



## New insights on fouling mechanisms explored by rheo-fluidics at the microscale

Margot Grostete, Jeehyun Lee, Zanele Msibi, Françoise Boissel, Maude Jimenez, Romain Jeantet, Luca Lanotte

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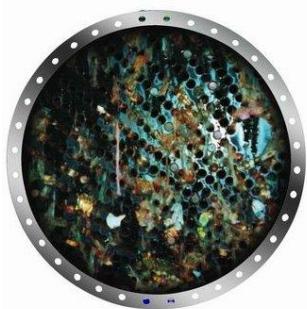
➤ New insights on fouling mechanisms explored  
by rheo-fluidics at the microscale

M. Grostete, J. Lee, Z. Msibi, F. Boissel, M. Jimenez, R. Jeantet, L. Lanotte



XVIII Italian Society of Rheology Conference  
Capri Island (Naples, Italy), 12th-14th September 2024

# > The fouling process in dairy industry



## What is fouling?

Accumulation of solid components  
of the dairy solutions  
on stainless-steel surface

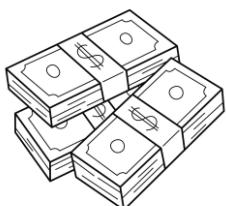


## Where do we observe fouling?

Membranes, filters  
(filtration process)

Evaporators, heat exchangers  
(pasteurization, sterilization)

DIFFERENT SCALE  
AND PHYSICO-CHEMICAL DYNAMICS



Daily cleaning sessions



## Which are the consequences?

- Significant pressure drop
- Lowered thermal exchange
- Pipe blockage



Visser J and Jeurnink Th J M (1997). Experimental Thermal and Fluid Science.

# > The emerging question of the evaporators



Concentration of dairy fluids before spray drying (energy saving)



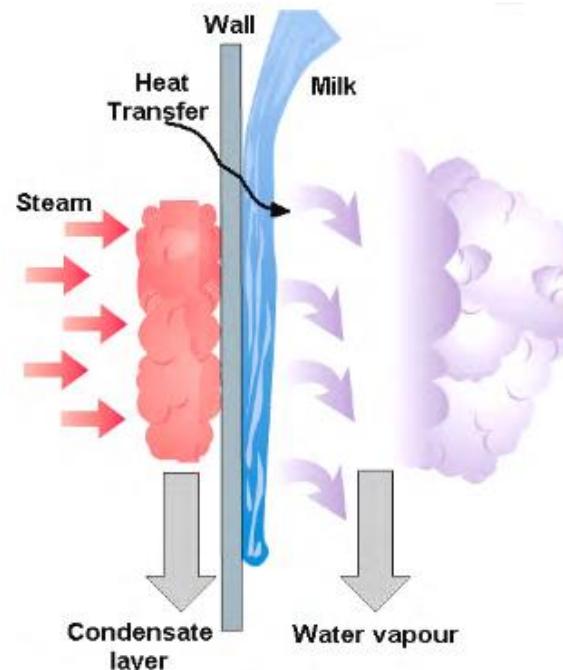
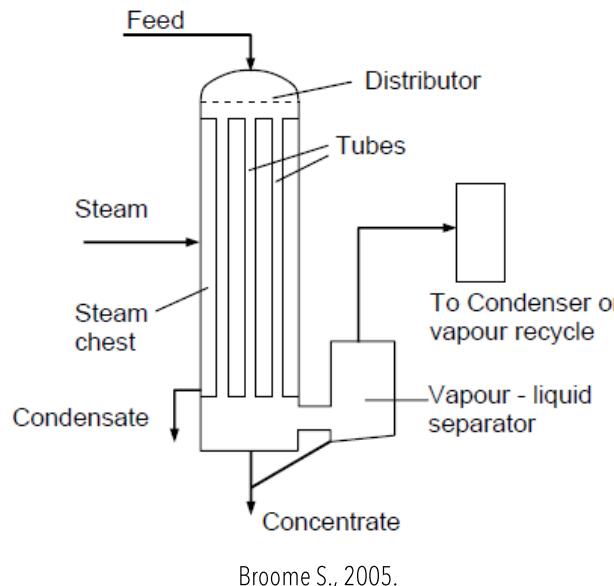
Spraying Systems Co.®

Production of "commodities"  
Wide range of processed fluids

Infant Milk Formulas (IMFs)



## FALLING FILM SETUP



## Main characteristics

- Constant surface temperature ( $T = 45-80^\circ\text{C}$ )
- Increasing top-to-bottom concentration
- Double interface

# State of the art on fouling mechanisms

No direct observation!

Bansal B and Chen X D (2006). Comprehensive Reviews in Food Science and Food Safety.  
Visser J and Jeurnink Th J M (1997). Experimental Thermal and Fluid Science.



## BULK SOLUTION

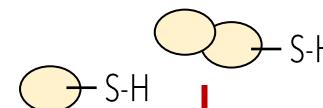
Milk, Protein Suspensions

Minerals ( $\text{Ca}^{2+}$ ) Whey Proteins (WP)



$T > 65^\circ\text{C}$

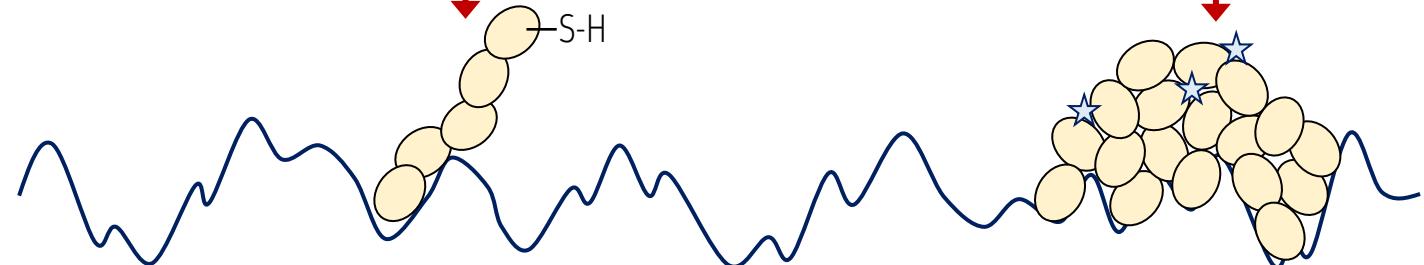
## PROTEIN DENATURATION



Active aggregates

Precipitates

Initiation Surface Reactions



## SURFACE PROPERTIES

Roughness, mechanical properties

Jimenez M, Delaplace G et al. (2013). Journal of Colloid and Interface Science



# > A microscopic journey: open questions



M. Grostete  
Ph.D. Thesis  
ENTEVAP Project (2022-25)



INRAe

Prof. G. Tomaiuolo

New insights on fouling mechanisms explored by microfluidics at the microscale  
12/09/2024 / L. Lanotte

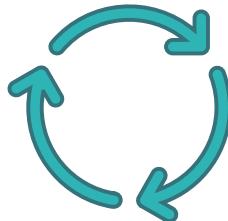
## MINIATURIZATION OF THE DAIRY EQUIPMENT CONDITIONS



### When?

Driving forces governing WP denaturation/accumulation

- Temperature
- protein concentration
- **shear rate**



### Where?

Microscopic dynamics at the **surface** and in the **bulk**  
Surficial deposit growth VS aggregate formation

### How?

Nature of **protein/surface interactions**

- Chemical adsorption
- Short-range attractions

## > Deposit formation: an alternative scenario



No fouling observed for  $T < 65^\circ\text{C}$

Jeurink Th J M, Walstra P, de Kruif C G (1996). Netherlands Milk and Dairy Journal.

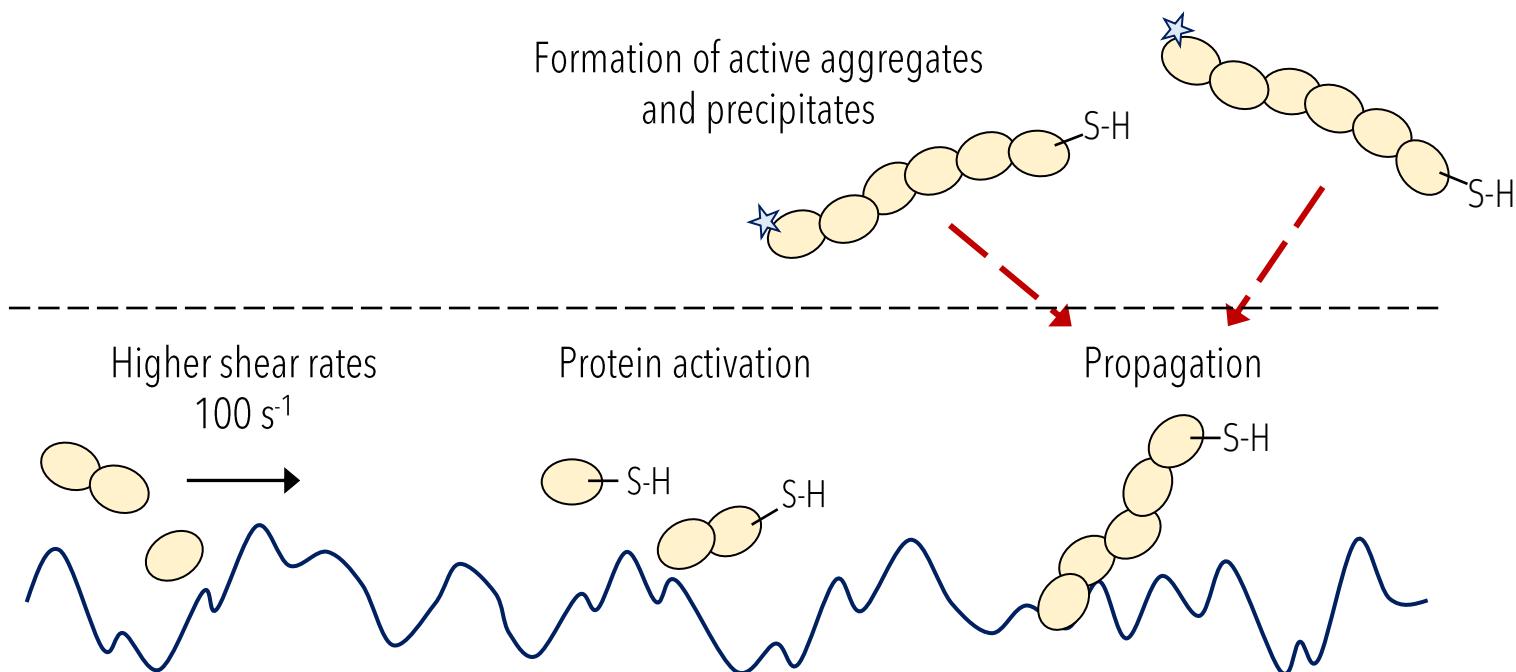
Absence of protein denaturation



Deposit development even at  $T=45^\circ\text{C}$  in evaporators



### DOUBLE IMPACT OF TEMPERATURE AND SHEAR RATE

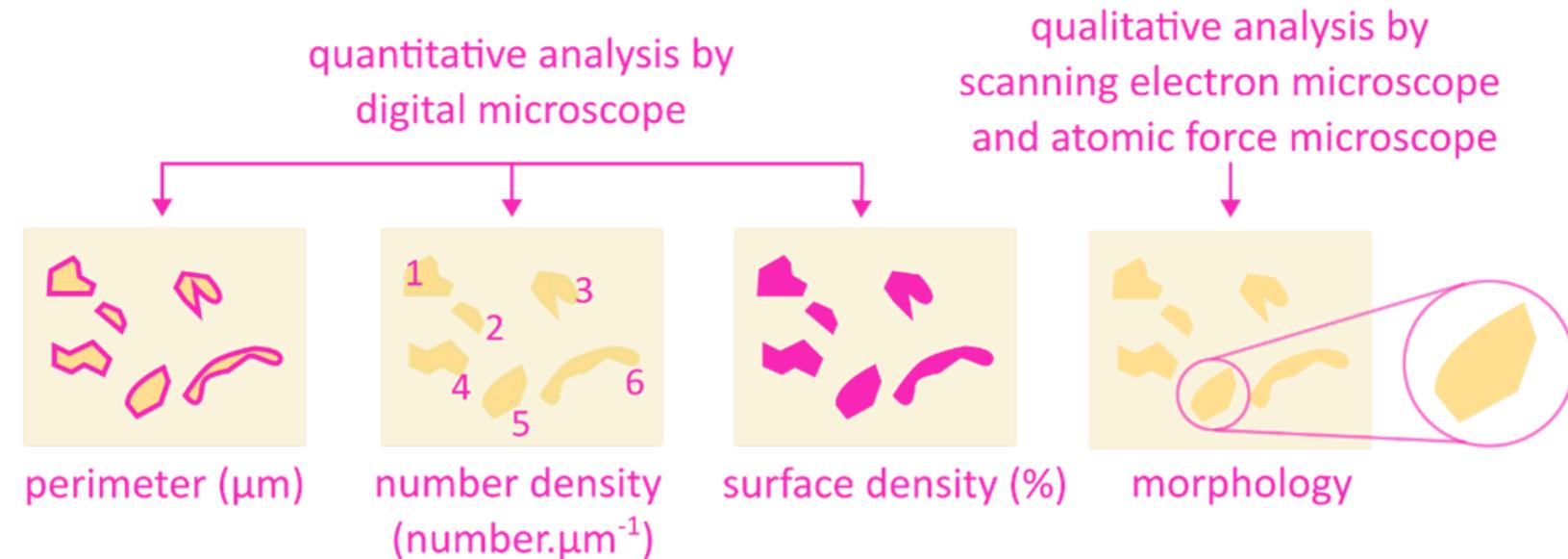
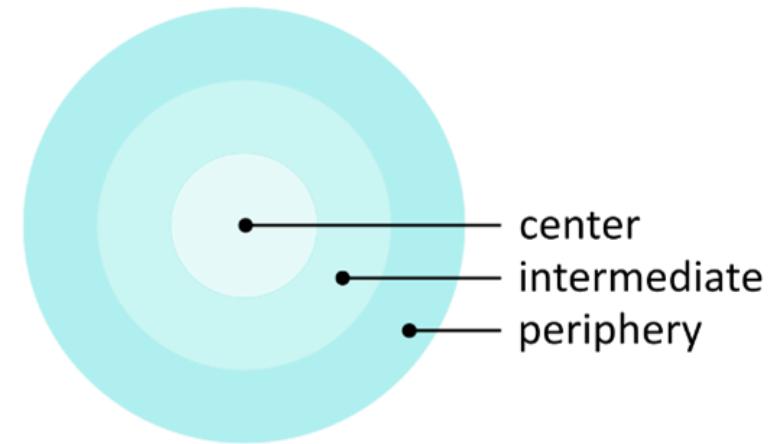
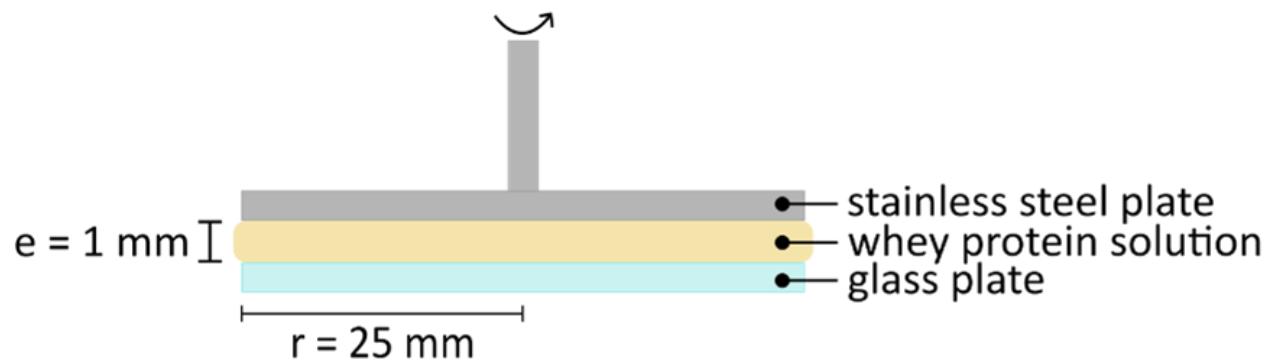


**Passive fouling  
PROPAGATION**

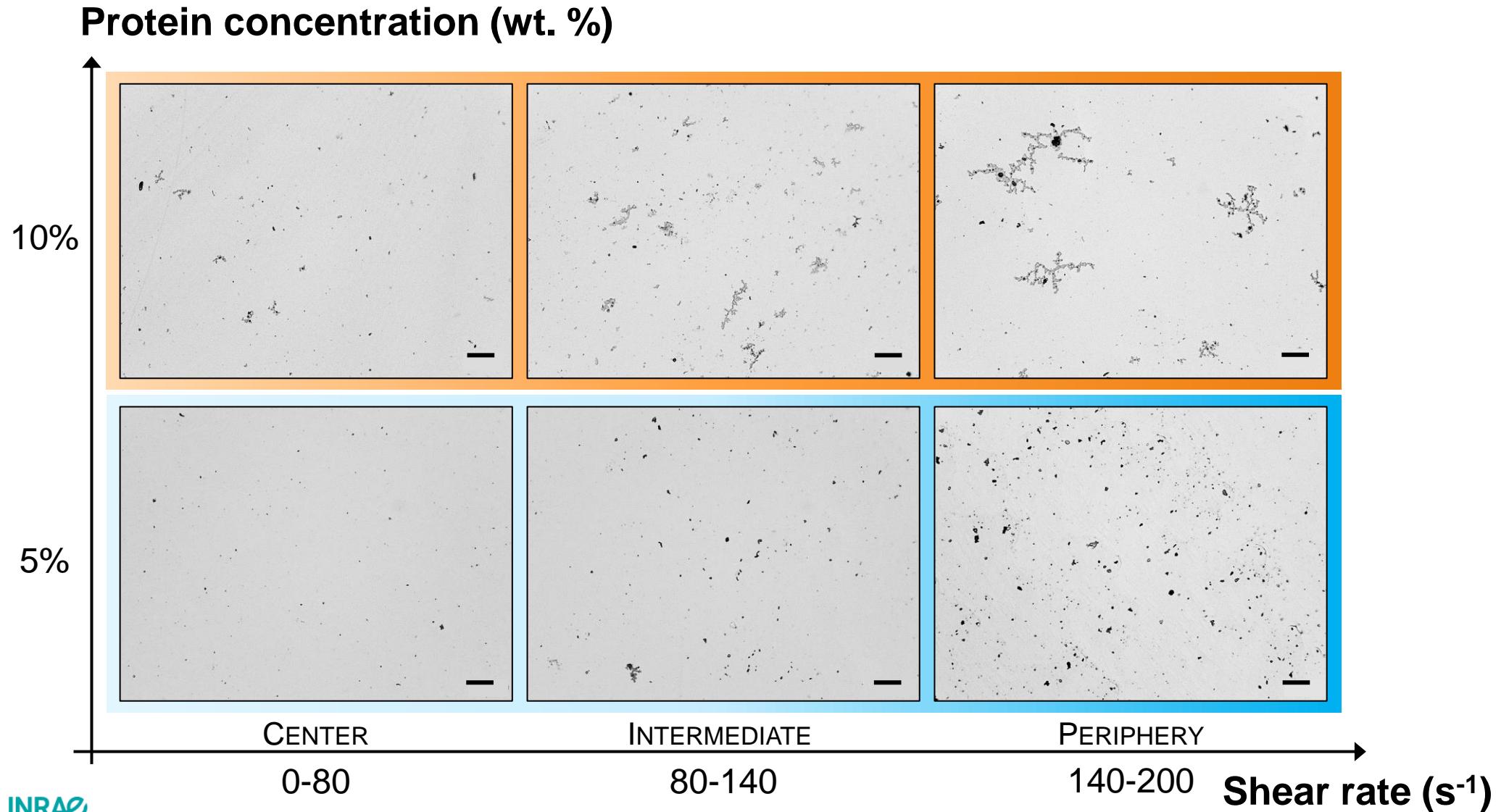
**Active fouling  
INITIATION**

INRAe

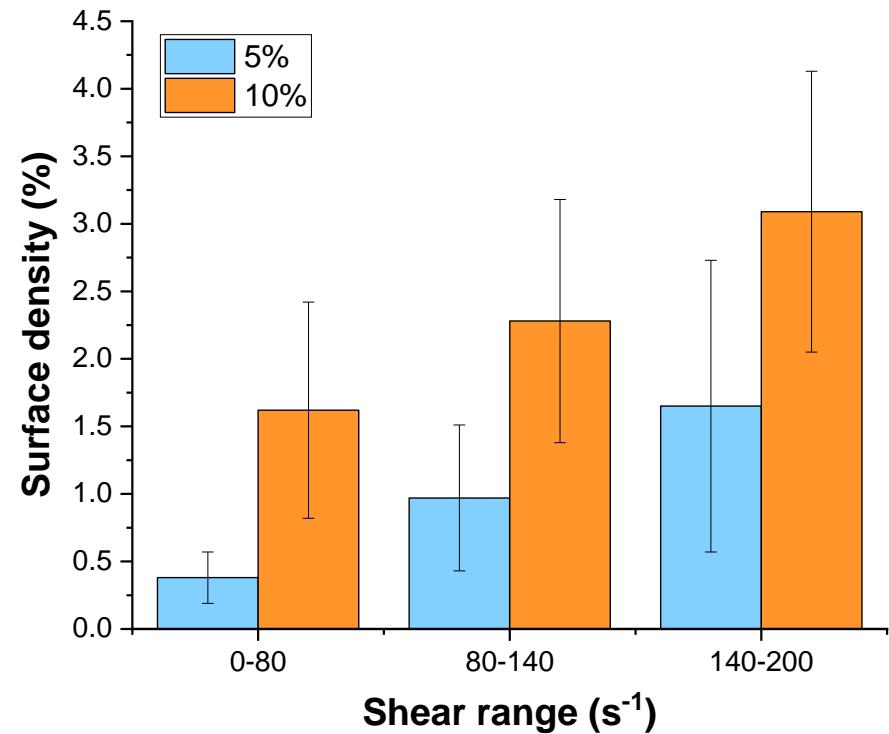
## > Rheofluidics: a simple and flexible strategy



## ➤ Qualitative observation of WP surficial deposits

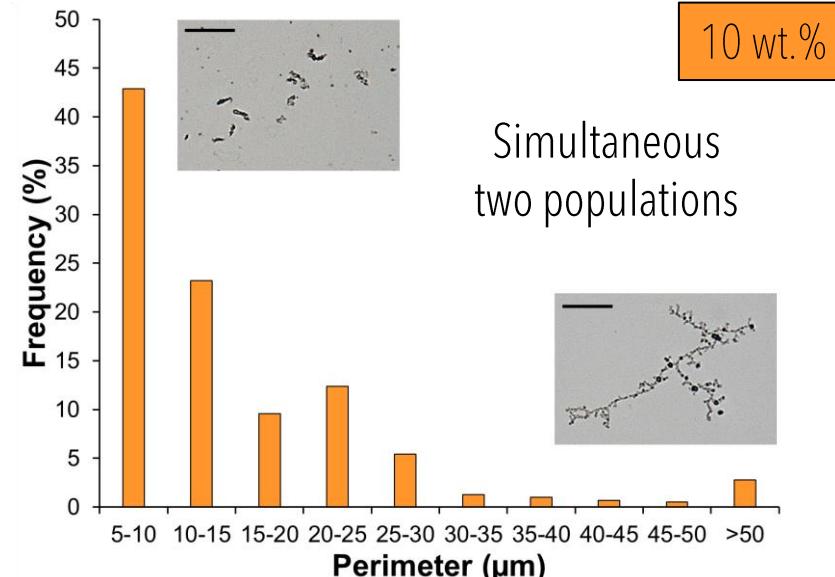
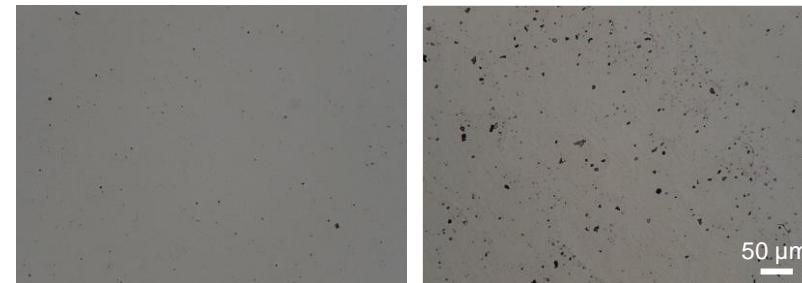


## Quantification of the shear impact



High standard deviations  
because of dynamics of  
protein accumulation

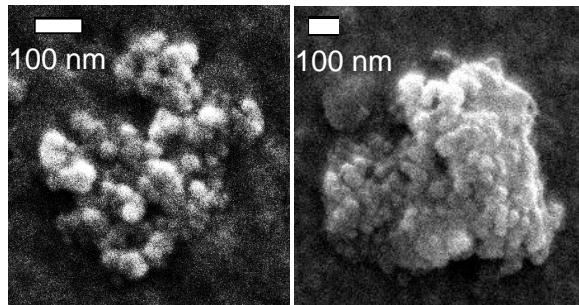
5 wt.%  
Non-homogeneous distribution  
on the surfaces



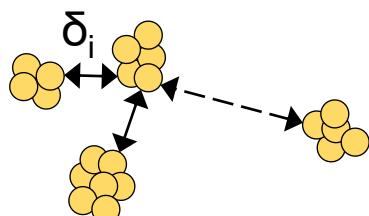
10 wt.%  
Simultaneous  
two populations

Grostete et al. (2024), Int J of Biological Macromolecules.

# > Hypothesis on the stages of protein accumulation

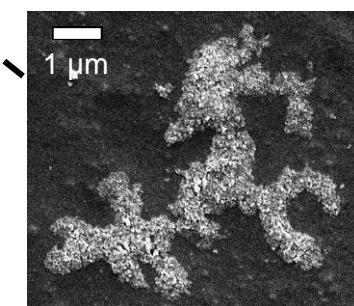
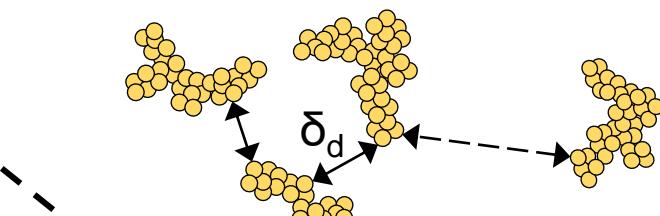


INITIATION POINTS



- Thermal denaturation and adsorption ✓
- Role of the shear stress ✗

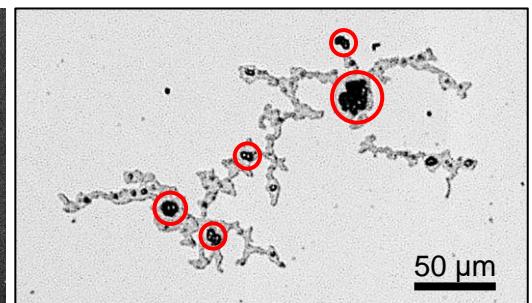
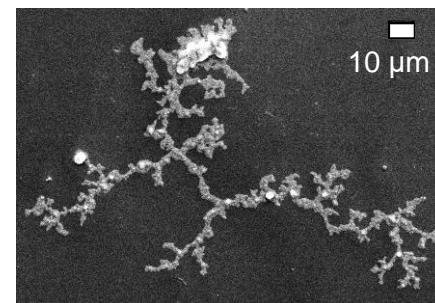
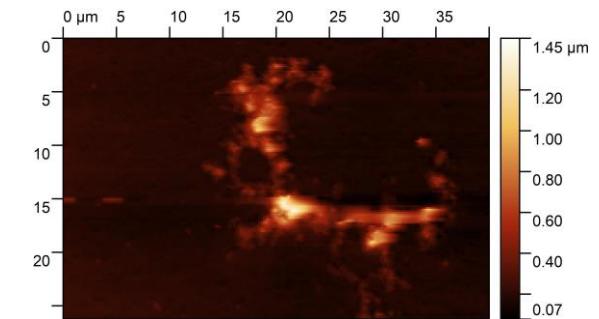
TIME AND DISTANCE ARE CRUCIAL PARAMETERS



PRIMARY DEPOSITS

- Interconnection of primary deposits
- Structure complexification

PHYSICO-CHEMICAL NATURE  
OF THE DEPOSITS



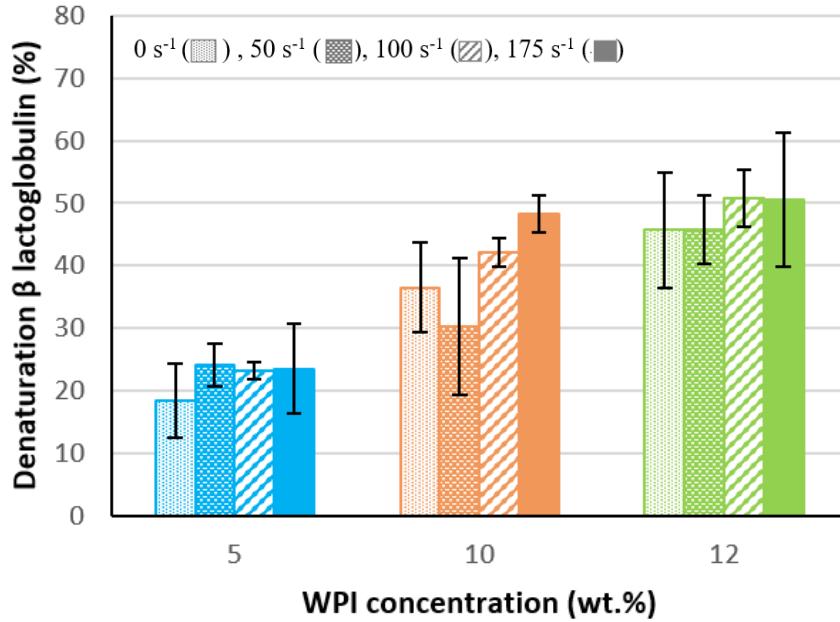
BRANCHED STRUCTURES

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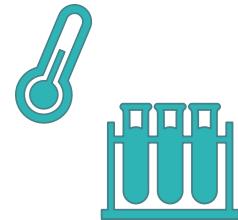
# What happens in the “bulk”?

## Tests by con-and-plate rheometers

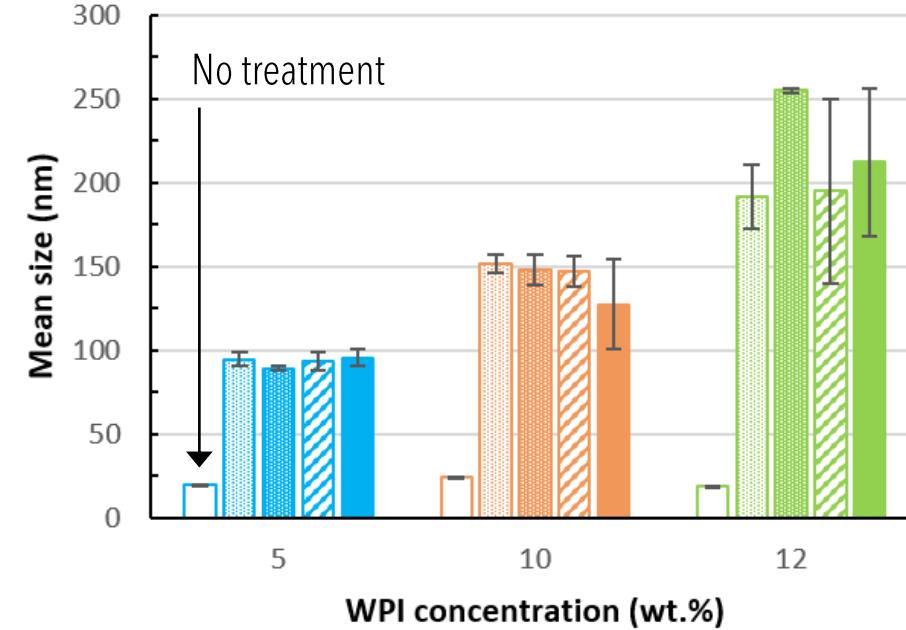
Grostete et al., JCIS, close to submission.



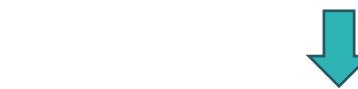
Denaturation in the bulk governed by thermal effect and protein concentration



Impact on protein-protein interactions and WP aggregate formation



Submicronic WP aggregates predominant in the solutions (confirmed by microscopy observations)



**ENHANCED DEPOSIT PROPAGATION  
DOES IT EXCLUDE SHEAR-INDUCED DENATURATION?**

## > Conclusions

### Surface

- Increasing shear stresses enhance deposit formation in terms of number (dilute conditions) and shape complexification (concentrate conditions)
- Evident effect of the shear when combined with thermal one
- Deposit formation possibly due to both chemical and physical interactions next the surface

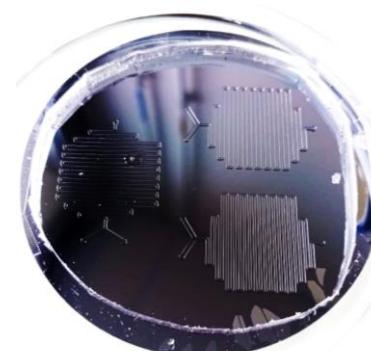
### Bulk

- No impact of the shear on protein denaturation and aggregate mean size
- Combined effect of temperature and protein concentration

## > What's next?

**Direct observation of the phenomenon**

Microfluidics



## ➤ Preliminary results and exponential difficulties

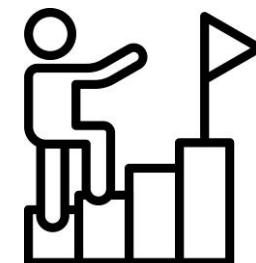
Visit the poster of Margot



### Experimental challenges

- *Repeatability* of the experiments  
(defects, bubble formation)
- *Control* of the temperature  
(device and solution)
- *Estimation* of the shear  
(rheometer vs wall shear)

- DEEPENED INVESTIGATION LEADS TO MORE COMPLEX QUESTIONS**
- Shear intensity and shearing time
  - Absolute temperature and  $\Delta T$
  - Air-liquid interfaces



# Grazie a tutti per la vostra attenzione Please, bring me back home!



Risultati per **Rennes, Francia**

