Microscopic investigation of fouling mechanisms in dairy protein mixes under shear

Margot Grostete, Msibi Zanélé Paméla, Françoise Boissel, Maude Jimenez, Romain Jeantet, Jehyun Lee, Jeehyun Lee, Luca Lanotte

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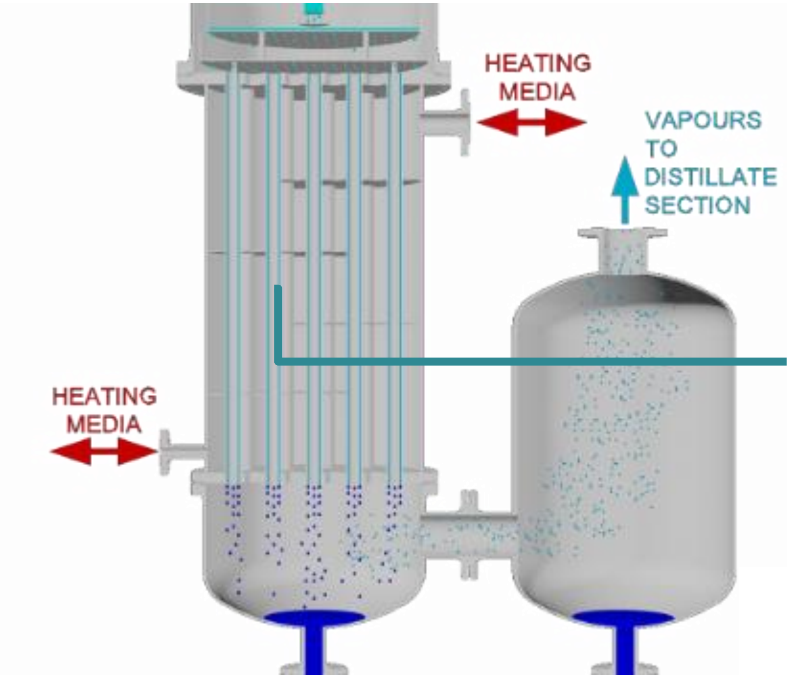
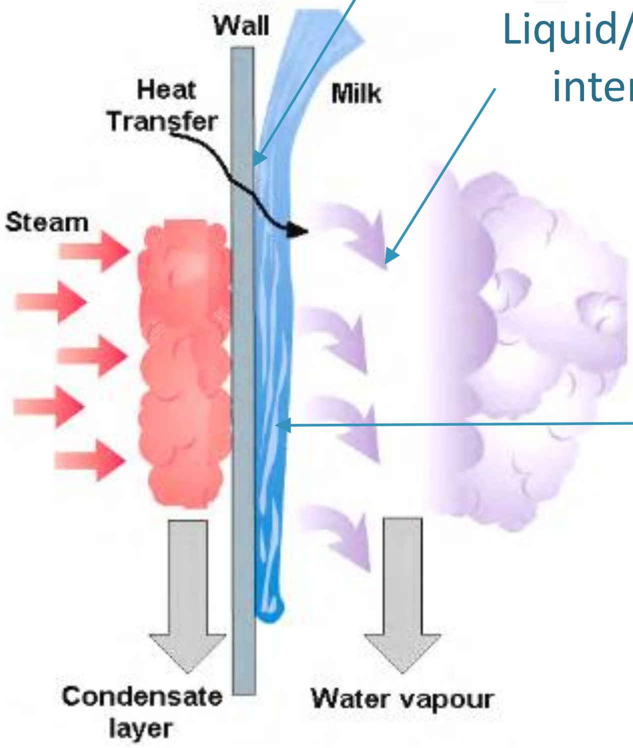
Falling film evaporator



Solid/liquid interface

Liquid/vapour interface



Simple flow : falling by gravity

(Broome S., 2005)

Understand the mechanism of fouling

Whey proteins

Evaporator conditions

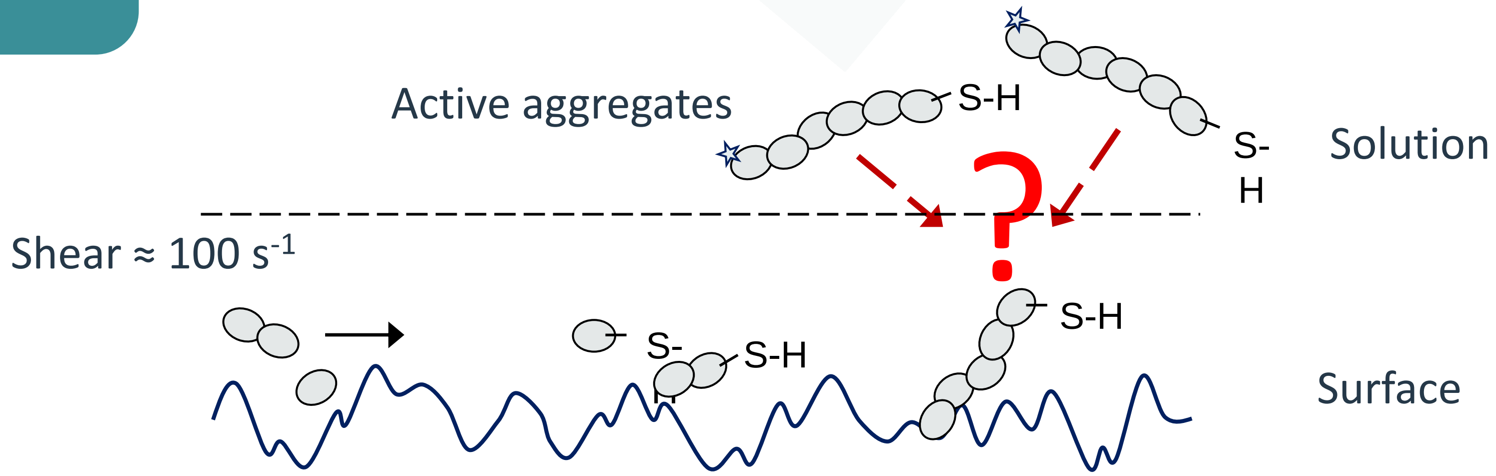
Impact of the shear on the aggregation and deposit

(Bansal B and Chen X.D., 2006 ; Blanpain-Avet P. at al., 2016; Magens O.M. at al., 2018)

Fouling initiation : at the wall surface or in the solution

Rheometry and microscopy

Microfluidics



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

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

Santos at al. (2006)

Simmons at al. (2007)

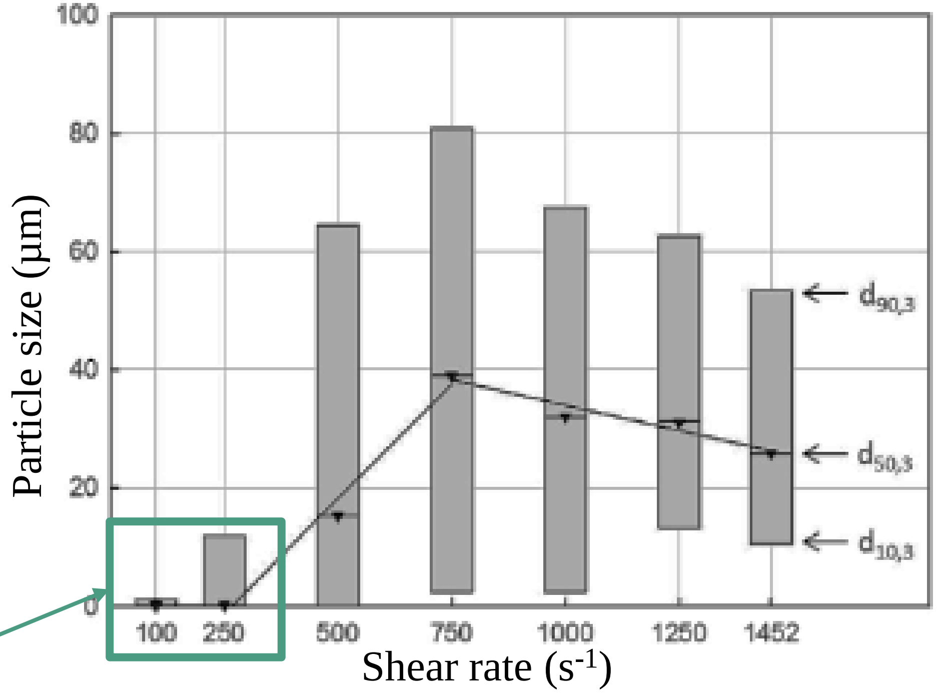
Moakes at al. (2015)

Erabit at al. (2014)

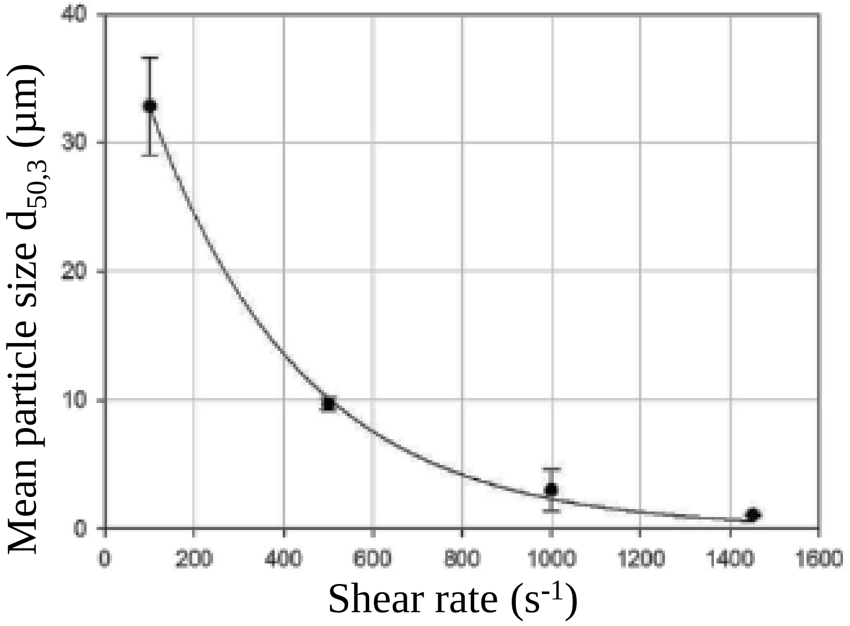
Wolz at al. (2016)

Bogahawatha at al. (2020)

Shear working range for the evaporator



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



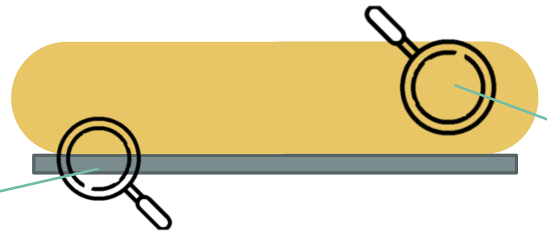
Influence of shear rate on the mean particle size $d_{50,3}$ for a protein concentration of 30 wt % heated 80 °C for 10 s with a degree of denaturation of > 95 %

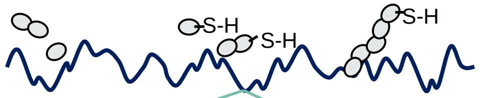
(M. Wolz et al., 2016)

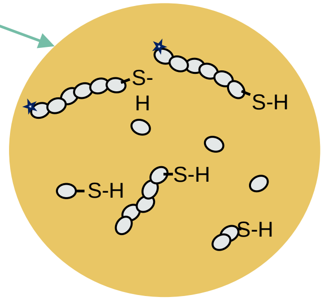
Rheometry and microscopy

Surface

Bulk







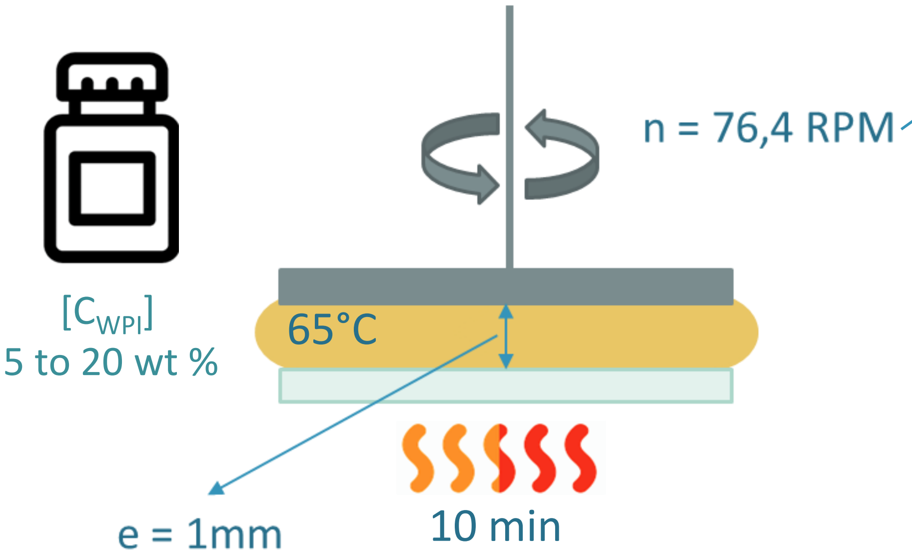
Digital Microscopy

- Large aggregates (> 10 µm)
- Quantitative analysis

SEM

Centre de Microscopie à Balayage et microAnalyse

- All aggregates (< 1 µm to > 50 µm)
- Qualitative analysis



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

What we know :

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

Shear rate for each diameter :

140 à 200 s^{-1} (Periphery)

80 à 140 s^{-1} (Intermediary)

0 à 80 s^{-1} (Centre)

0 s^{-1}

Objective: keep only the aggregates attached to the surface

MiliQ water Rince

Surface

Wasted solution

Oven (40 °C)

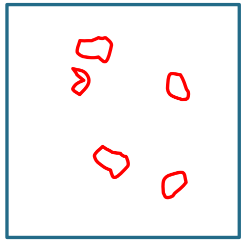
Dry the slides

Clean Surface

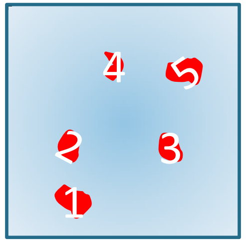
30 pictures/region

Digital microscope (Keyence, VHX 7000)

Keyence software

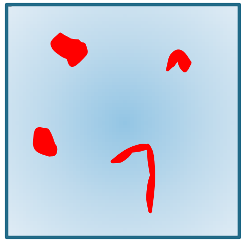


Perimeter (µm)



Density (by number)

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



Area ratio (%)

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

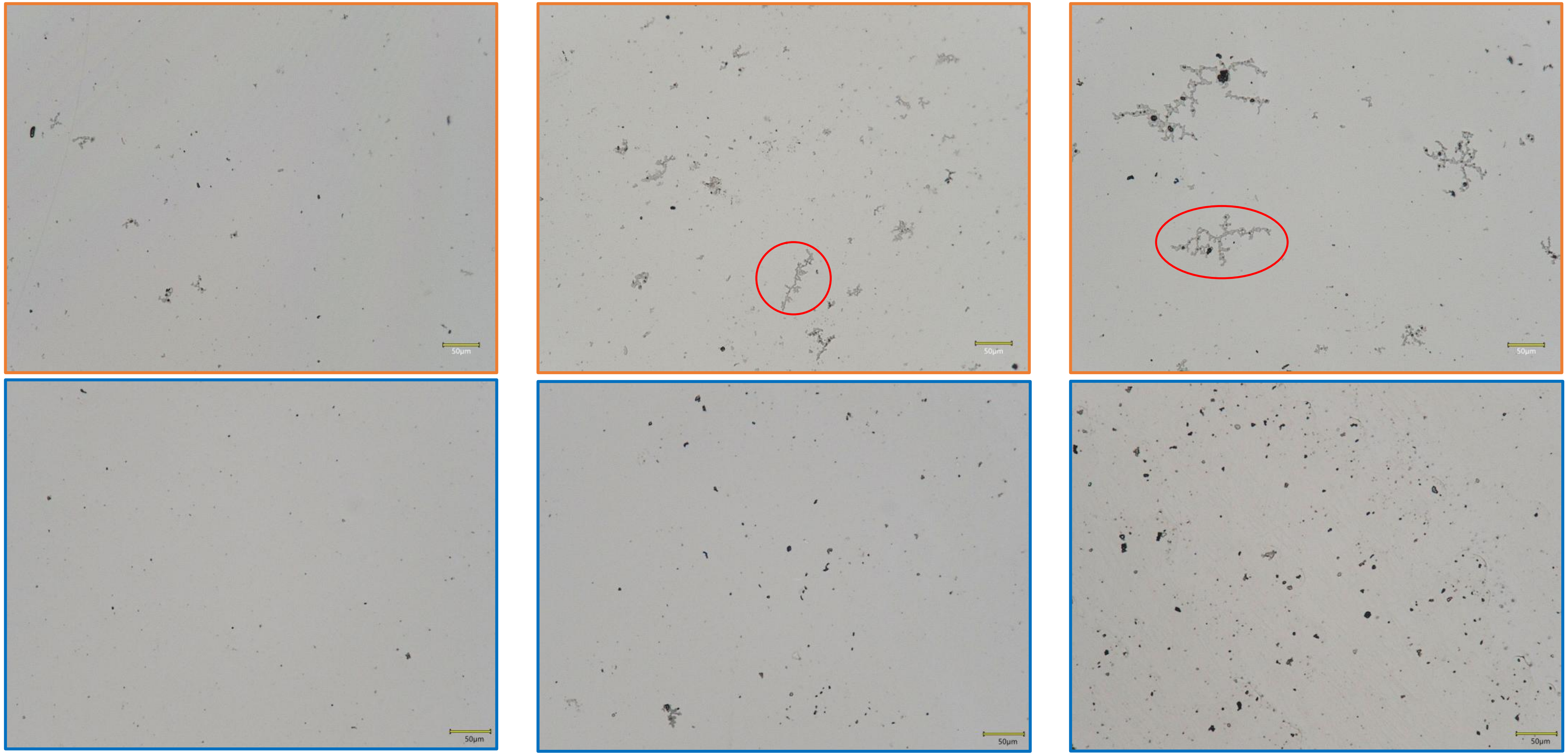
Morphological characterisation

Frequency occurrence

Microscopy General observation (G*500) - SURFACE

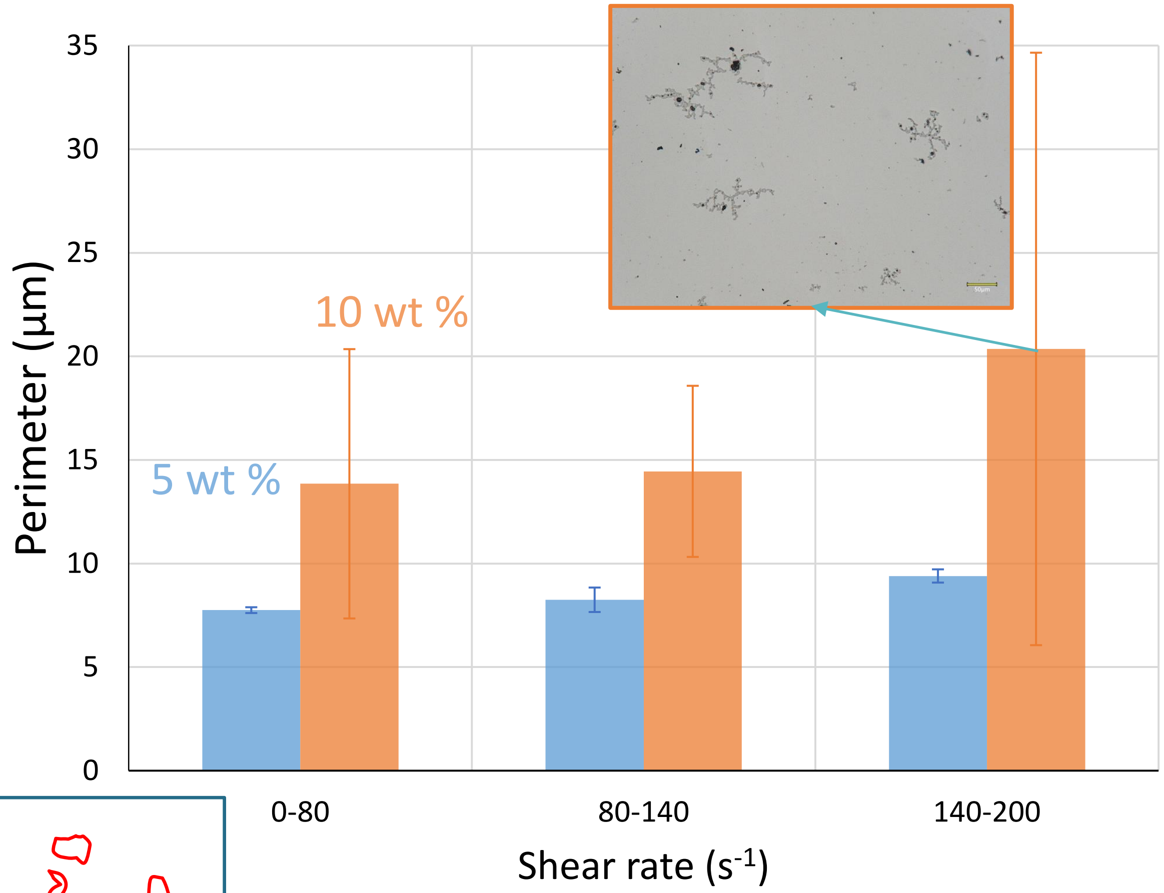
10 wt % WPI

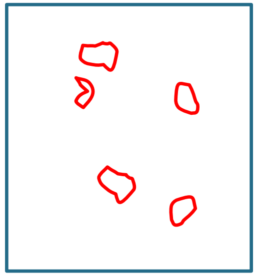
5 wt % WPI



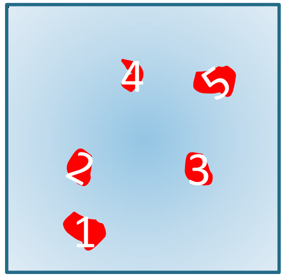
0-80 s^{-1} | 80-140 s^{-1} | 140-200 s^{-1}

Aggregates analyses by Keyence software- SURFACE



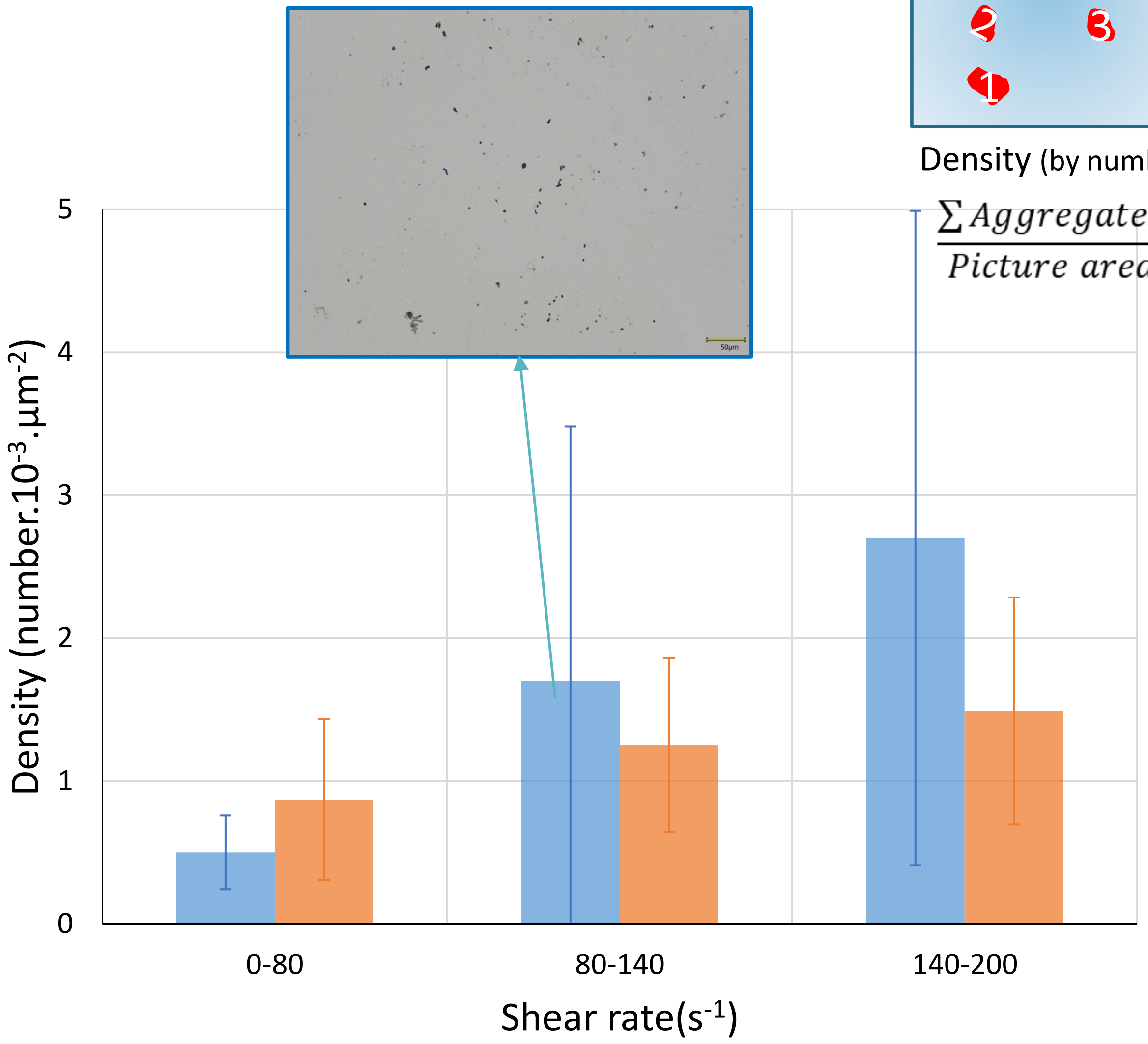


Perimeter (µm)

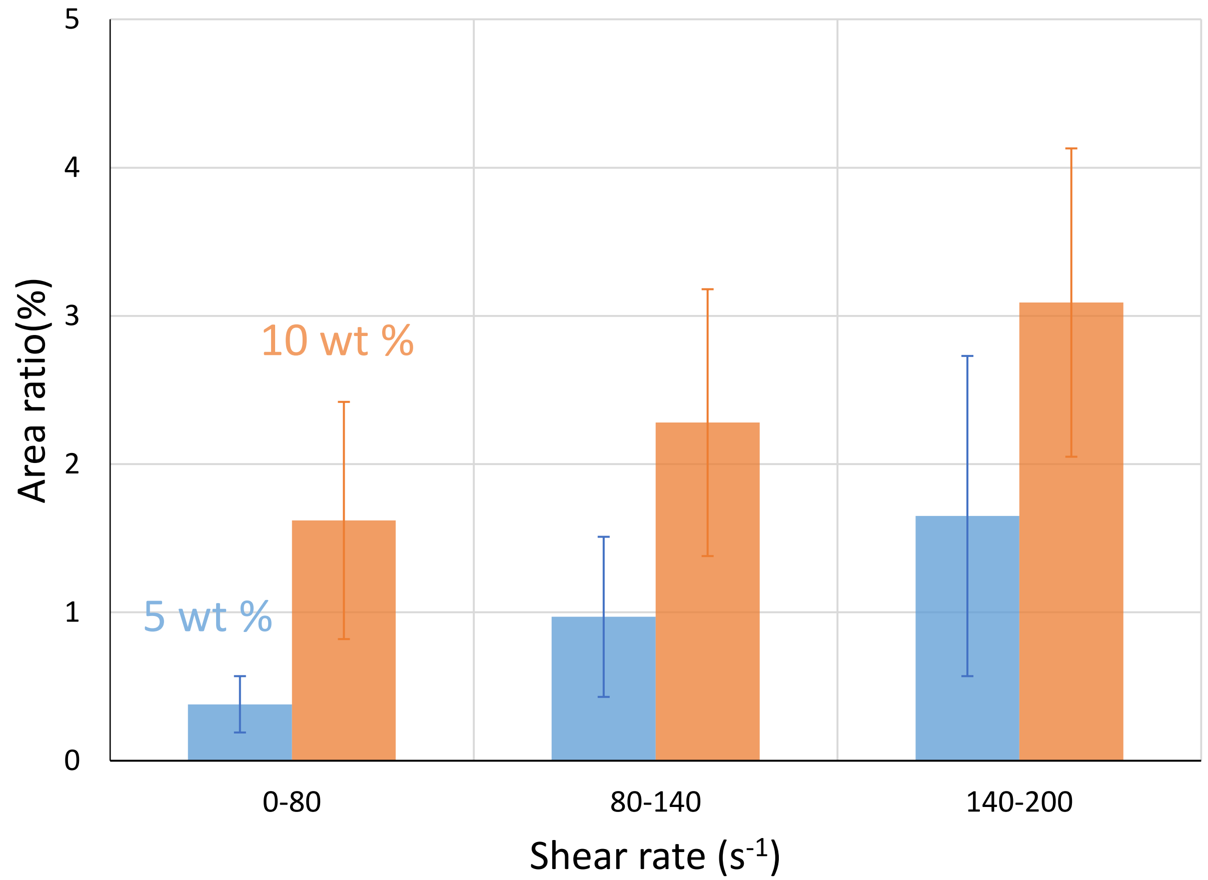


Density (by number)

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



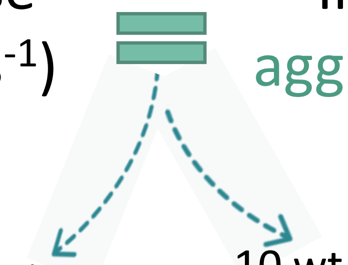
Aggregates analysis by Keyence software- SURFACE



Area ratio (%)

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

Increase Shear (s^{-1})



Impact aggregation

5 wt % WPI increased number of aggregates

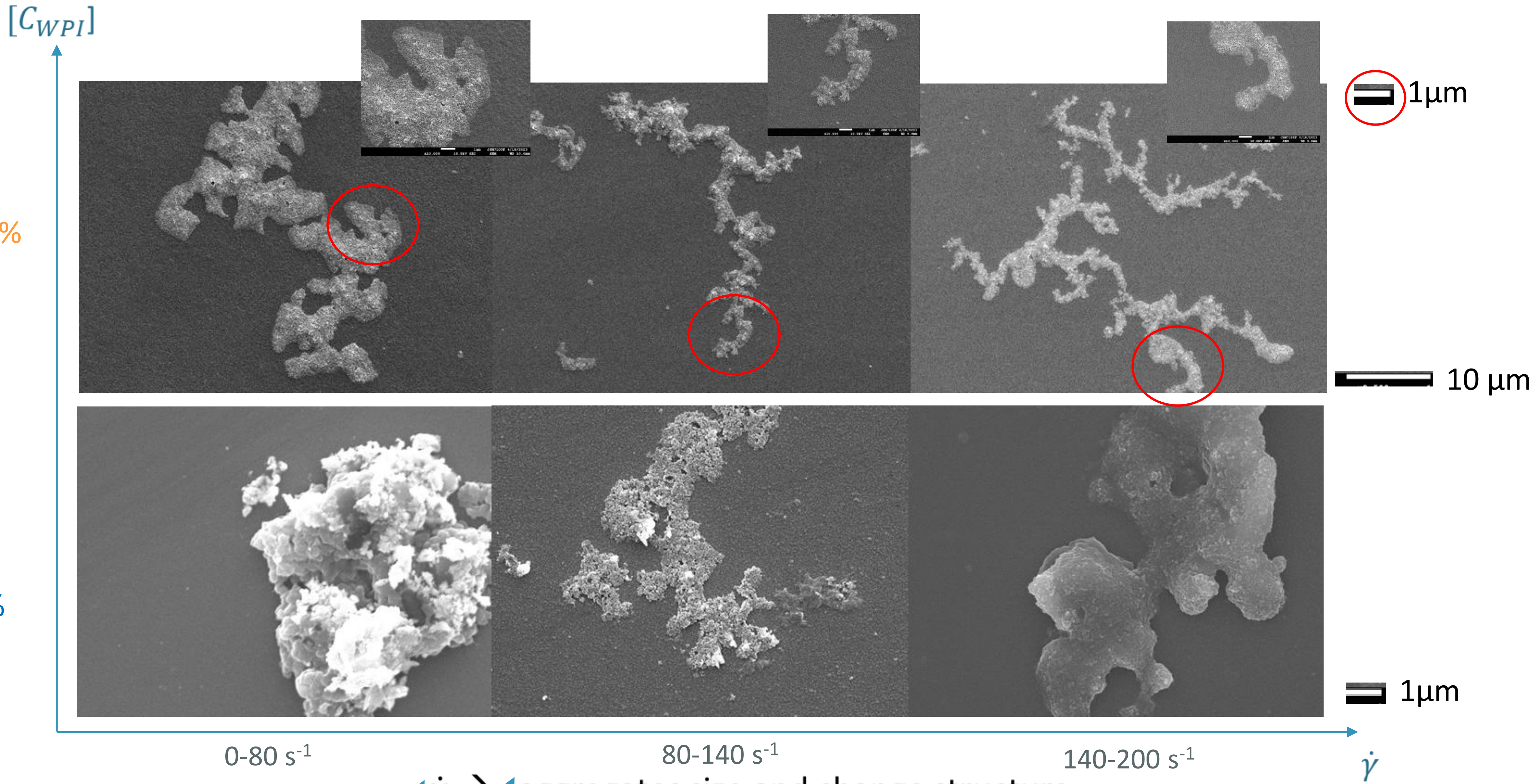
10 wt % WPI increased size of aggregates

Standard deviation

- Shear gradient of each region?

$$Shear \Leftrightarrow Deposit$$

SEM 5 wt % and 10 wt % WPI



$[C_{WPI}]$ = 10 wt %

↑

Increase aggregates size

↑

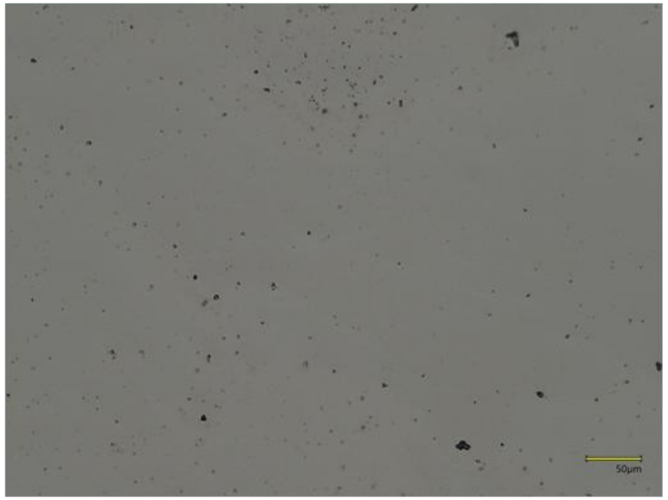
$[C_{WPI}]$ = 5 wt %

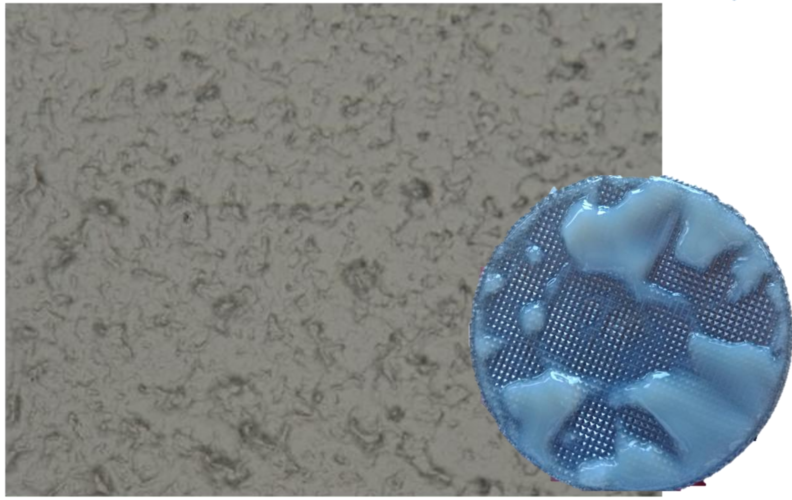
↗$\dot{\gamma}$ → ↗aggregates size and change structure

Microscopy of 15 wt % and 20 wt % WPI solution

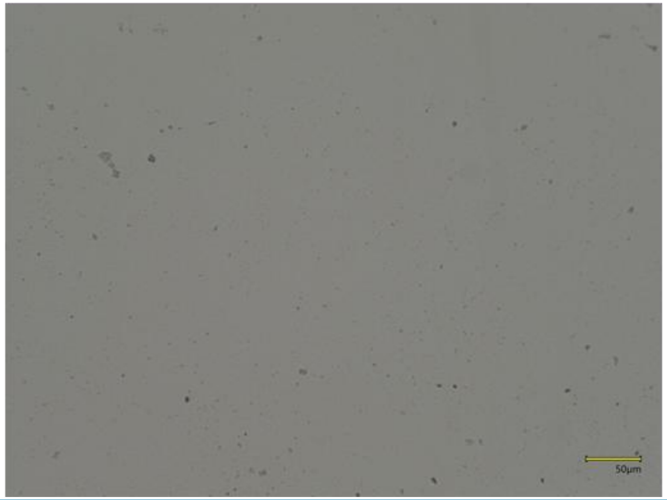
2min | 5 min | 10min

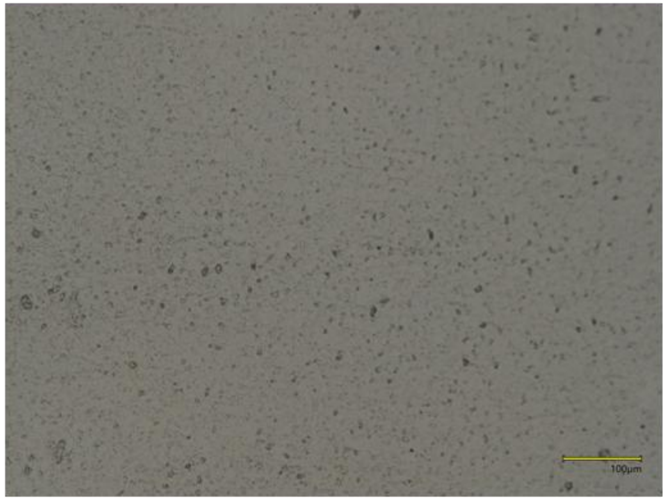
$[C_{WPI}]$ = 15 wt %

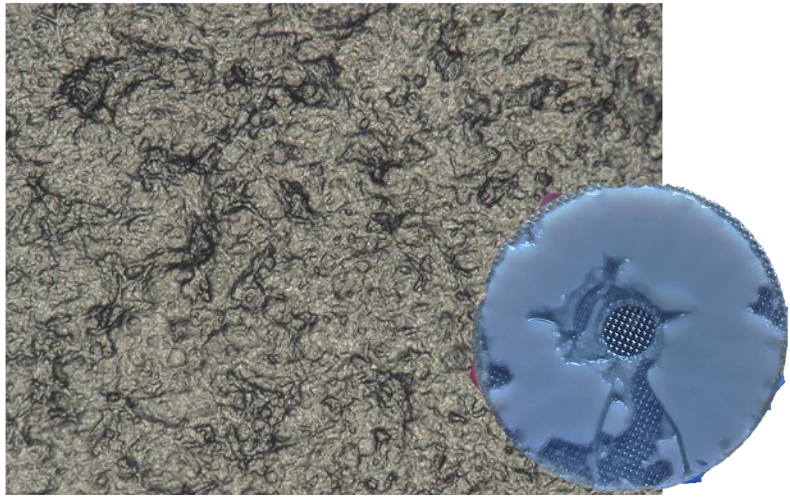




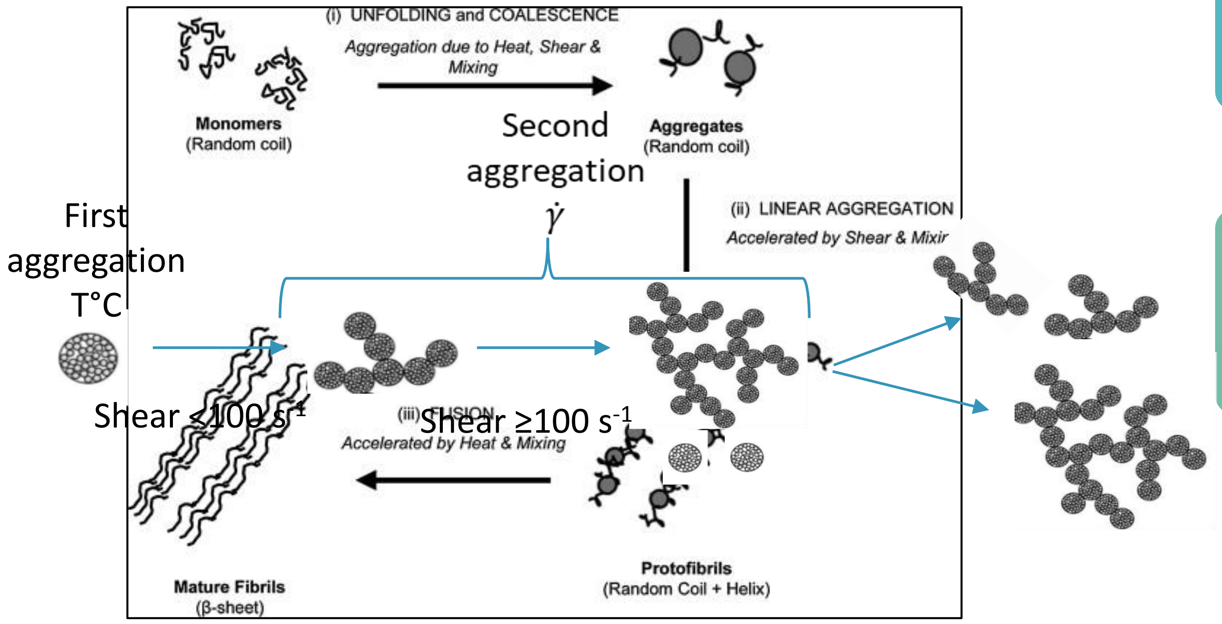
$[C_{WPI}]$ = 20 wt %







- 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

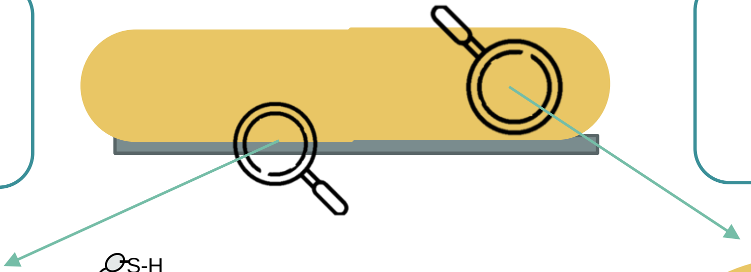
Shear effect for $[C_{WPI}] < 10$ wt %

- Low shear ($< 100\ s^{-1}$)
  - Increase aggregation (Perimeter)
- High shear ($\geq 100\ s^{-1}$)
  - Increase aggregation (Perimeter and Area ratio)
  - Increase aggregates formed with varied size (Density)
    - Increase aggregates denaturation or fragmentation

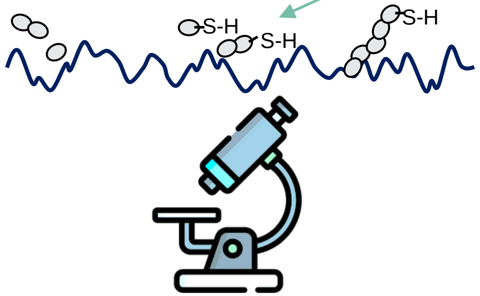
Increase aggregation (Perimeter) + Increase aggregation (Perimeter and Area ratio) → Increase proteins encounter

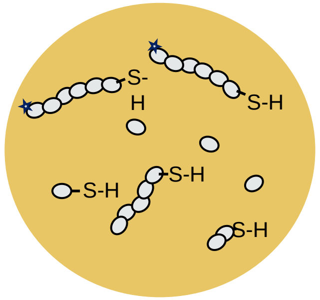
Rheometry and microscopy

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

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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