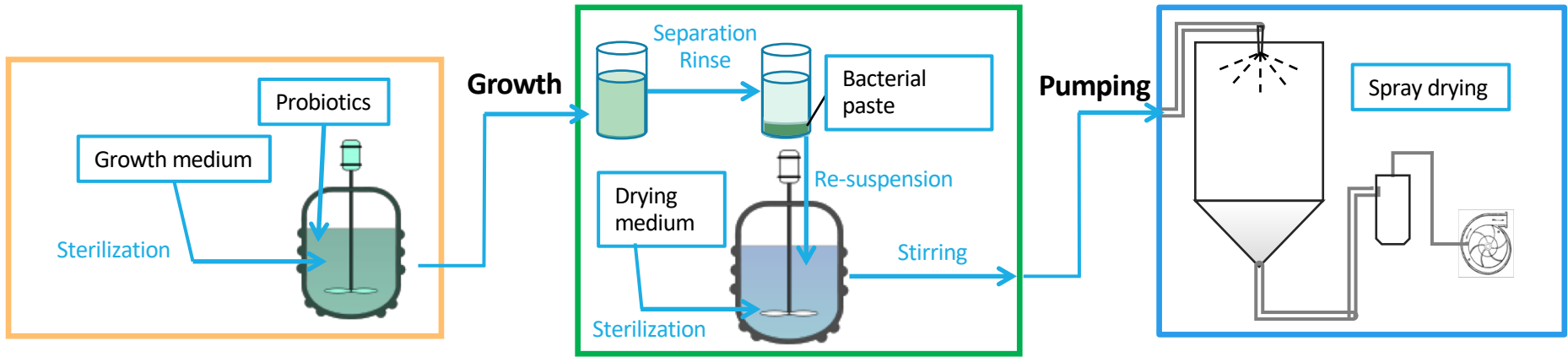


Protective food grade constituents: Carbohydrates, proteins, polymers, minerals

Coupling strategies to improve probiotics viability?



# A coupled approach for a novel process



**Cross-protection**  
Sublethal osmotic

**Stress Tolerance**

**Concentrated 2-in-1 (Growth & Drying) Medium**

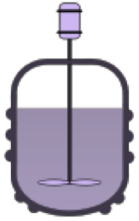
Carbohydrates  
Proteins  
Fats  
Minerals  
...

**Multi-stage drying**  
Pre-concentration

**Lower  $\theta$  / Energy savings**

# Osmoregulation induce protection mechanisms before drying

*L. casei*  
growth



30 SW

Optimal  
content

## Osmoregulation

### Protein upregulation

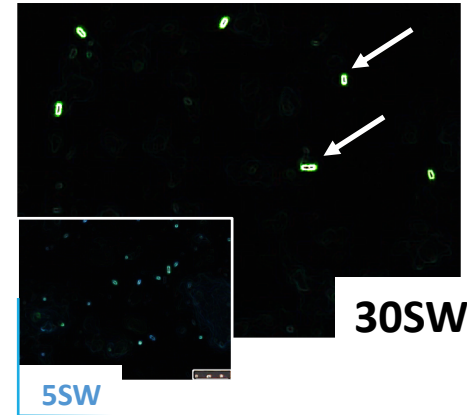
(Label-free proteomics):

- **Osmoregulation**  
*Glycine betaine transporter*
- **Stress tolerance**  
*Chaperone, Cysteine synthase*

+

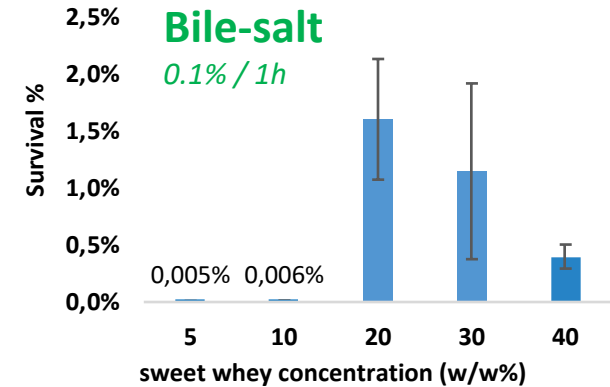
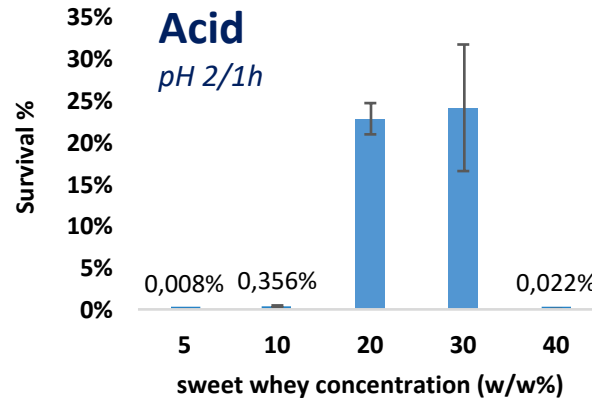
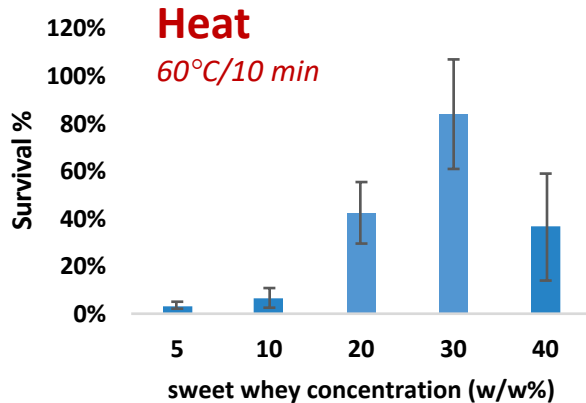
### Accumulation

- **Polyphosphates**
- **Glycogen** (*energy for long term survival*)
- **Trehalose** (*involved in anhydrobiosis*)



## Multi-stress resistant phenotype

*App Env Microbiol 82*  
(2016) 4641 - 4651

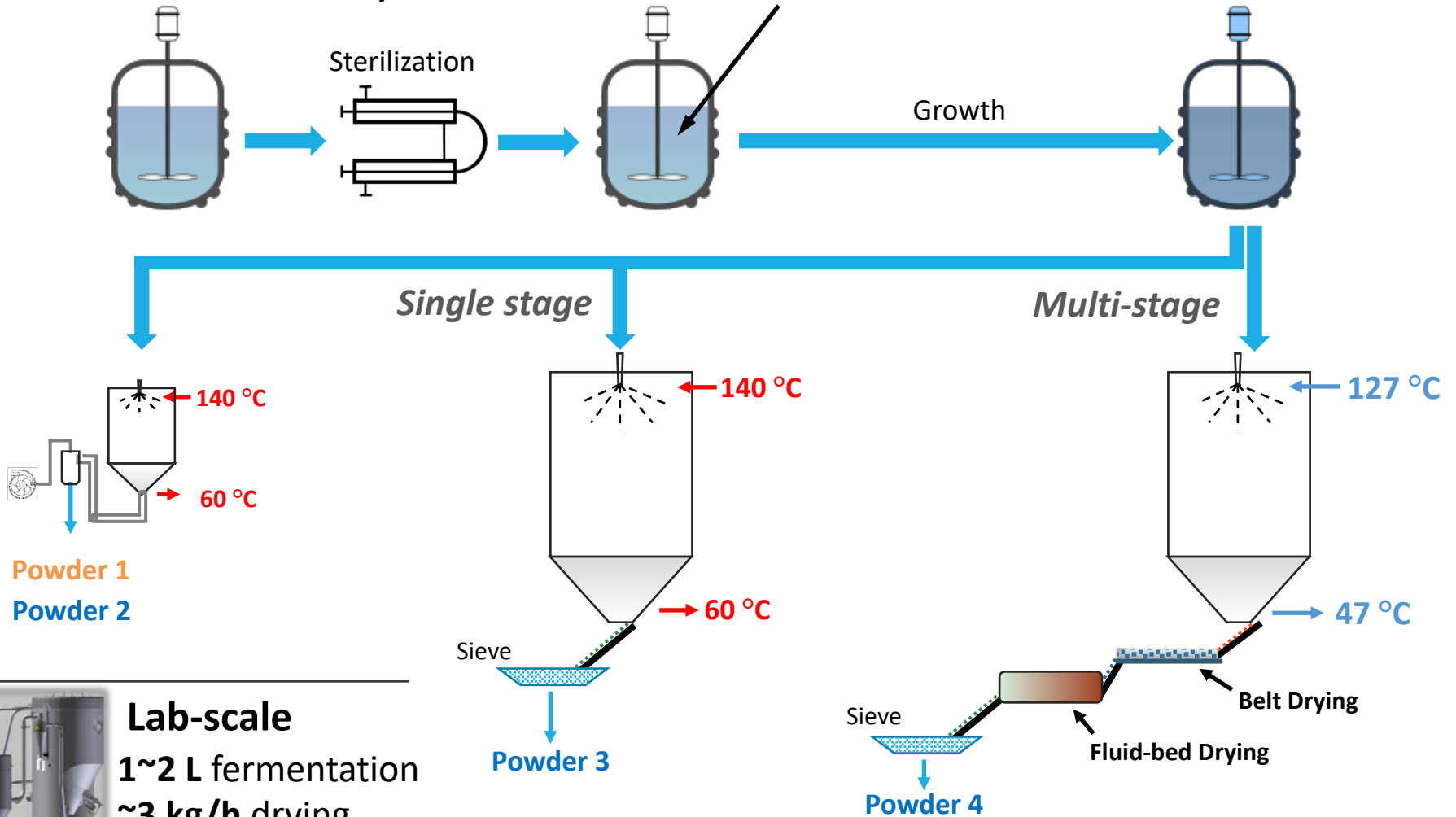


# Drying bacteria at different scales

5% or 30% sweet whey

Inoculation of bacteria

Bacterial culture



**Lab-scale**

1~2 L fermentation  
~3 kg/h drying

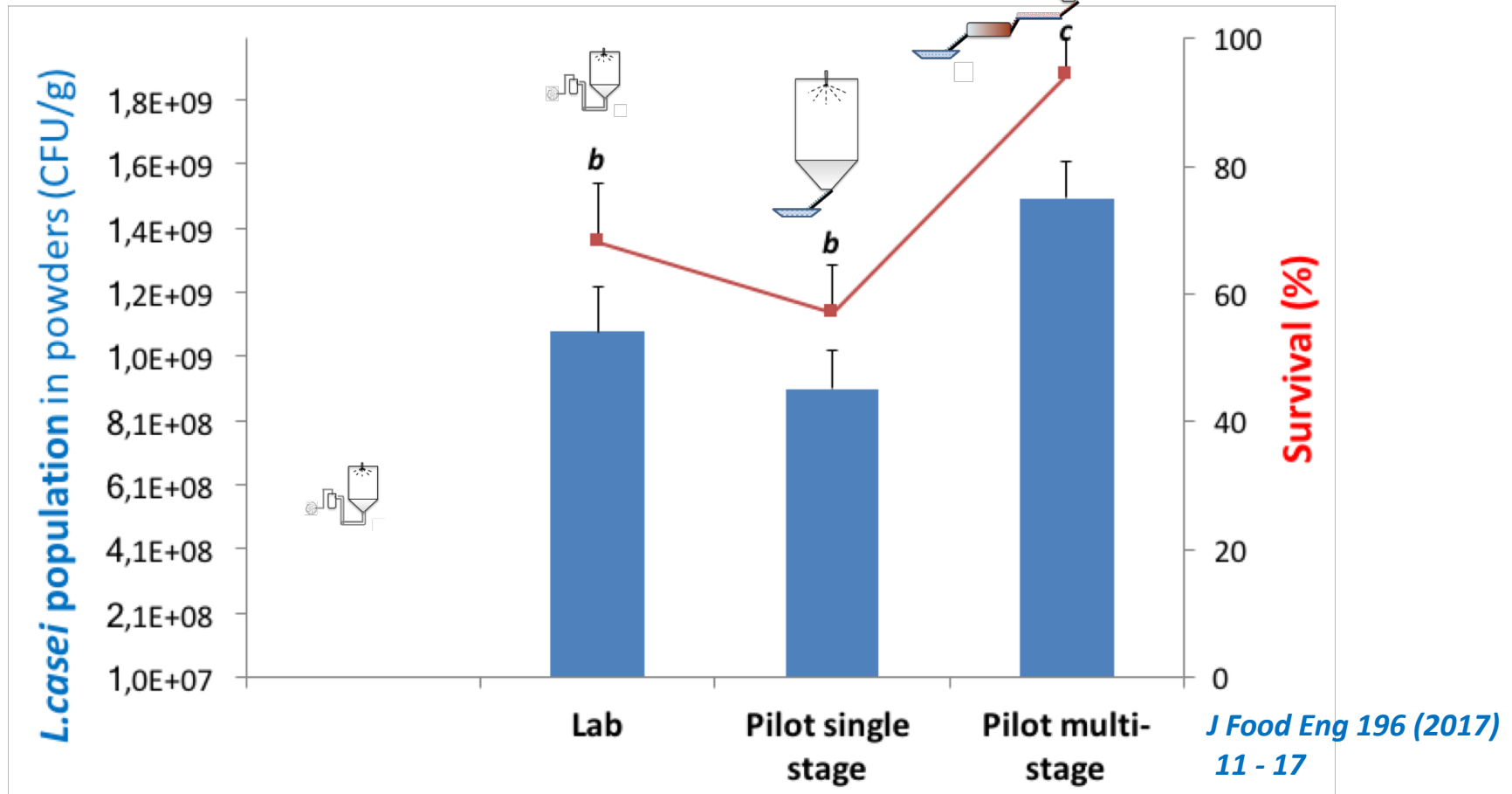


**Pilot-scale BIONOV**

500 L fermentation  
~100 kg/h drying

*J Food Eng 196 (2017) 11 - 17*

# Bacteria survival upon drying



Acquired cross protection with multi stage scheme provides 100% survival

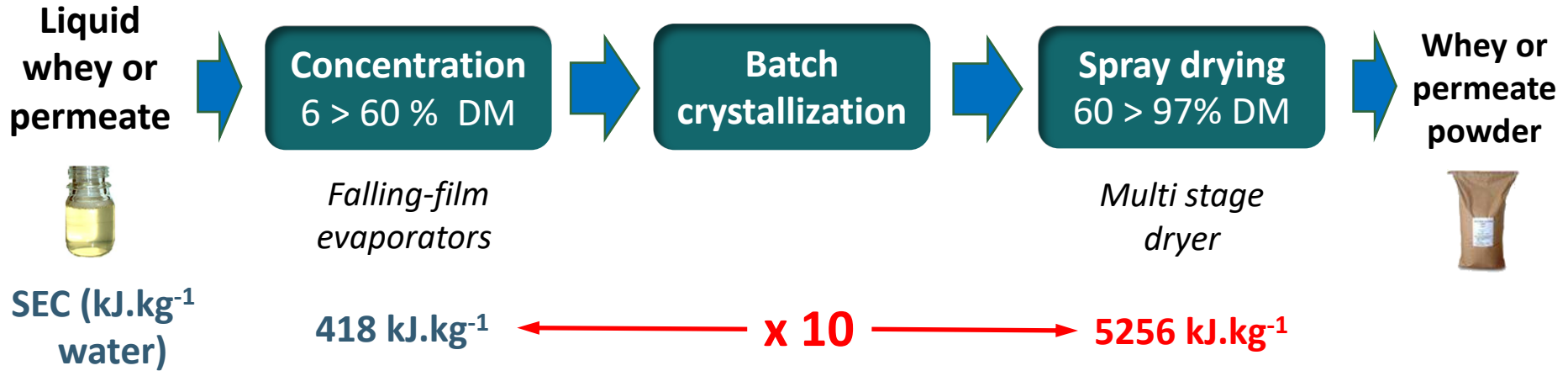
⇒ Patented process EP 15 306465.4 - 1357



3.


***Redesigning the process for the production of whey/permeate powders***

# Current bottlenecks in whey/permeate powder production

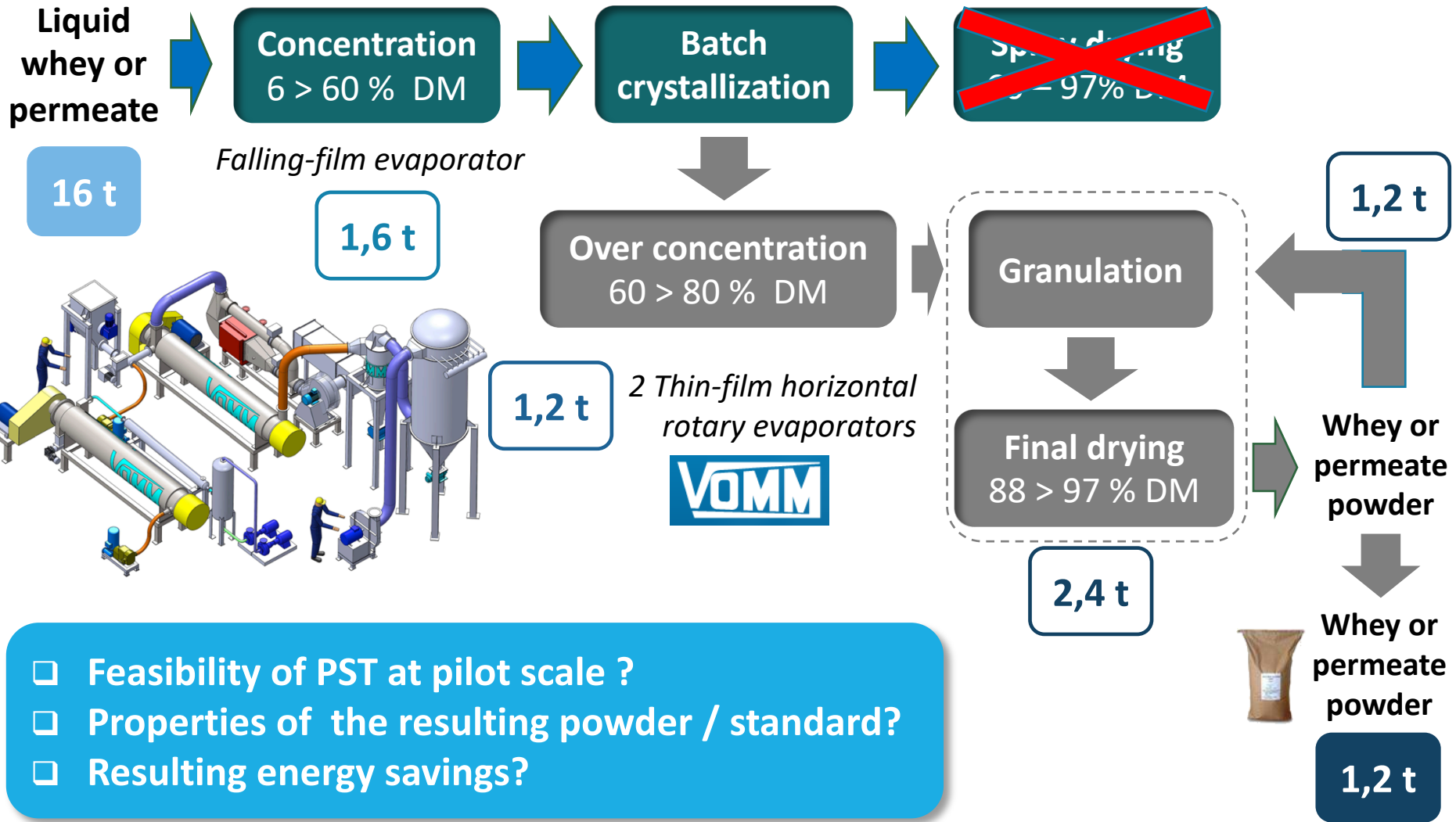


↗ concentrate DM, but limiting viscosity / pumping and spraying

55% of the overall energy for # 4% of the water removed

 Innovative process PST based on thin-film rotary evaporator where viscosity is controlled by a vigorous mechanical treatment that maintains a fluid flowing state at high DM

# PST: a new process for whey/permeate powder production

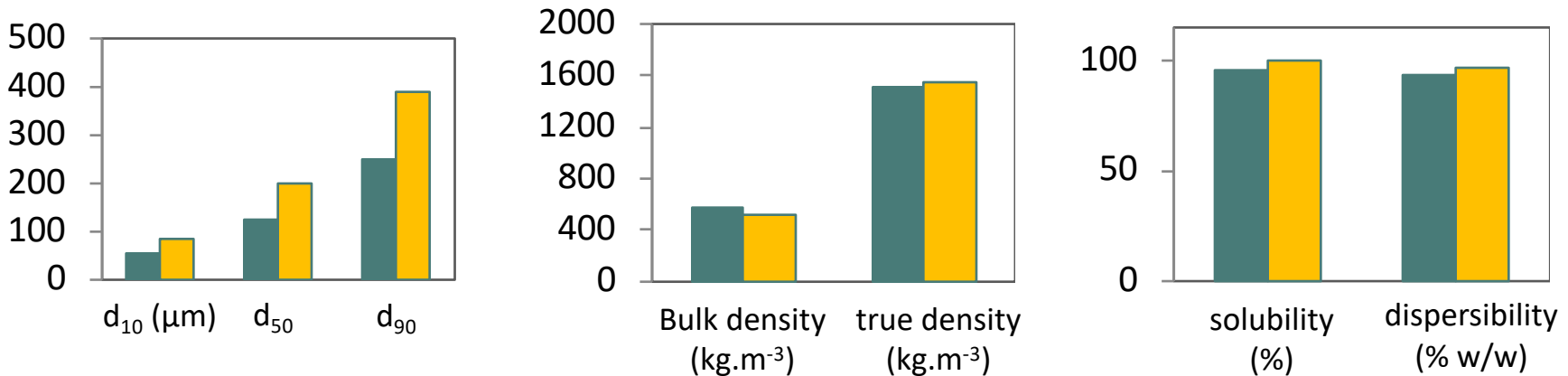


- Feasibility of PST at pilot scale ?
- Properties of the resulting powder / standard?
- Resulting energy savings?

# Similar powder characteristics, lower energy requirements

- ❑ PST process was run successfully for **long duration experiments > 6 hours**

- ❑ **Non significantly different features of PST powder**  **compared to control** 



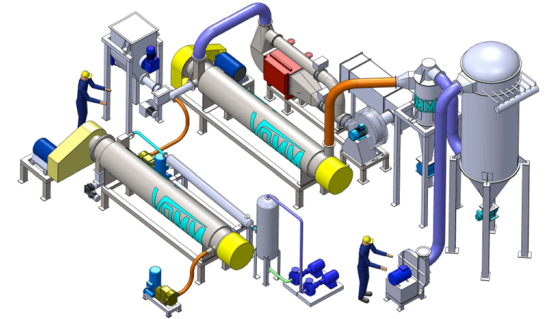
- ❑ **PST process significantly saves energy compared to the standard drying process**
  - ❑ Estimation of energy costs on the sole basis of the energy required for removal of water
  - ❑ **Drying step (60 to 97 % DM): 32% energy savings**
  - ❑ **Whole process (6 to 97 % DM): 11% energy savings**

*Innov Food Sci Emerging Technol* 41 (2017) 144 -149

# PST process in short

## VALIDATION

- Feasibility of PST process at pilot scale / long duration for the production of permeate powder
- Satisfactory properties of the resulting powder



## REDUCTION

- Energy savings estimation range from 10 to 30%
- Building savings estimated at 40%
- Water, CIP solutions and waste volumes should be cut as well

## INNOVATION

- Potential new products ...
- Produced in a sustainable way...
- Using breakthrough innovation

Patent EP 15752957.9 - 1358 (2015)

# Conclusion and perspectives

## Extensive work on IMF formulation is needed

- Determines nutritional properties
- **Conditions particle intrinsic features, then powder properties**
- **Enhances probiotics survival** during drying and in the dry state

## Research strategy to overcome industry challenges

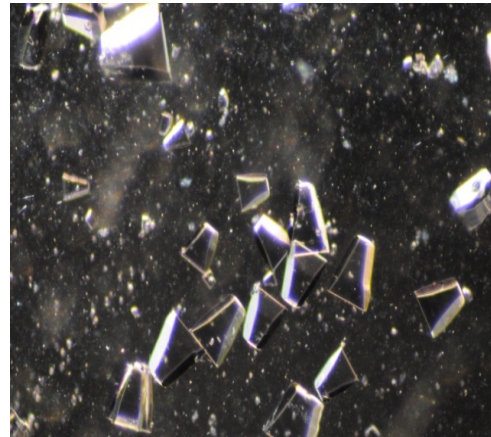
- Needs to **address growing market bottlenecks / societal issues**
- **Identifying the mechanisms at small scale, scale up needed**
- **Multidisciplinary approach** is mandatory: physics / biology / *medical*

## Next step in IMF research / development

- Understand the mechanisms governing the skin formation in colloid mixes
- Better control the end use properties while **considering real complex formula in single drop experiments**



***MERCI***



***THANK YOU FOR  
YOUR ATTENTION***