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## Microscopic investigation of fouling mechanisms in dairy protein mixes under shear

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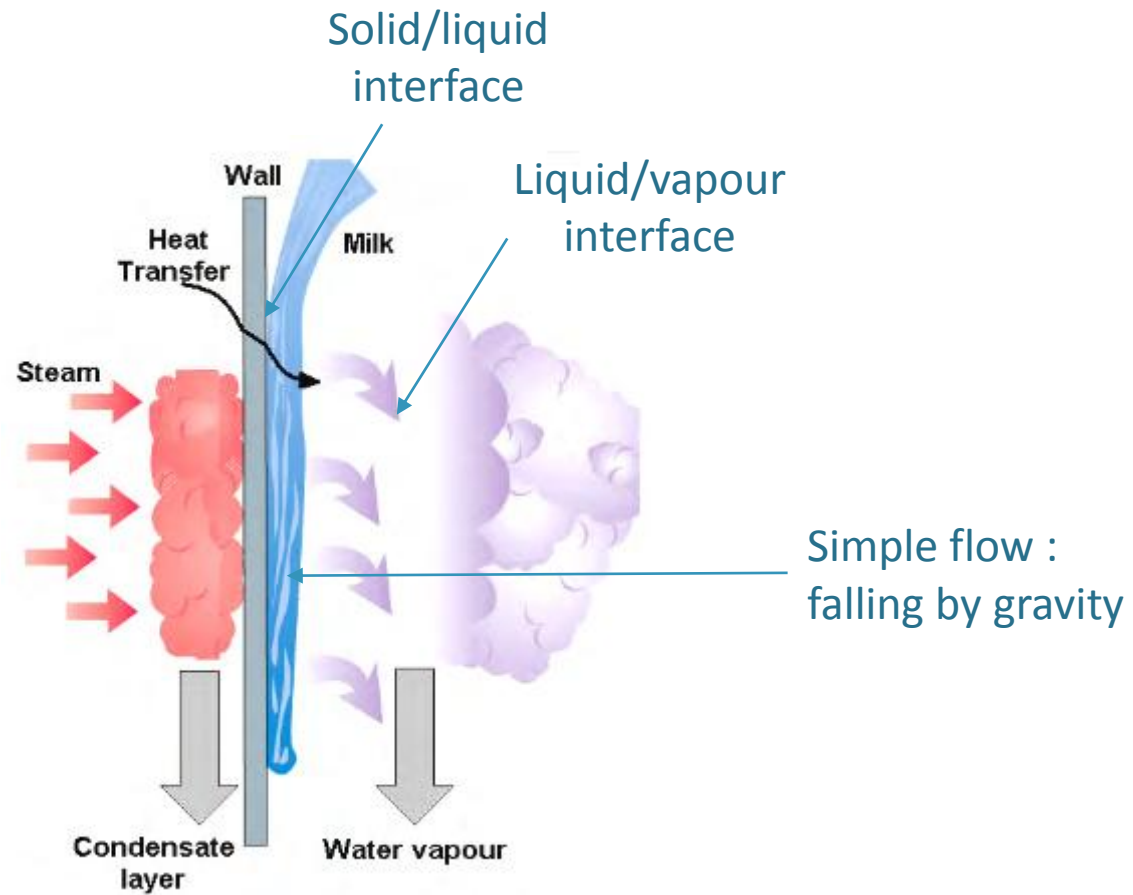
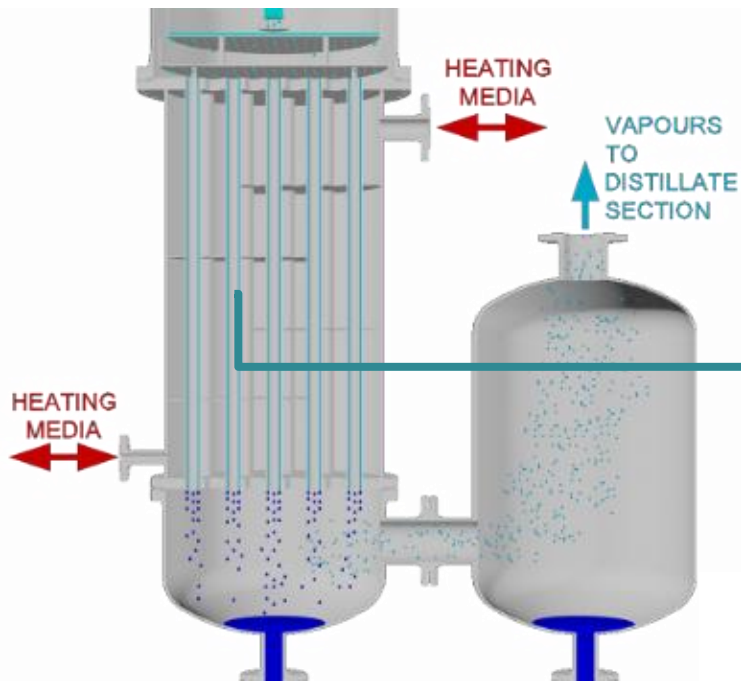
# Microscopic investigation of fouling mechanisms in dairy protein mixes under shear

Margot Grostete, Romain Jeantet, Jeehyun Lee, Maude Jimenez, Luca Lanotte

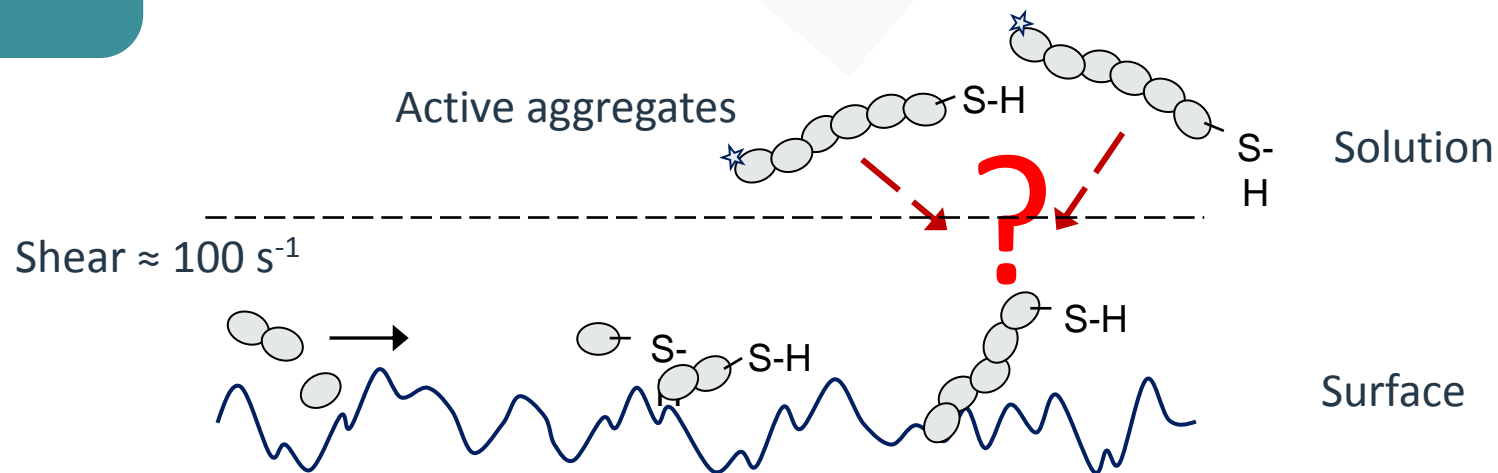
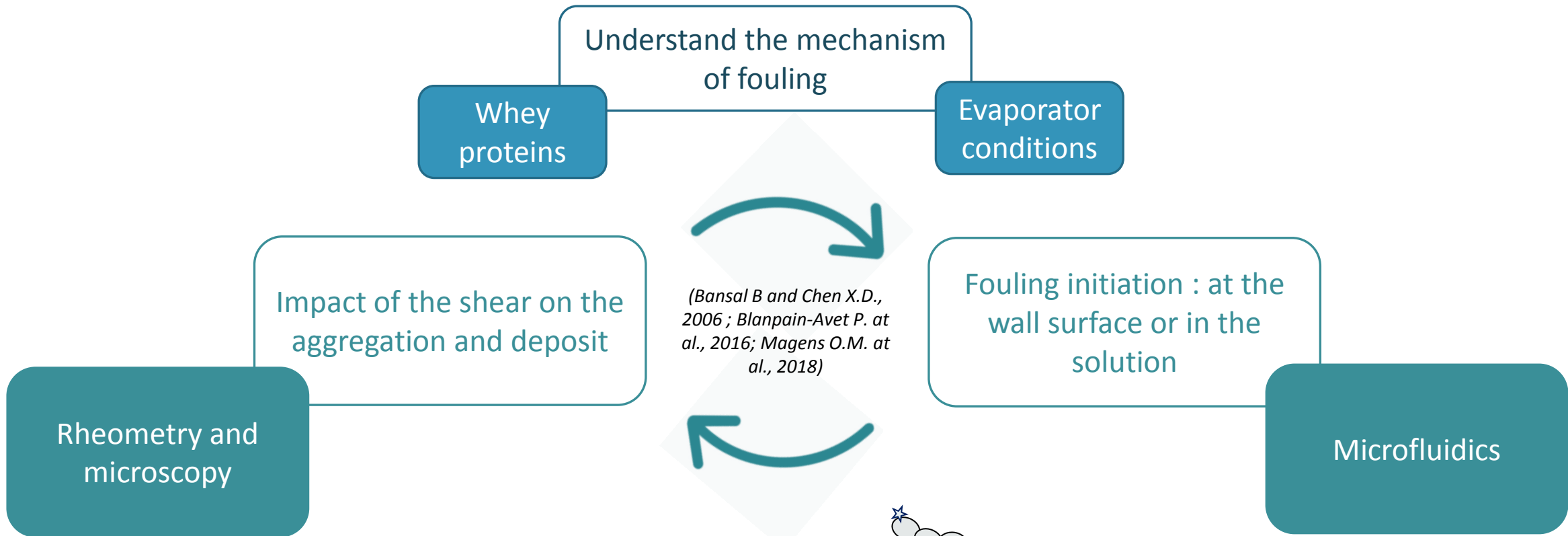


01/08/2023

## Falling film evaporator



(Broome S., 2005)



Reference	Type	Geometry	Shear rate / rotation speed
Taylor, freyer, 1994	Rheology	Cone/plan	0 - 40 s <sup>-1</sup>
Simmons et al., 2007		Couette	111 - 625 s <sup>-1</sup>
Samy Gaaloul et al., 2009		Brookfield, cylinder	28 s <sup>-1</sup>
Erabit et al., 2014		Couette	0 - 400 s <sup>-1</sup>
Mediwathe et al., 2018		Bob/cup	0 - 1000 s <sup>-1</sup>
Quevado et al., 2020-2021		Close cavity CCR	0.06 - 50 s <sup>-1</sup>
Bogathawa et al., 2020		Cylinder	1000 s <sup>-1</sup>
Bogathawa et al., 2021		Bob/cup	1000 s <sup>-1</sup>
Wolz et al., 2016		Mooney/Erwart	100 - 1452 s <sup>-1</sup>
Moakes et al., 2015		Cylinder	200 - 800 s <sup>-1</sup>
Byrne et al., 2002		Stirrer	100 - 1639 s <sup>-1</sup>
Santos et al., 2006		Flow cell	135 ; 205 and 157 ; 238 s <sup>-1</sup>
Kerche et al., 2016		Tubular exchanger	/
Zhang et al., 2019		Heat exchanger condition	Spinning disc apparatus
Clarkson et al., 1999	Bubble column apparatus		/
Walkenstrom et al., 1999	Spinning disc apparatus		100, 500, 900 or 1300 RPM
Koh et al., 2014	Ultraturax Stainless tubular container		17500 min <sup>-1</sup> 1000 min <sup>-1</sup>
Vilotte et al., 2021	Microfluidics	Continuous small scale millifluidics	32 - 2666 s <sup>-1</sup>

### Current literature

- Focus only on the solution
- Heat exchangers : conditions are different in evaporators
- High temperature (> 80°C) predominant effect ?
- Various methodologies and shear ranges

## Literature : Effect of shear on the aggregation

Few articles hypothesize on the shearing effect:

- Increase aggregates at  $[C_{\text{protein}}] < 10 \text{ wt } \%$
- Decrease aggregates at  $[C_{\text{protein}}] > 10 \text{ wt } \%$

*Santos et al. (2006)*

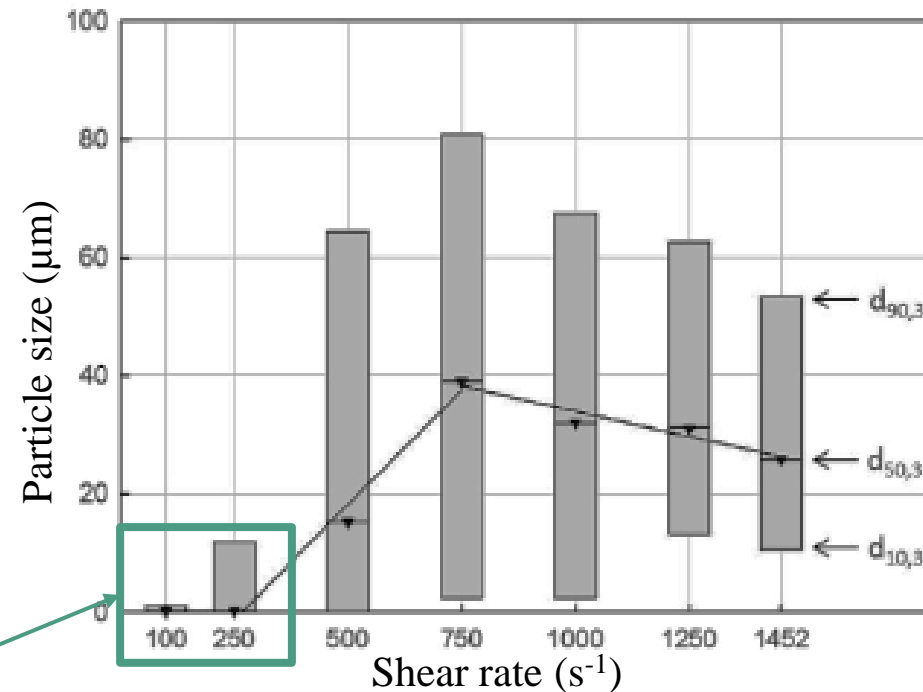
*Simmons et al. (2007)*

*Moakes et al. (2015)*

*Erabit et al. (2014)*

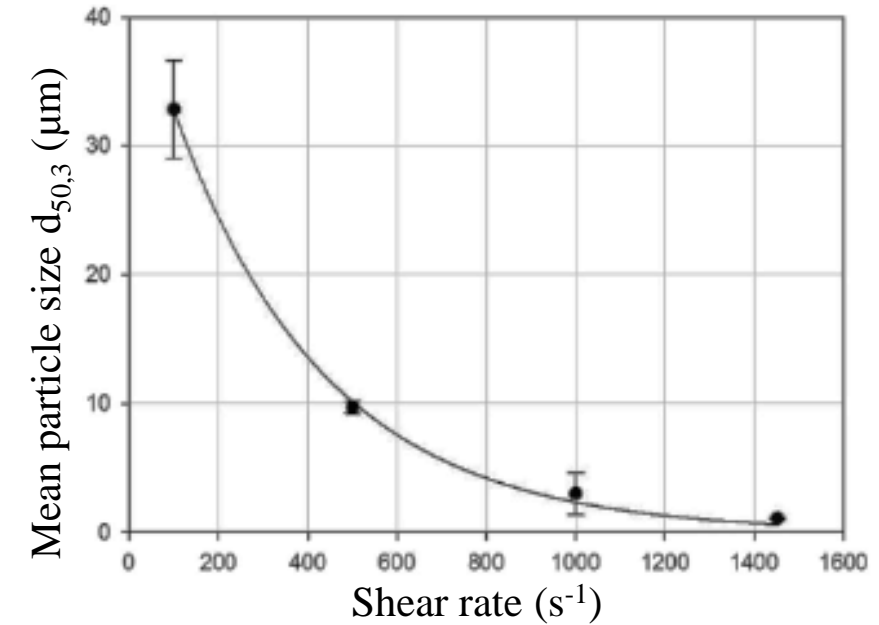
*Wolz et al. (2016)*

*Bogahawatha et al. (2020)*



Shear working range  
for the evaporator

Influence of shear rate on the particle size of whey protein solutions  
with  $C_{\text{protein}} = 5 \text{ wt } \%$  heated at  $80 \text{ } ^\circ\text{C}$  for 10min with a degree of  
denaturation  $> 95 \text{ } \%$



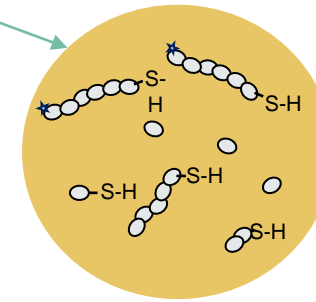
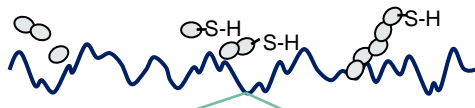
Influence of shear rate on the mean particle size  $d_{50,3}$  for a protein  
concentration of  $30 \text{ wt } \%$  heated  $80 \text{ } ^\circ\text{C}$  for 10 s with a degree of  
denaturation of  $> 95 \text{ } \%$

*(M. Wolz et al., 2016)*

Rheometry and  
microscopy

Surface

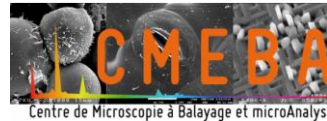
Bulk



## Digital Microscopy

- Large aggregates (> 10  $\mu\text{m}$ )
- Quantitative analysis

## SEM



- All aggregates (< 1  $\mu\text{m}$  to > 50  $\mu\text{m}$ )
- Qualitative analysis

What we want explore : shear rate between 0 to  $200 \text{ s}^{-1}$   
 What we know :

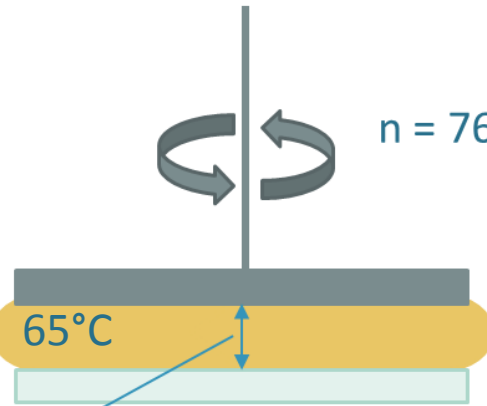
- Diameter geometry = 5 cm
- Shear rate limit =  $200 \text{ s}^{-1}$
- Parallel plate : shear gradient on the surface



$[C_{WPI}]$

5 to 20 wt %

Parallel plates  
(PP)

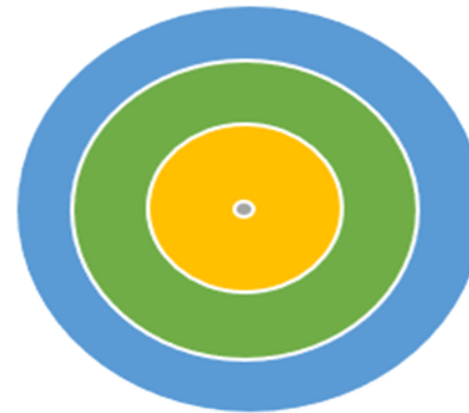


$n = 76,4 \text{ RPM}$

$e = 1\text{mm}$

10 min

Shear rate for each diameter :



$140 \text{ à } 200 \text{ s}^{-1}$  (Periphery)

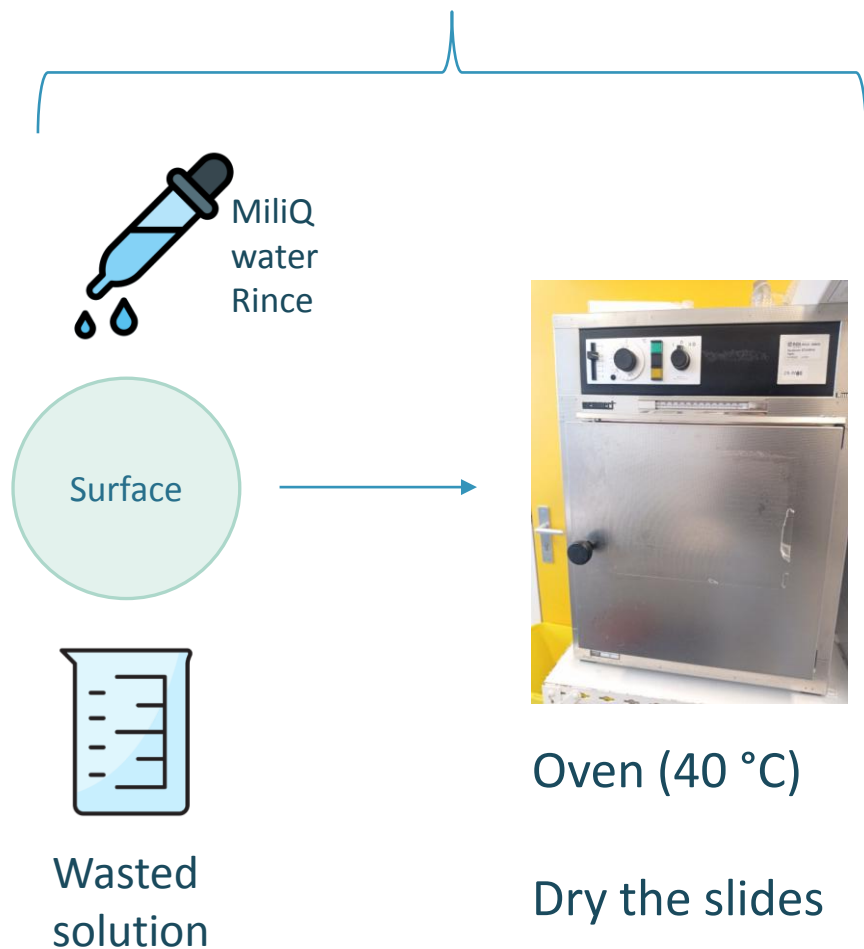
$80 \text{ à } 140 \text{ s}^{-1}$  (Intermediary)

$0 \text{ à } 80 \text{ s}^{-1}$  (Centre)

$0 \text{ s}^{-1}$



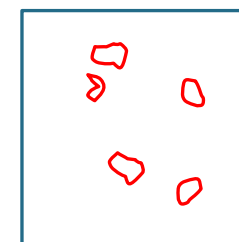
**Objective:** keep only the aggregates attached to the surface



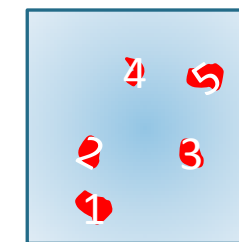
30 pictures/region

Digital microscope (Keyence, VHX 7000)

Keyence software

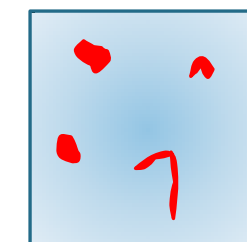


Perimeter ( $\mu\text{m}$ )



Density (by number)

$$\frac{\sum \text{Aggregates}}{\text{Picture area}}$$



Area ratio (%)

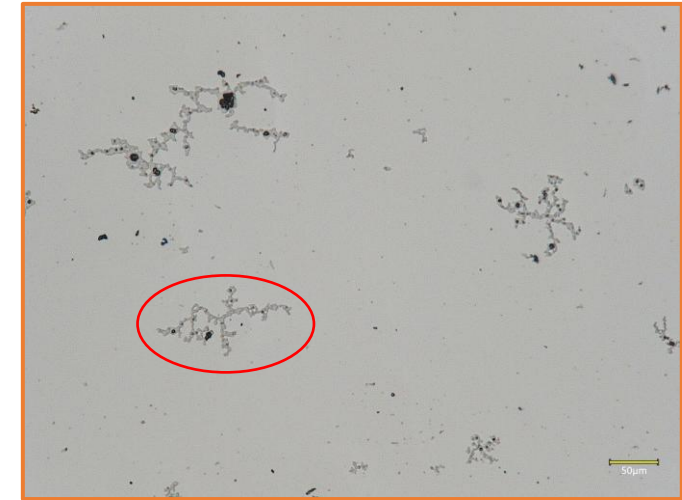
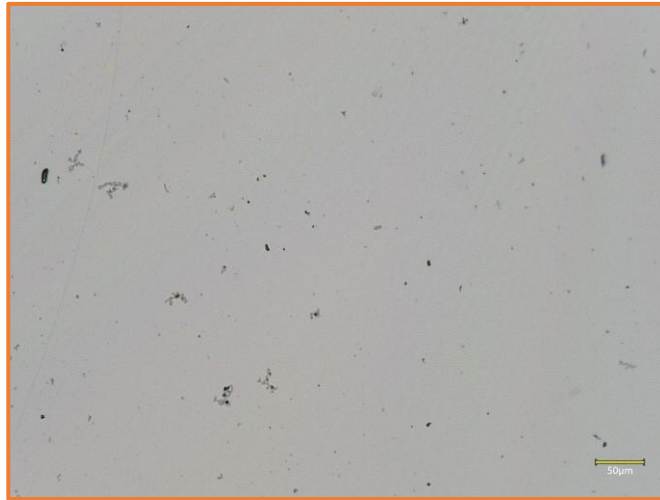
$$\frac{\sum \text{Aggregates Area}}{\text{Picture Area}}$$



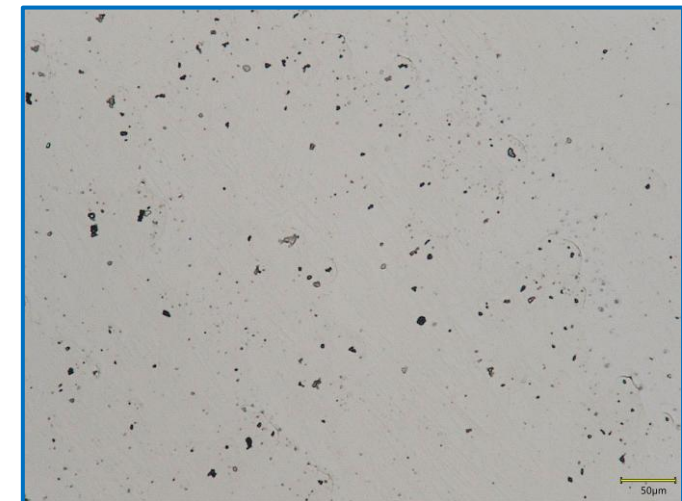
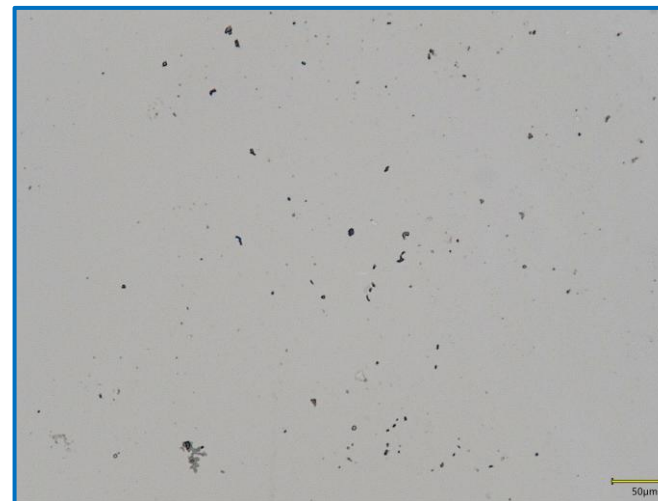
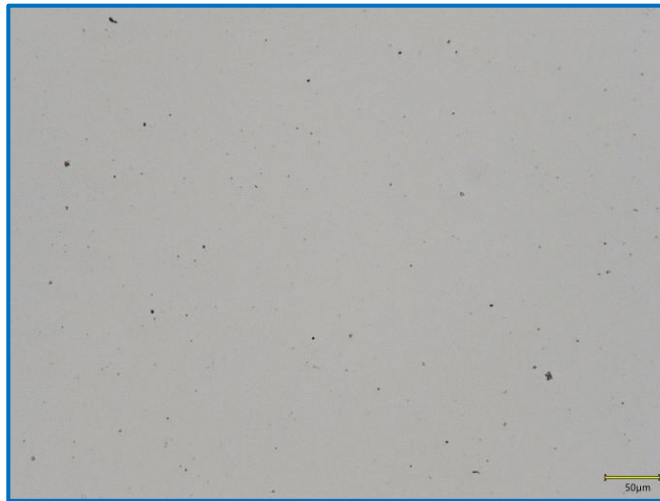
Morphological  
characterisation  
Frequency occurrence

## Microscopy General observation (G\*500) - SURFACE

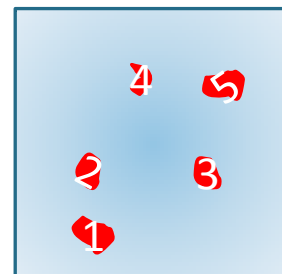
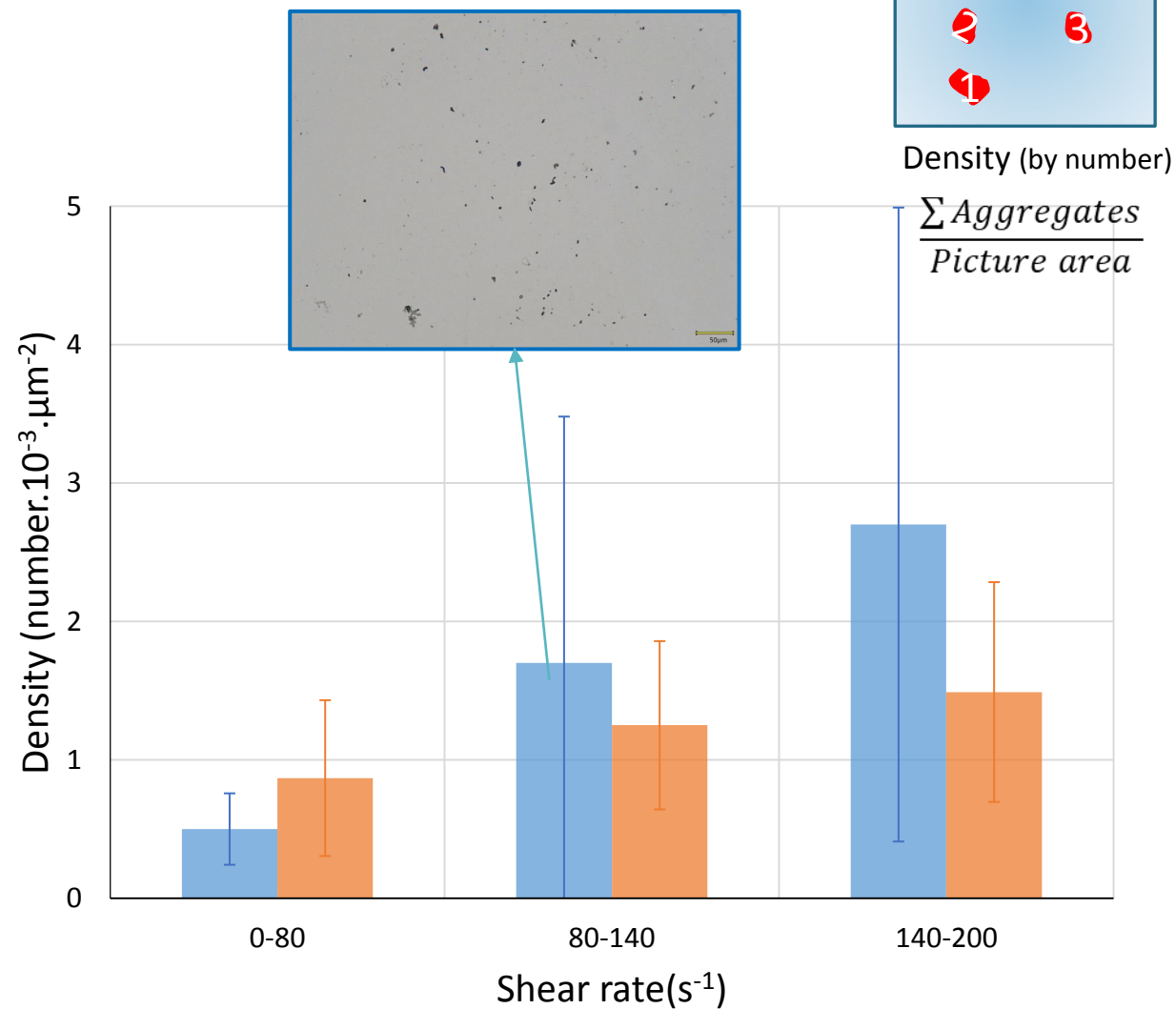
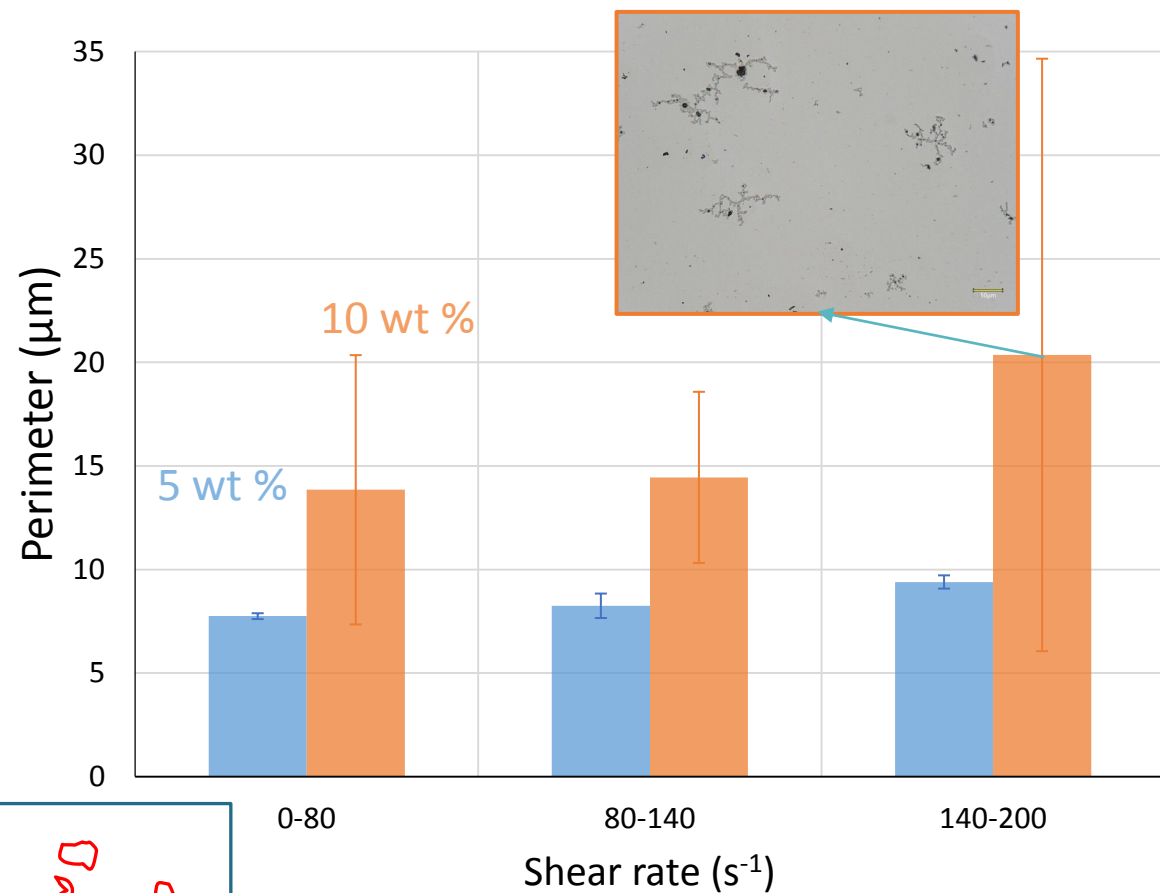
10 wt % WPI



5 wt % WPI

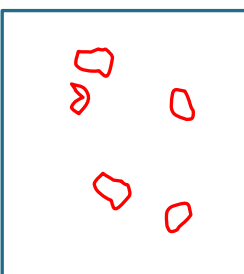
0-80 s<sup>-1</sup>80-140 s<sup>-1</sup>140-200 s<sup>-1</sup>

## Aggregates analyses by Keyence software- SURFACE

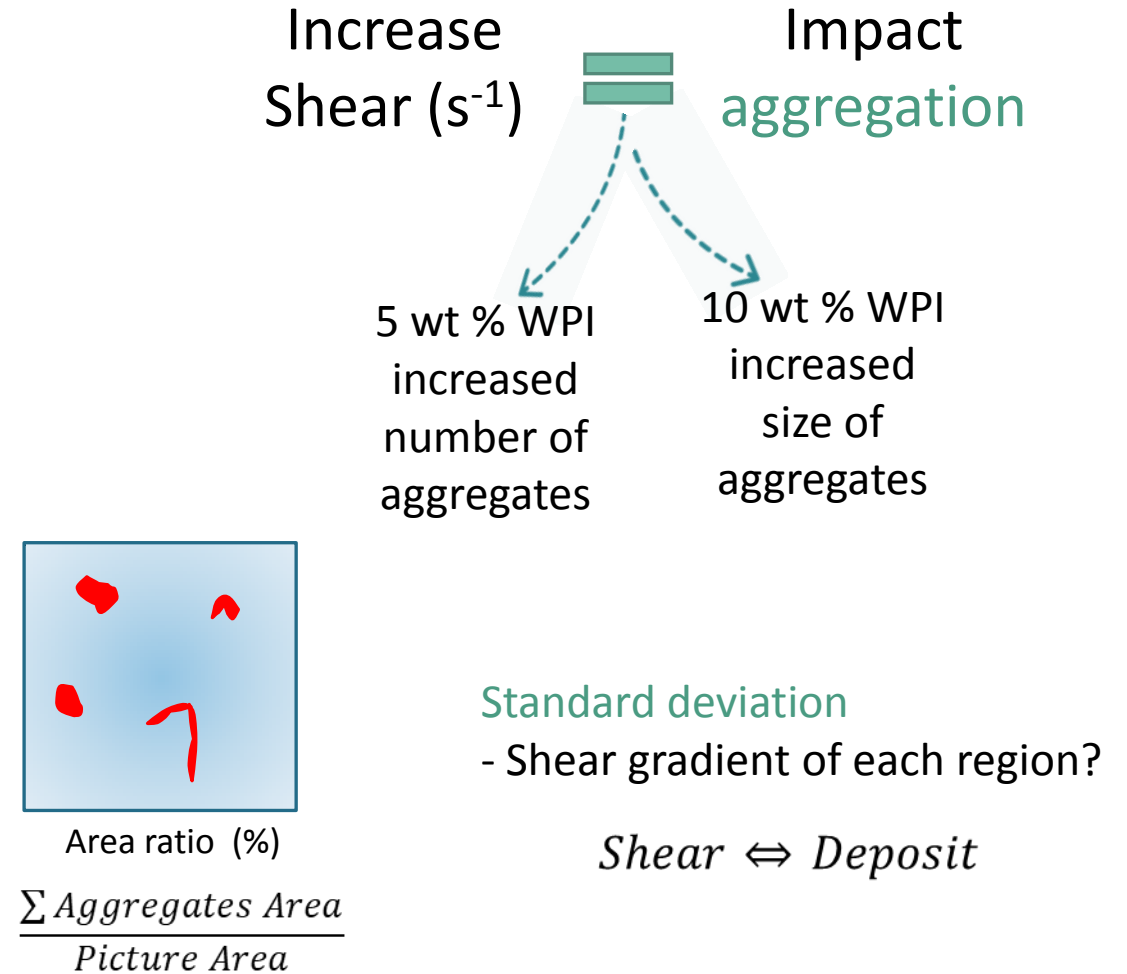
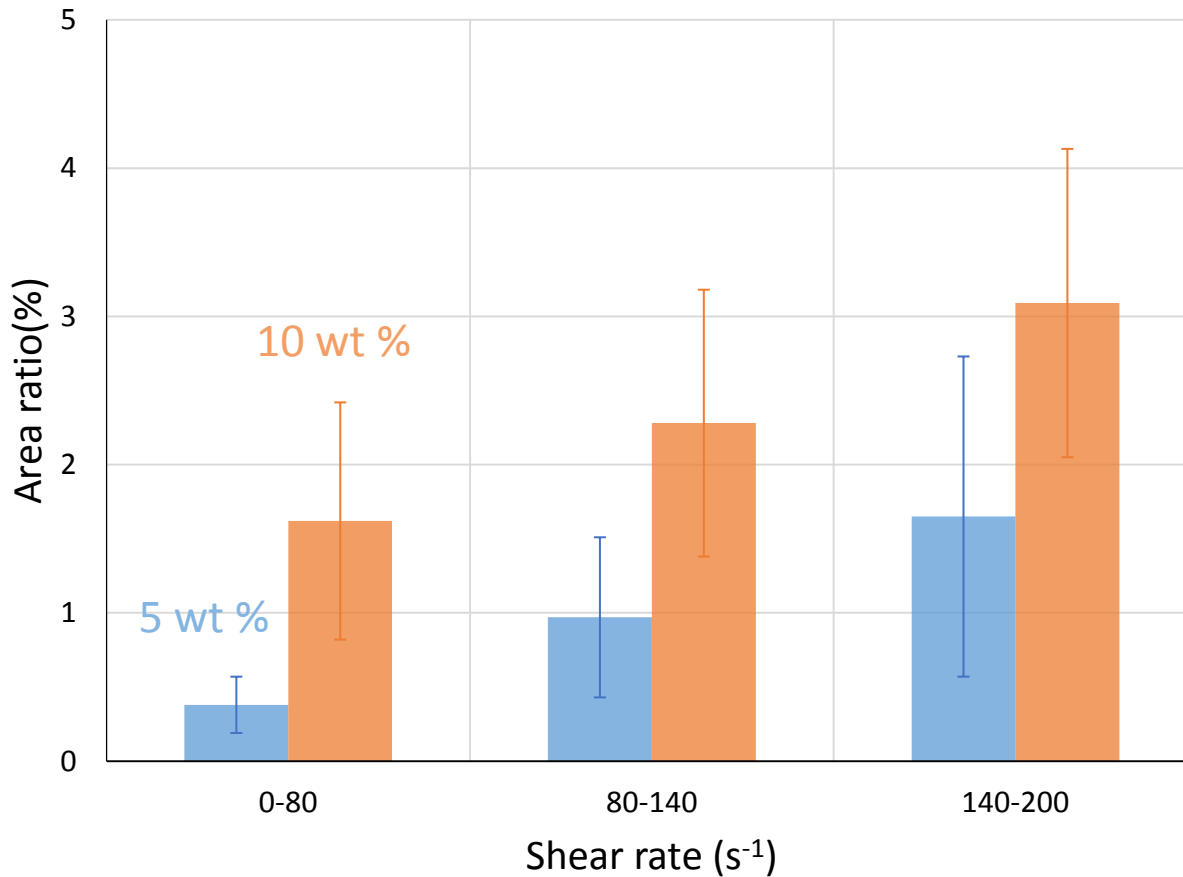


Density (by number)

$$\frac{\sum \text{Aggregates}}{\text{Picture area}}$$

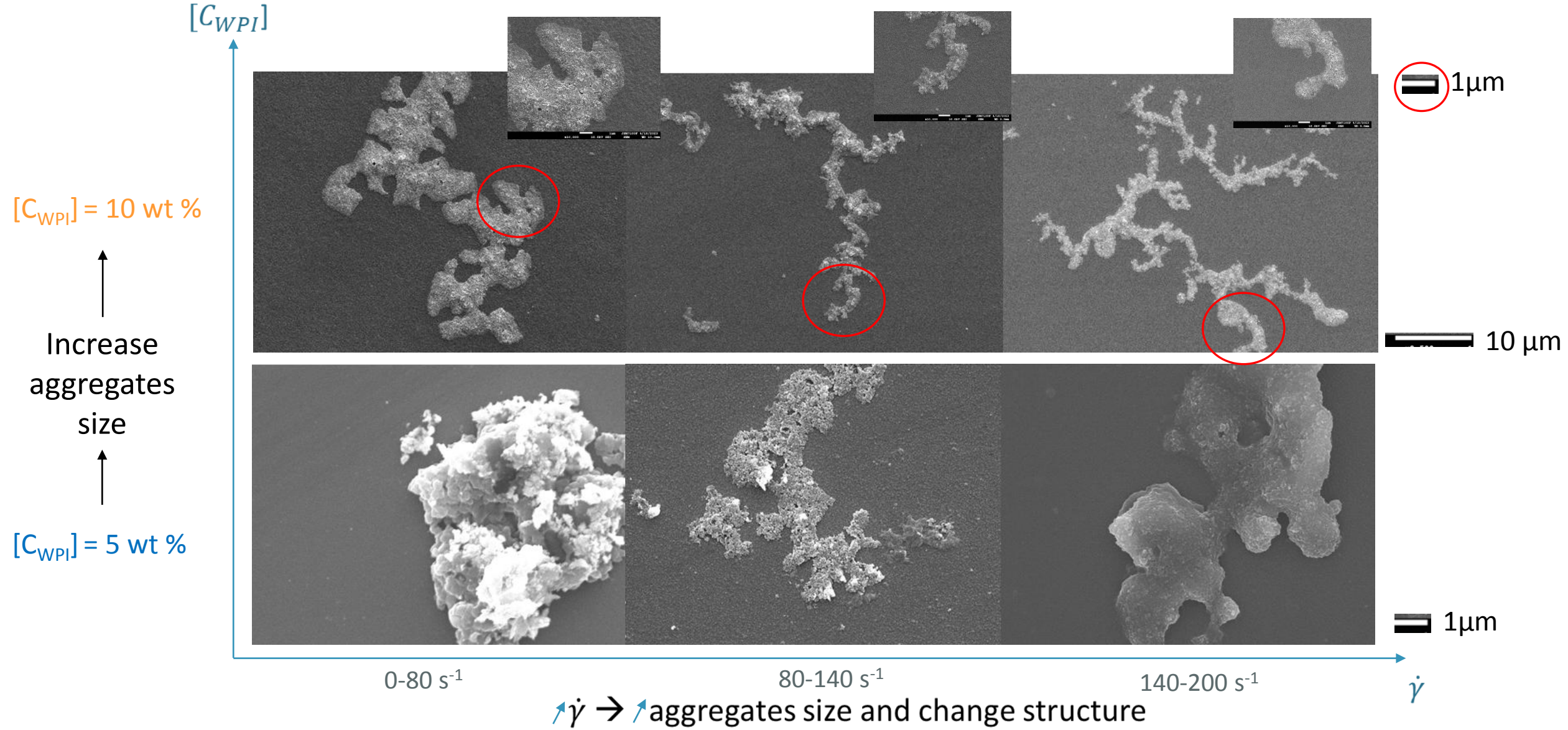
Perimeter ( $\mu\text{m}$ )

## Aggregates analysis by Keyence software- SURFACE





# SEM 5 wt % and 10 wt % WPI

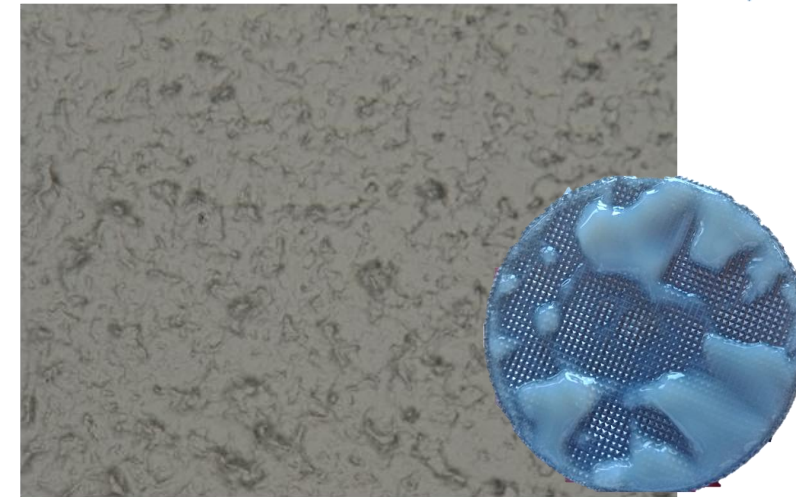
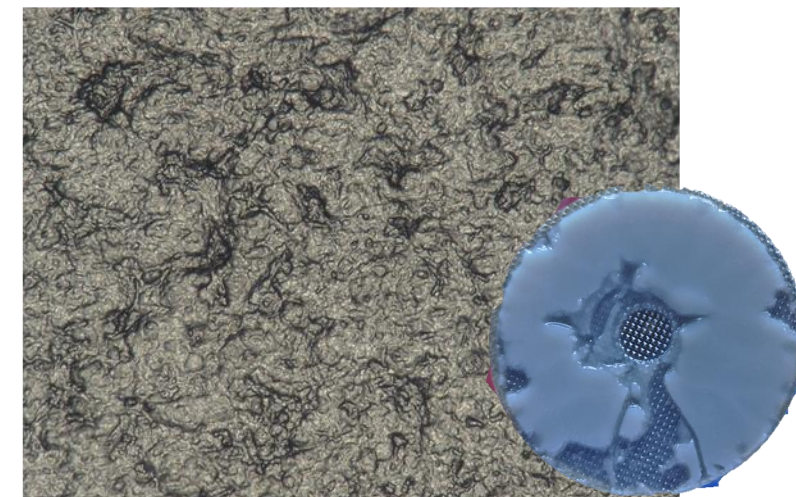


## Microscopy of 15 wt % and 20 wt % WPI solution

2min

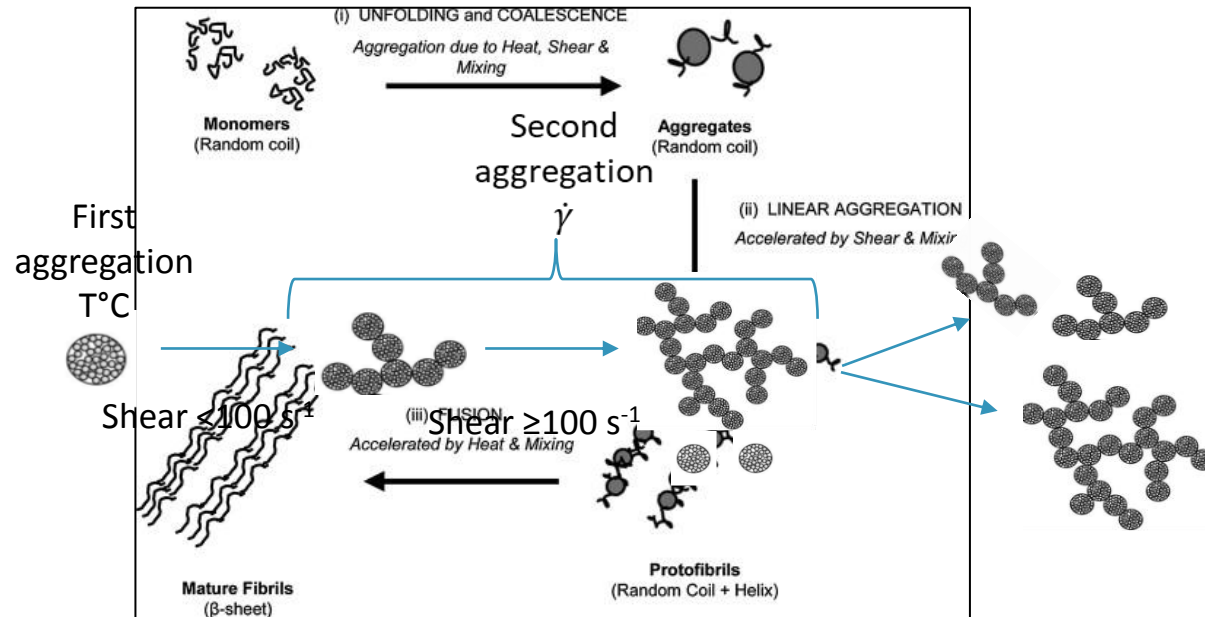
5 min

10min

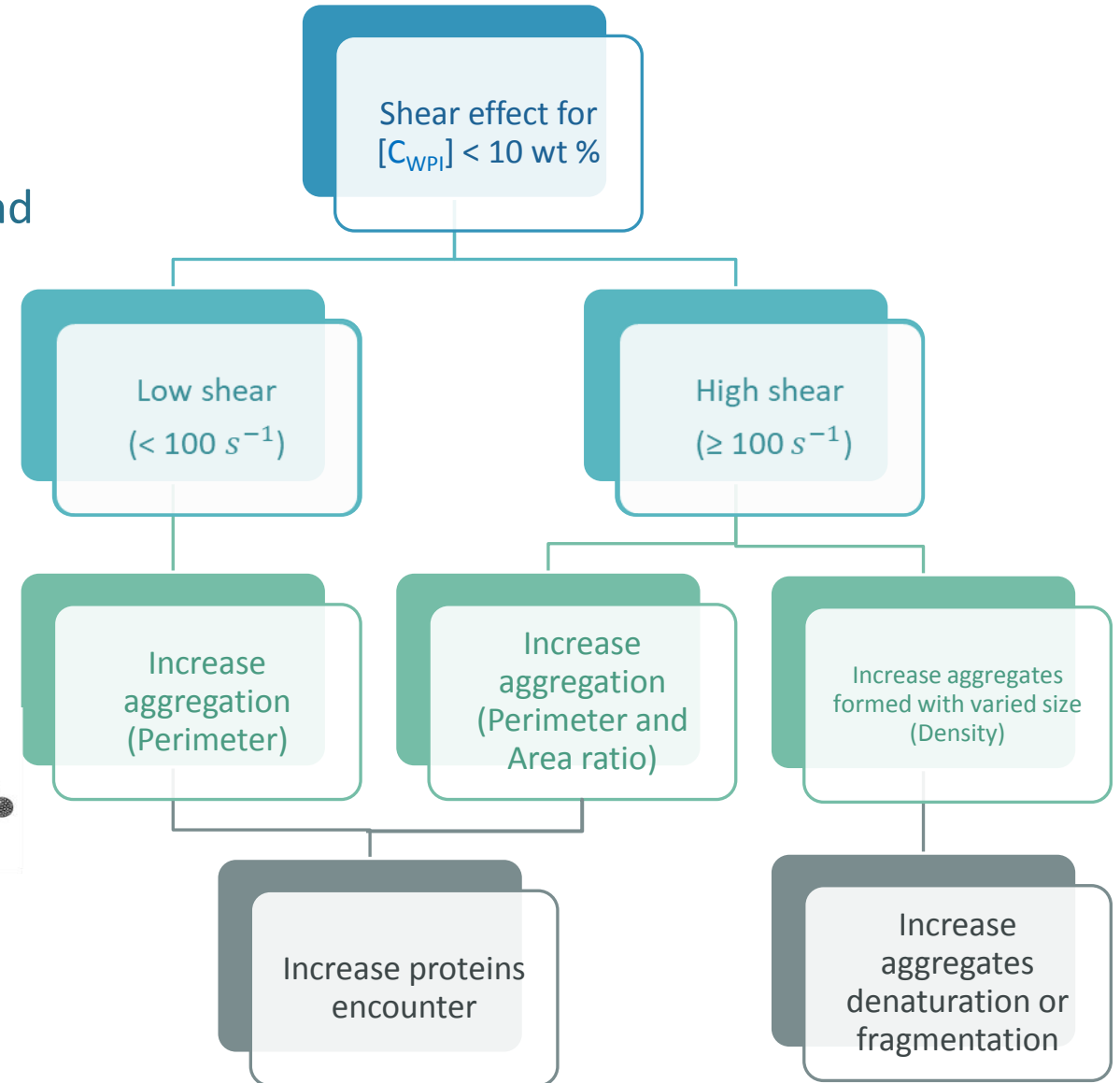
 $[C_{WPI}] = 15 \text{ wt } \%$  $[C_{WPI}] = 20 \text{ wt } \%$ 



- Shearing have an effect on the aggregation : Quantity, compactness, size
- Concentration have an effect on the agregation : Size and structure (branched aggregates)



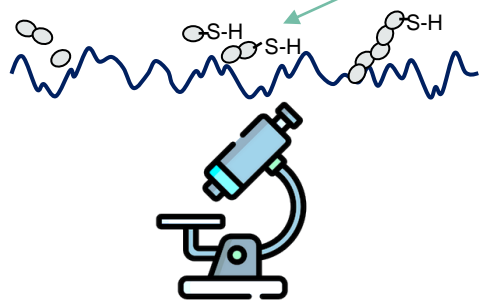
I.B Bekard and D.E. Dunstand, 2014



## Rheometry and microscopy

Surface

Bulk



Explore highest concentration

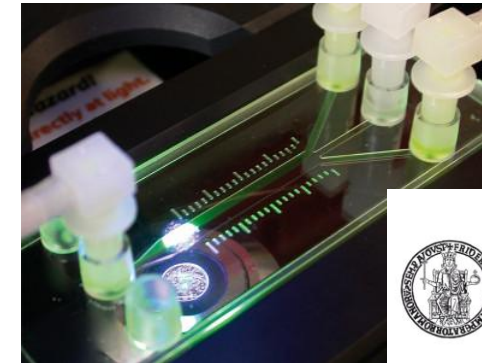
**Cone plate geometry** to confirm effect of shear rate (compare without shear and range of shear)

By using **cone plate geometry** :  
HPLC : Denaturation degree

Flow sweep viscosity : compare a range of concentrations

Frequency sweep : Behaviour of high concentration

## Microfluidic



**DI**  
**C**  
**Ma**  
**PI**  
Dipartimento  
di Ingegneria Chimica,  
dei Materiali e della  
Produzione Industriale  
Università degli Studi  
di Napoli Federico II

**Continuous system** → online and offline analyses

Closer to evaporator system  
Devices with combined stainless steel and glass surfaces

Monitor fouling on the surface and bulk in parallel



Thank you for your attention

Merci de votre attention

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M. Grostete<sup>1</sup>, Z. Msibi<sup>1</sup>, F. Boissel<sup>1</sup>, M. Jimenez<sup>2,3</sup>, R. Jeantet<sup>1</sup>, J. Lee<sup>1</sup>, L. Lanotte<sup>1</sup>

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