

35th Conference “Rheology of Building Materials”

Effect of sample loading procedure on parallel-plate rotational rheometry of cement paste

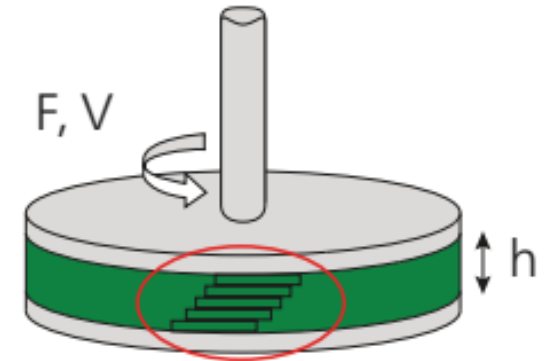
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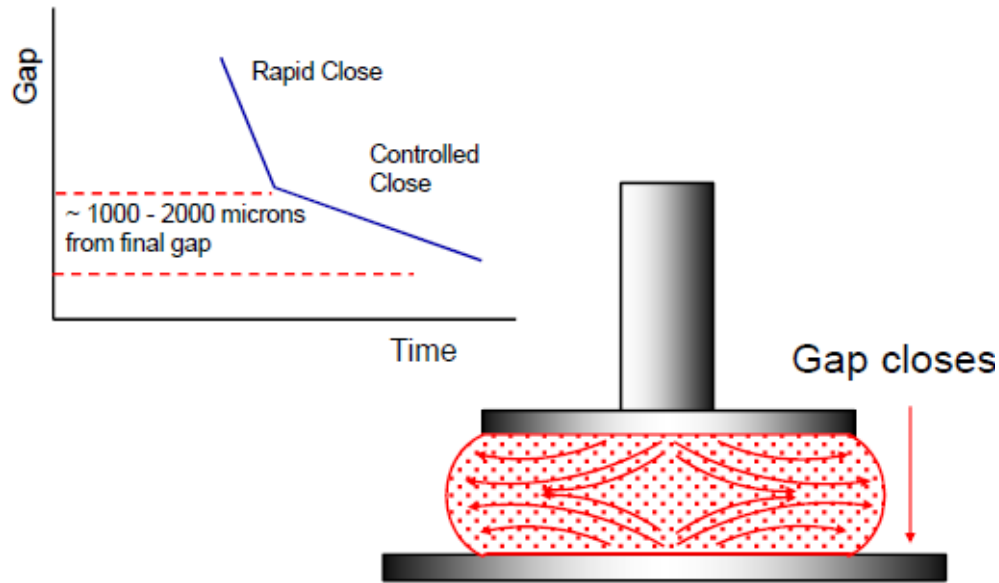
Parallel-plate rheometry

- Parallel-plate geometry is extensively used for the rheometry of cement pastes and other concentrated suspensions.
- Shear or oscillatory flow
- Shear rate range can be adjusted by gap, diameter and velocity
- Gap adjustments according to particle size
- Testing protocols and pre-shearing steps are mentioned.
- However, the procedure used to load the sample and closing the gap is neglected in scientific papers**

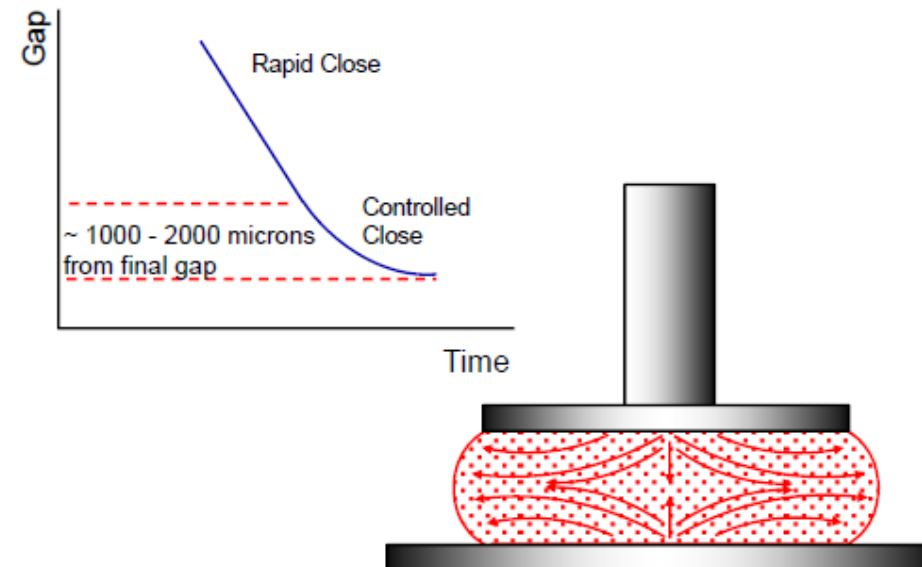


$$\dot{\gamma} = \frac{\Omega R}{h} \quad \sigma = \frac{2\Gamma}{\pi R^3}$$

Linear Gap Closing



Exponential Gap Closing

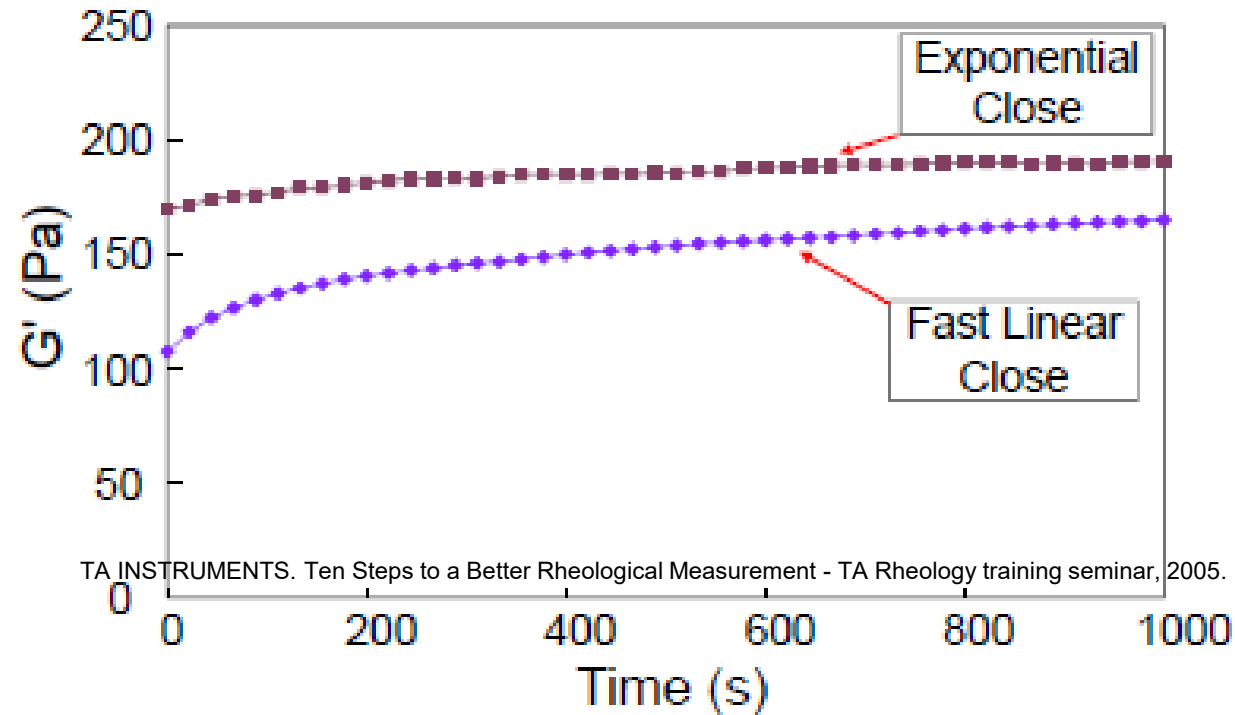


TA INSTRUMENTS. Ten Steps to a Better Rheological Measurement - TA Rheology training seminar, 2005.

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- Rheometer manufacturers suggest to close the gap (i.e. squeeze the sample) slowly to avoid disturbing the suspension's structure.
- This is worthy for structured fluids, gels and highly viscoelastic materials, **but may not be the best choice for all suspensions!!**

Comparison of Linear and Exponential Gap Closing



- Rheometer manufacturers suggest to close the gap (i.e. squeeze the sample) slowly to avoid disturbing the suspension's structure.
- This is worthy for structured fluids, gels and highly viscoelastic materials, **but may not be the best choice for all suspensions!!**

Determine the influence of sample loading procedure on the rotational flow behaviour of a cement paste.

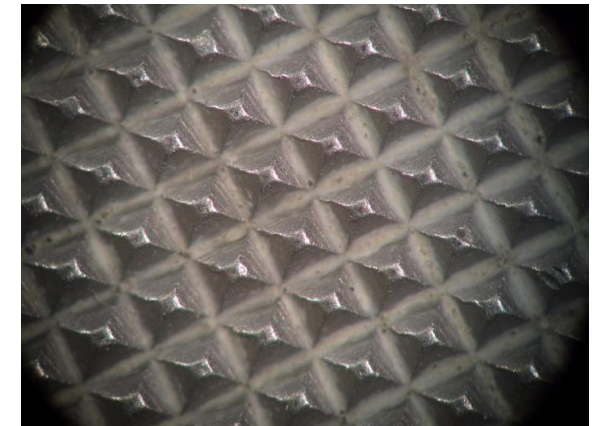
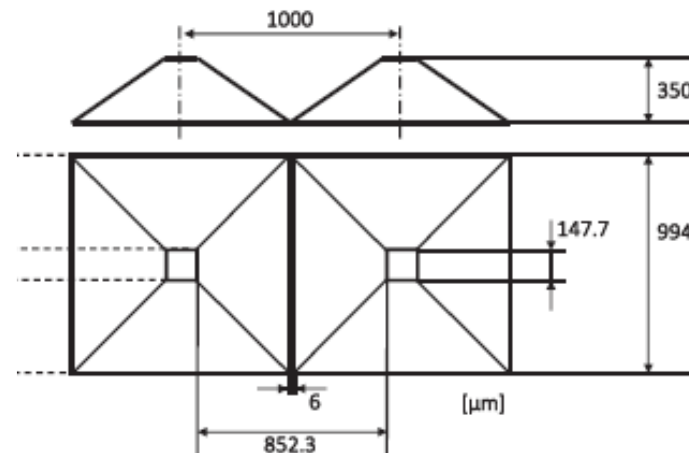
Paste

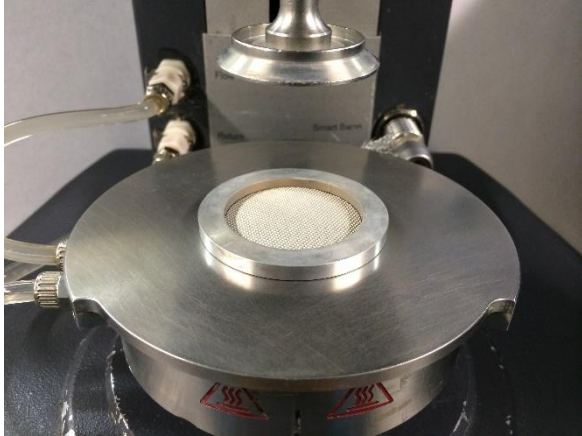
- Portland cement CEM I 52.5N Lafarge Ciments - Usine Du Havre, France;
- Water/cement ratio = 0.40; concentration = 44.65%vol, 2% entrained air

Cimento	ρ (g/cm ³)	SSA (m ² /g)	D10 (μ m)	D50 (μ m)	D90 (μ m)
CEM I 52.5 N	3.10	1.05	4.1	21.2	55.0

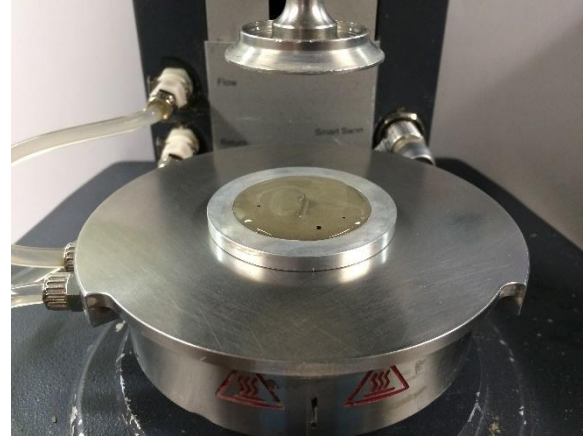
Rheometer:

- AR2000 Ex (TA Instruments) with \varnothing 40mm parallel-plate geometry and crosshatched surfaces
- 2.8mm gap for squeeze-flow and 1mm gap rotational shear cycles

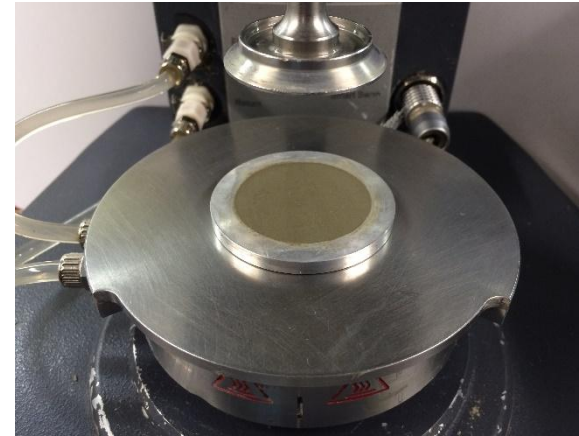




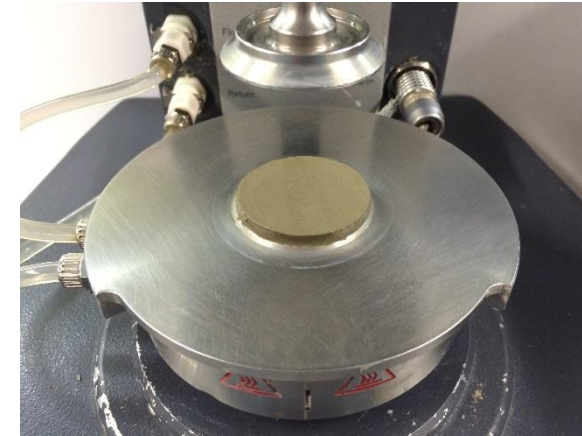
1. Mold preparation



2. Put the sample



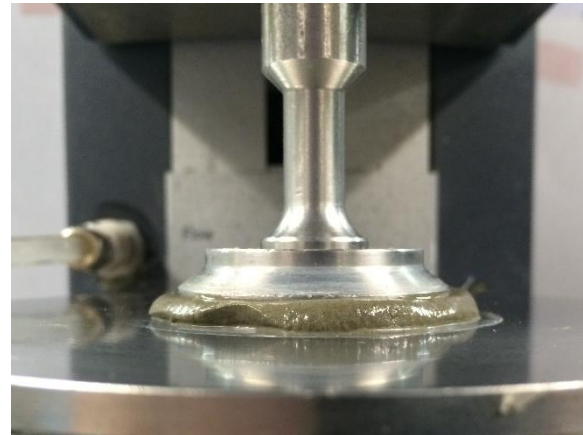
3. Flatten the sample



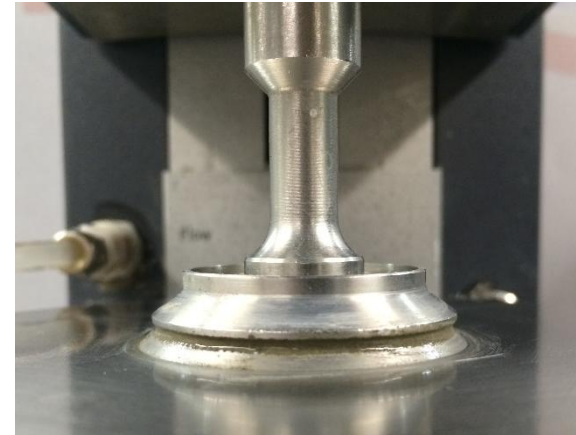
4. Demolding



5. Positioning top plate



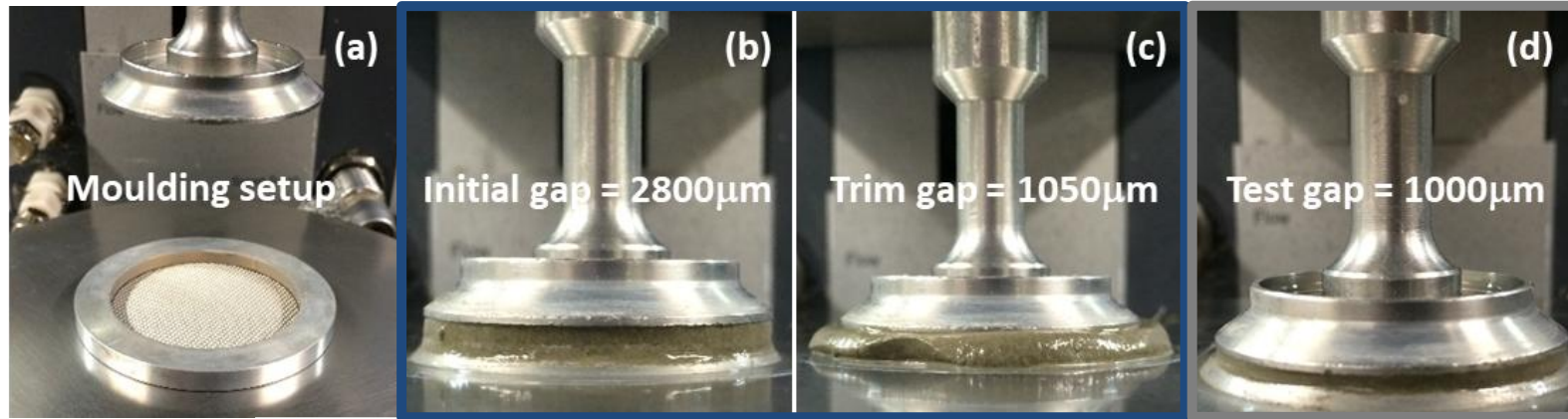
6. Squeeze flow until reaching gap=1050µm



7. Trimming the sample at gap=1050µm

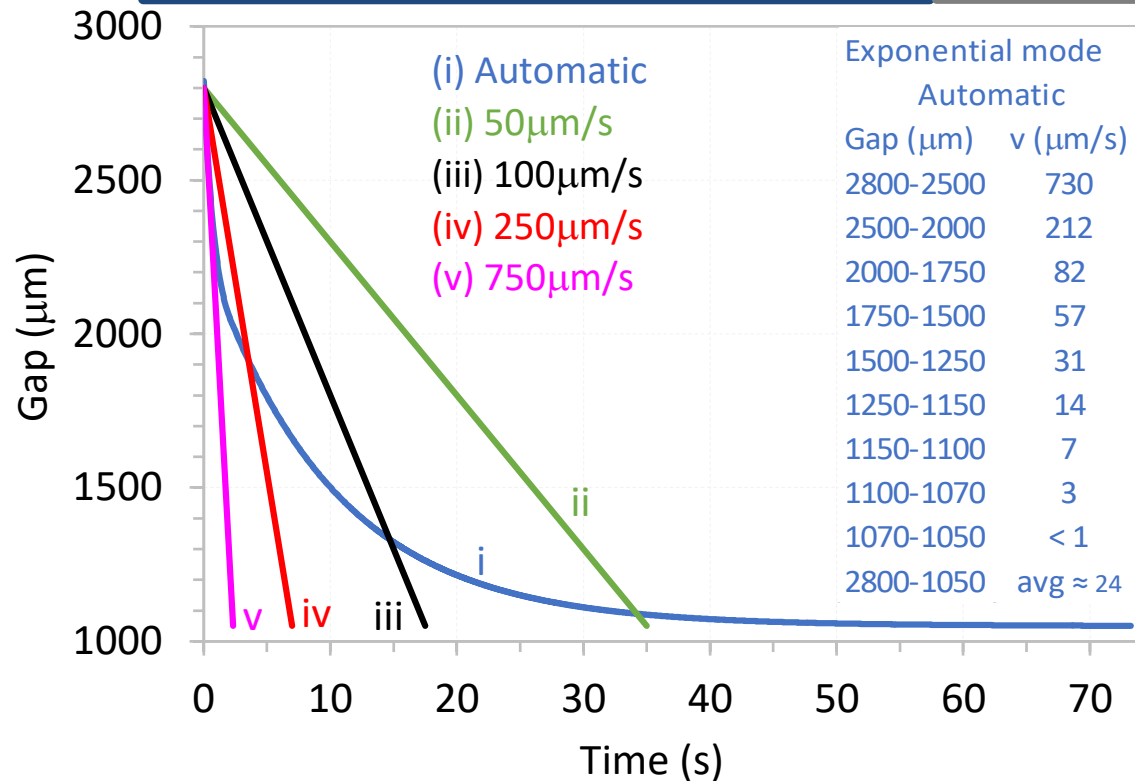


7. Head down in exponential automatic mode until gap=1000µm



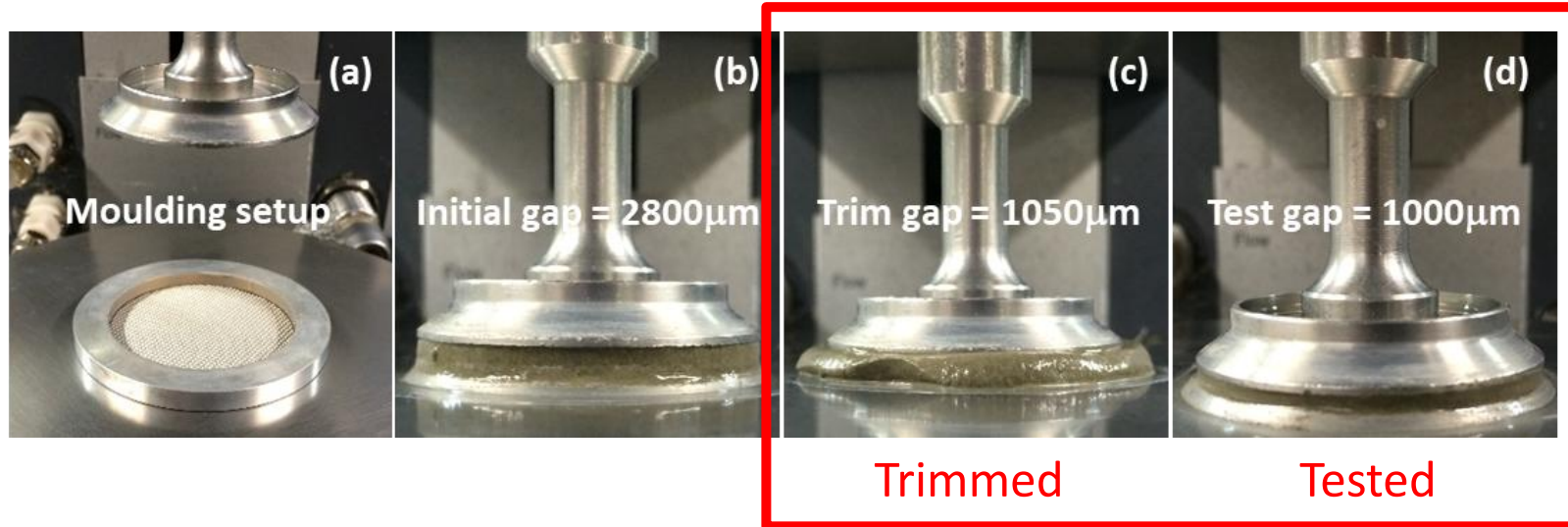
Gap positioning: first step

- 2800-1050µm
- Squeeze-flow test at different speeds
- Measuring normal force
- Performed at 5min and 35min after mixing



Gap positioning: second step

- 1050-1000µm
- automatic exponential decay mode
- $v \approx 1.4\mu\text{m/s}$
- Ensure exact gap
- The same for all tests

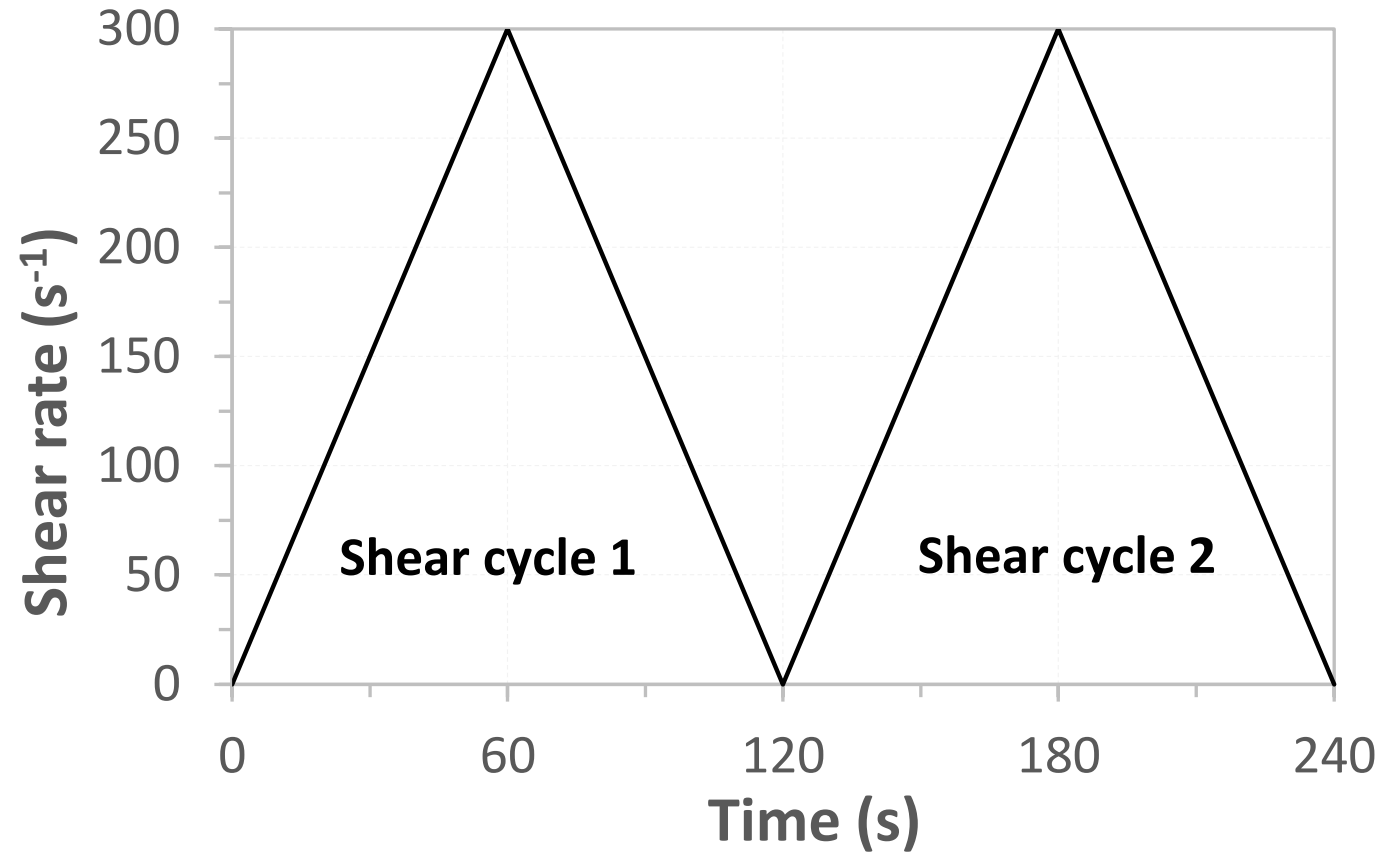


Phase separation evaluation: determination of water content

- Trimmed portion of the pastes and the ones tested under rotational cycle were microwave dried for 7min (Micro-ondes 800W, – Samsung France).

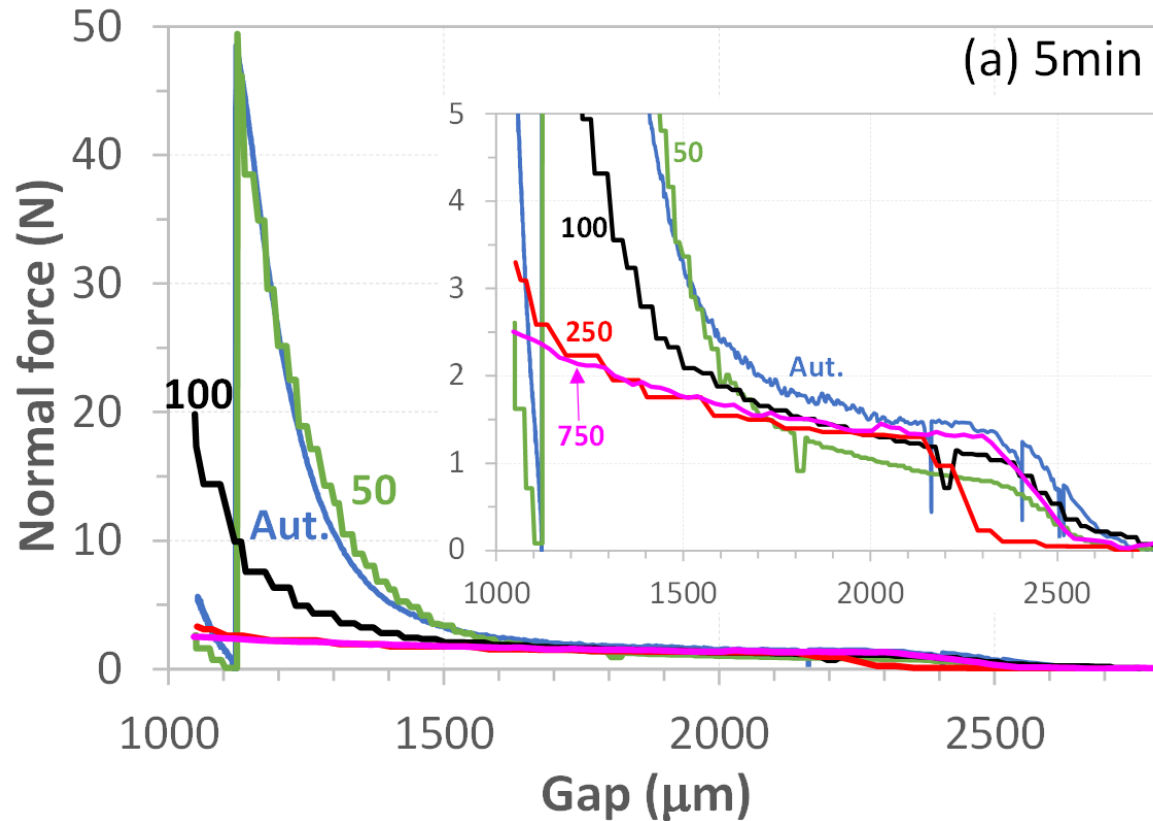
Shear-controlled cycles

- Two consecutive ramp cycles
- Maximum rate 300s^{-1}
- Time per cycle = 120s
- Total time 240s
- Constant temperature 23°C
- Solvent trap
- Performed at 5 and 35min after mixing

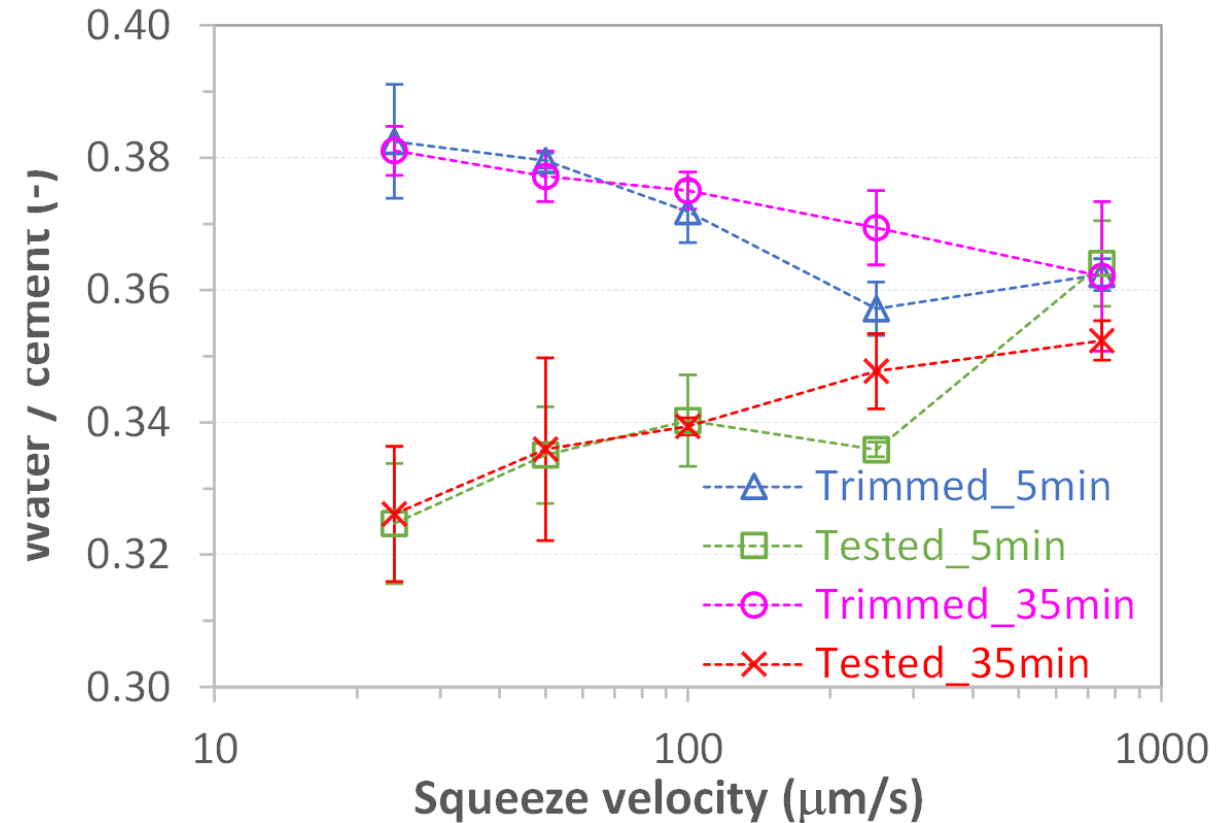


- The whole experimental procedure (mixing, sample loading, phase separation and rotational shear cycles) was performed 3 times. Three paste batches were prepared and tested for each squeezing speed.
- Squeeze (sample loading), phase separation and rotational shear cycles were performed at 5 and 35min after mixing.

Squeeze-flow

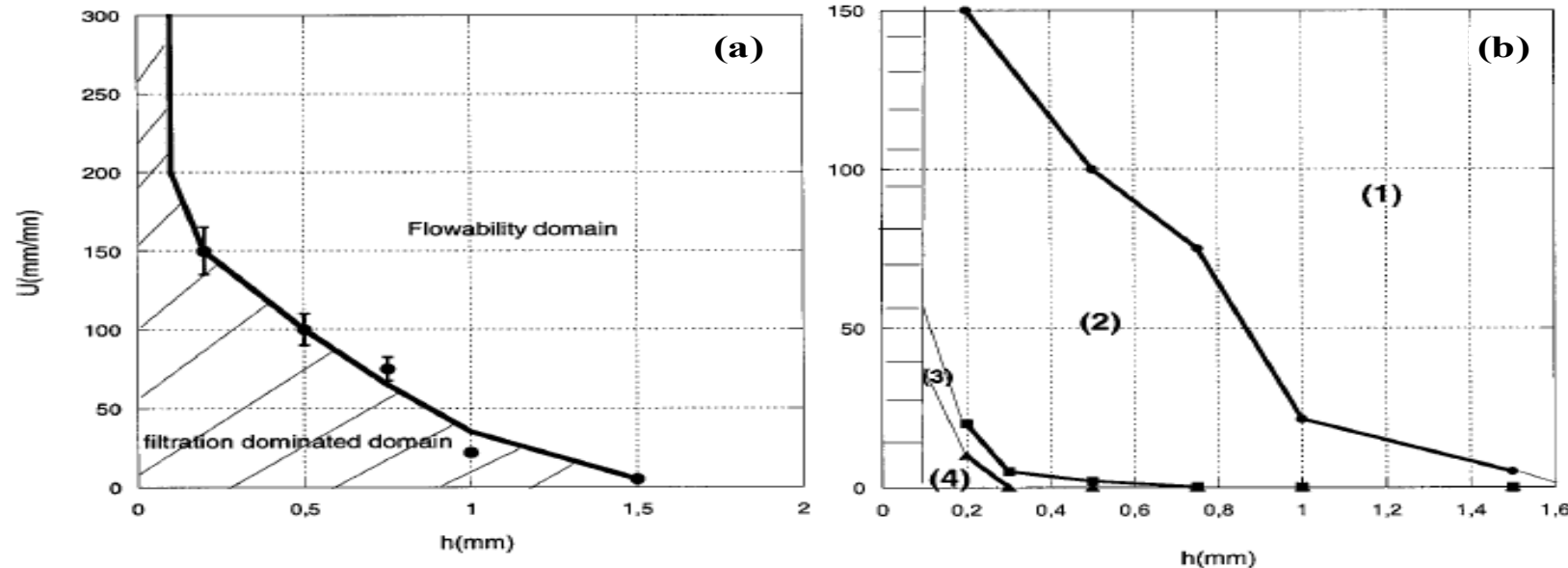


Phase separation



- ❖ Low speeds caused substantial increase of normal force (strain hardening) due to liquid radial migration: squeezed out (trimmed) portions had more water, while samples subjected to rotational tests had higher solid concentration.
- ❖ At 750mm/s, samples were more homogeneous.

- Suspensions under squeeze flow: liquid phase migration plays an important role on their rheological behaviour

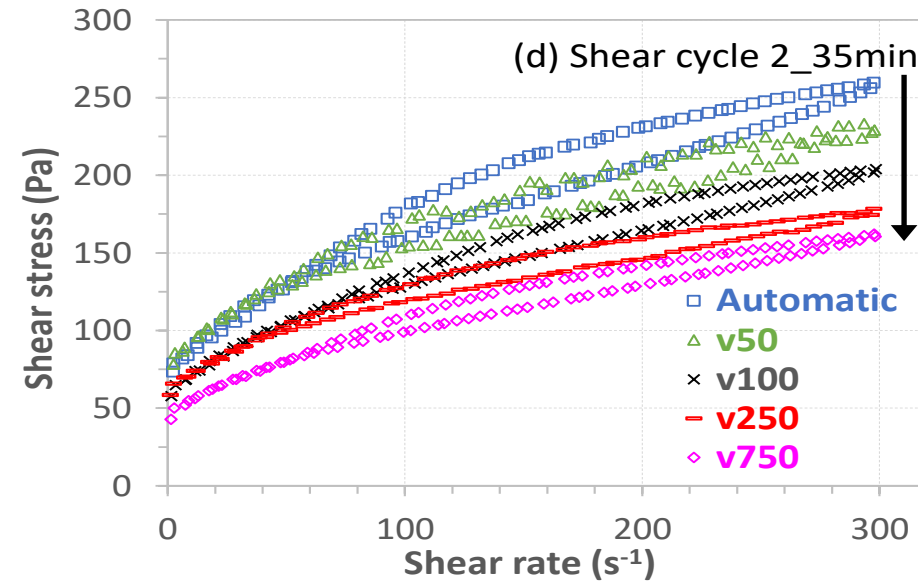
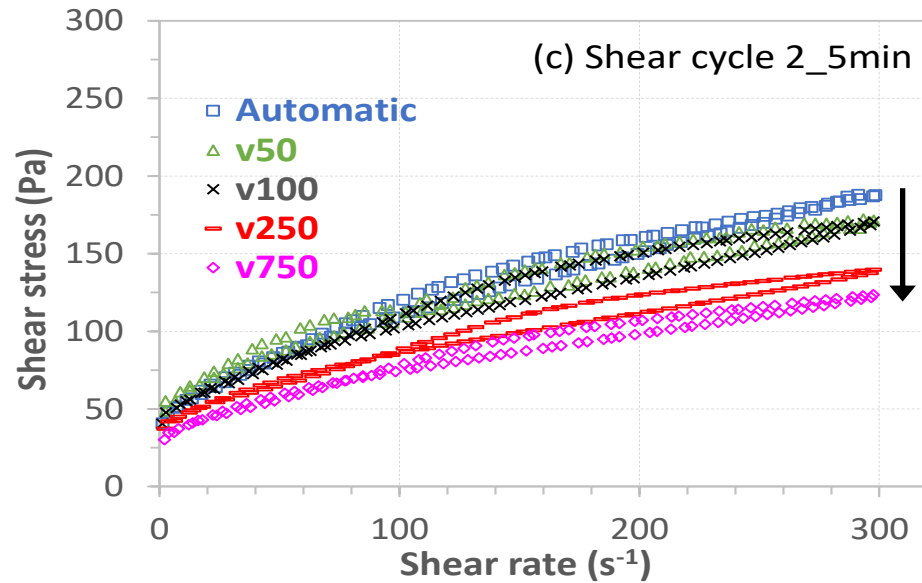
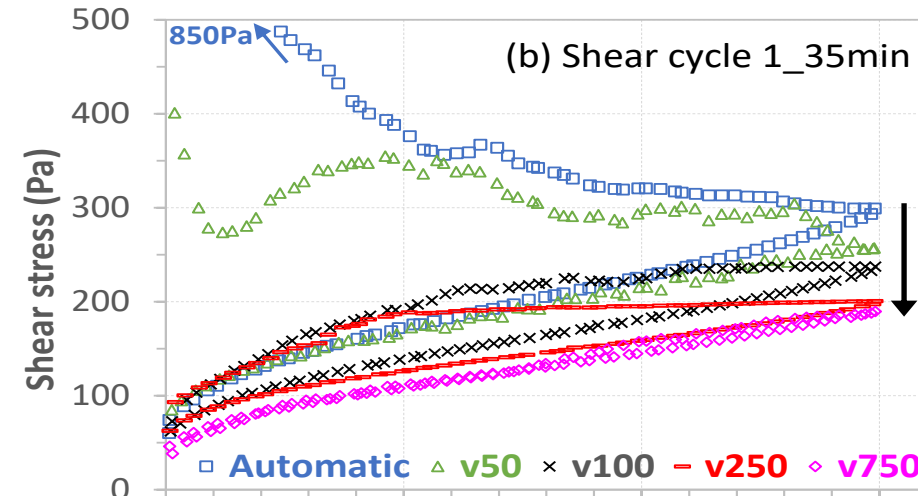
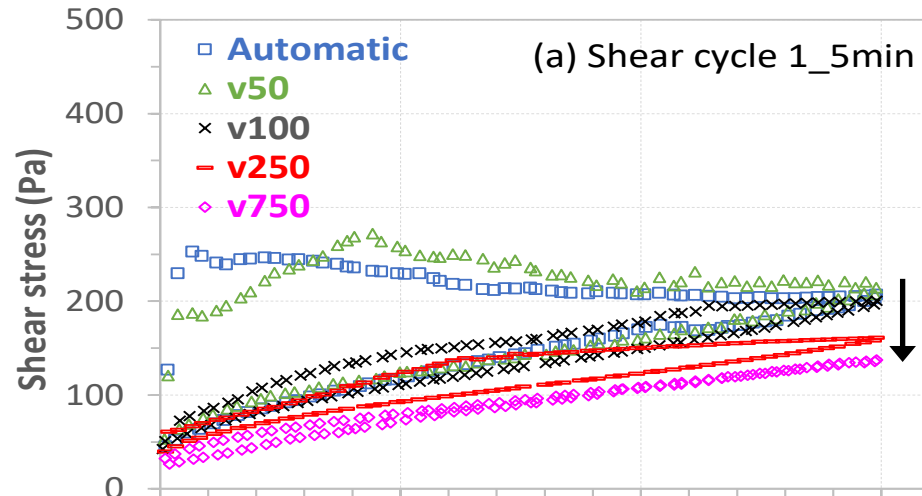


COLLOMB, J.; CHAARI, F.; CHAOUCHÉ, M. Squeeze flow of concentrated suspensions of spheres in Newtonian and shear-thinning fluids. *Journal of Rheology*, 48, 405-416, 2004.

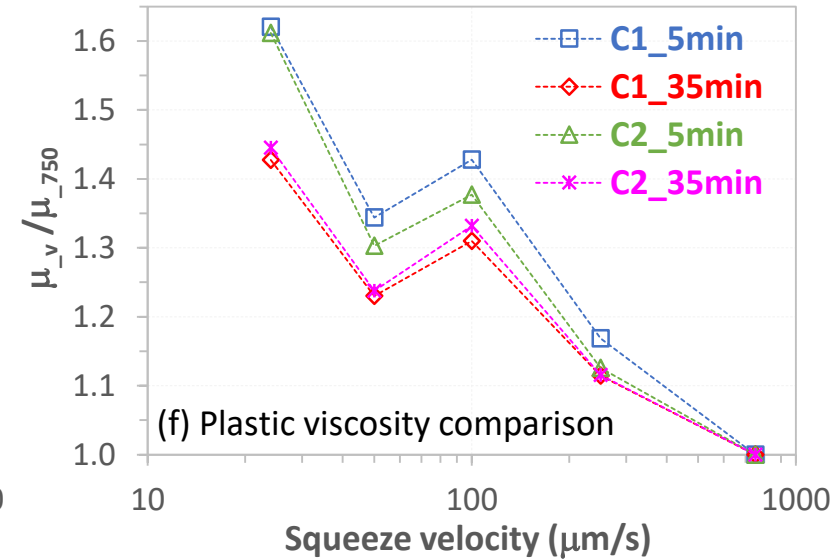
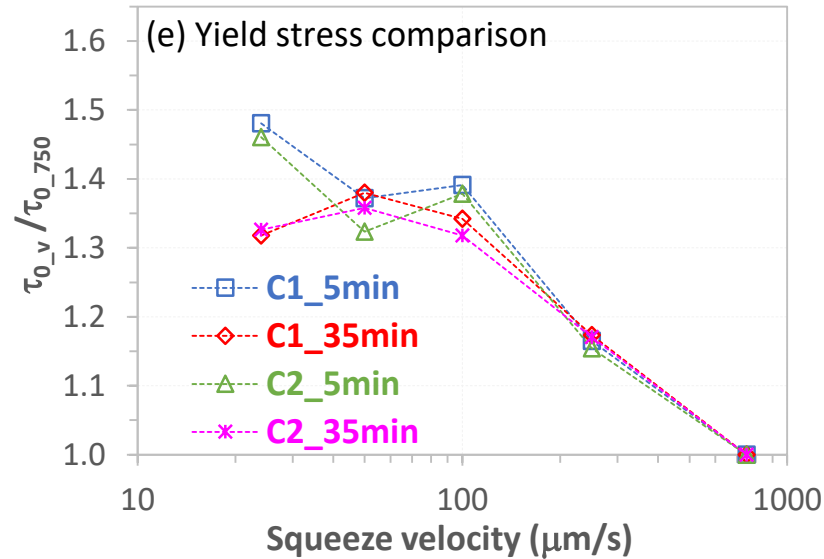
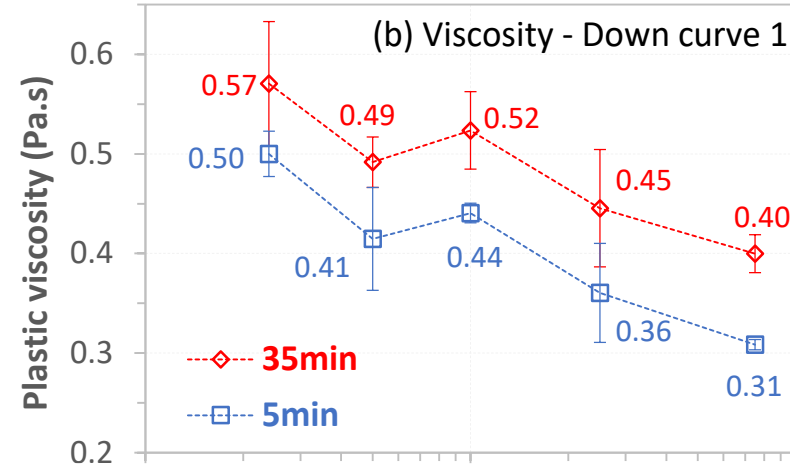
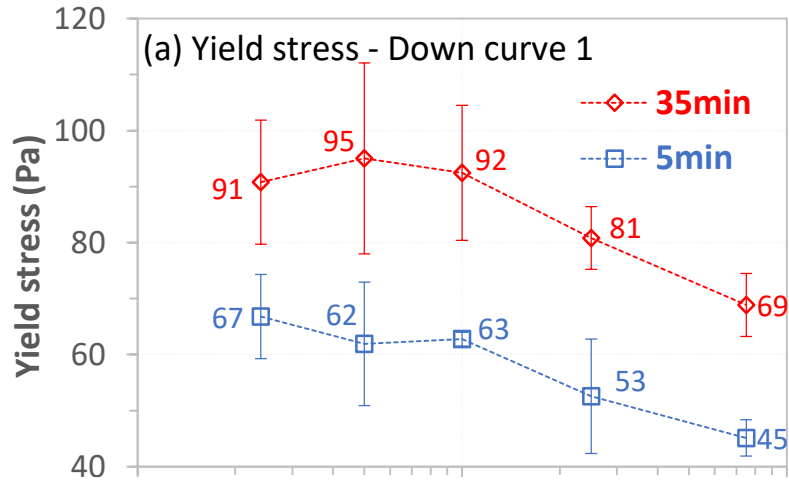
$$Pe = \frac{\tau_w}{\tau_s} = \frac{\mu_w U^{1-m} h^{m+1}}{Ak}$$

- Homogeneous flow and filtration domains
- Combination of velocity and sample height
- \uparrow Displacement rate = \downarrow Liquid phase migration likelihood
- \downarrow Sample height = \uparrow Strain = \uparrow Liquid phase migration likelihood

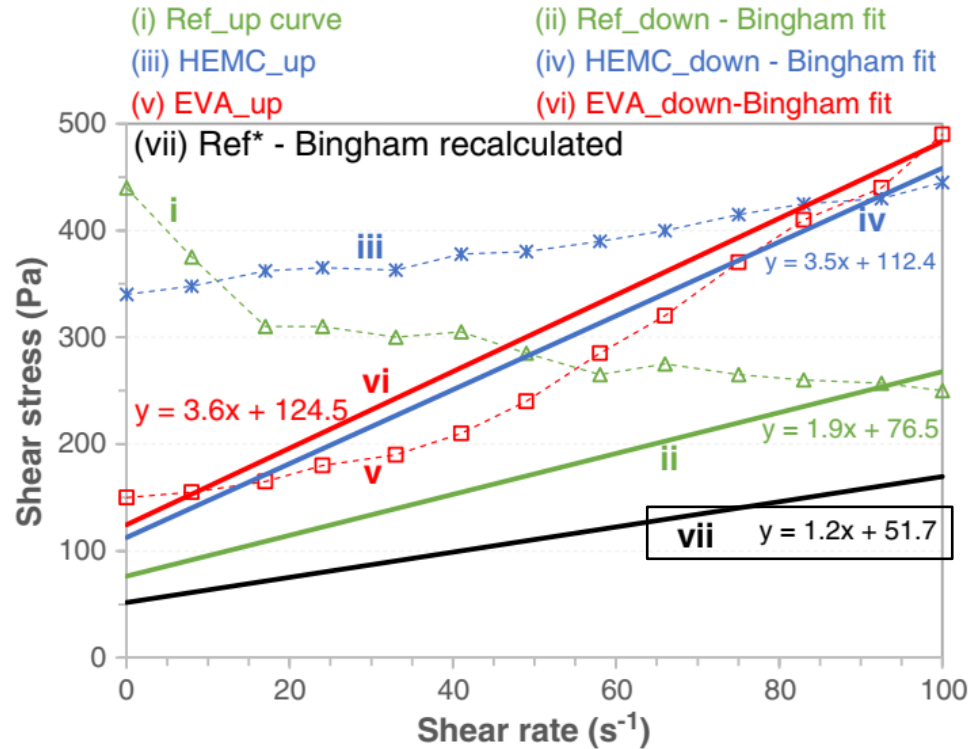
- ❖ Competing phenomena: Flow vs. Liquid filtration
- ❖ High Pe = low/no liquid-solid phase separation
- ❖ Low Pe = high liquid-solid phase separation



❖ Due to their higher solid content, the samples loaded at slower velocities presented much higher hysteresis area during the first cycle and higher shear stress levels.



❖ Yield stress and plastic viscosity of samples loaded with the automatic exponential mode or slow linear squeezing speeds were overestimated, 30 to 65%, due to their higher solid content.

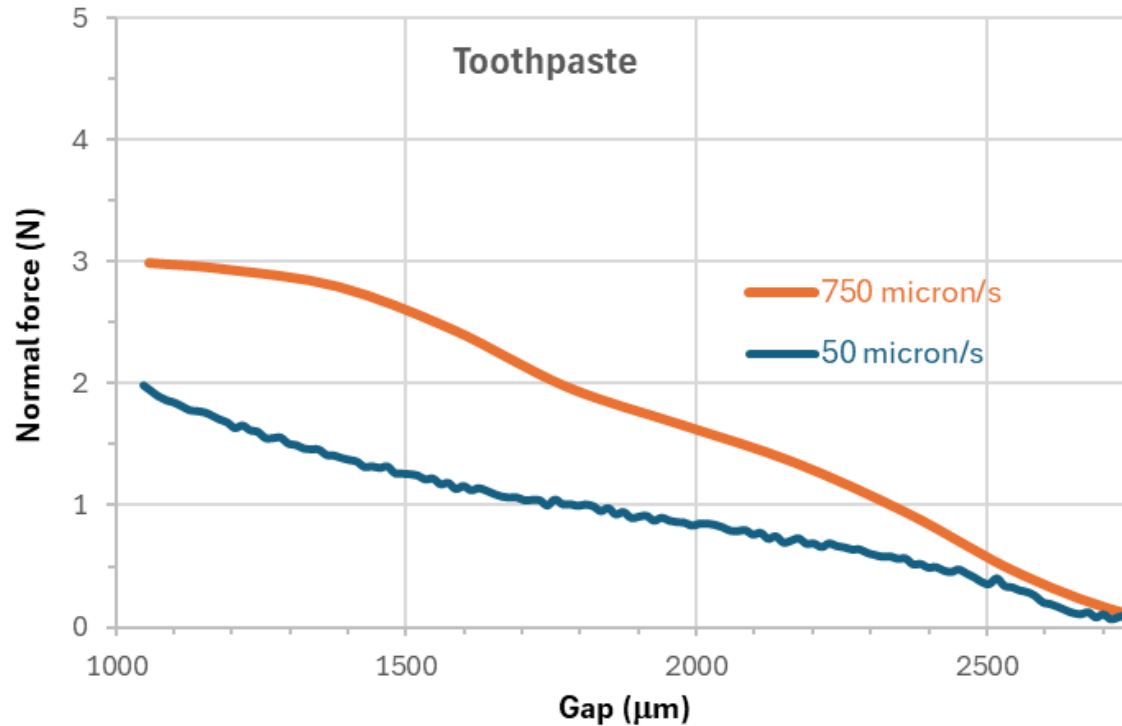


Paste	$100 * \left(\frac{\mu, \tau_0}{\mu_{Ref}, \tau_{0Ref}} - 1 \right) (\%)$			
	μ/μ_{Ref}	μ/μ_{Ref*}	τ_0/τ_{0Ref}	τ_0/τ_{0Ref*}
0.25% HEMC [16]	84	192	47	117
5% EVA [14]	90	200	63	141

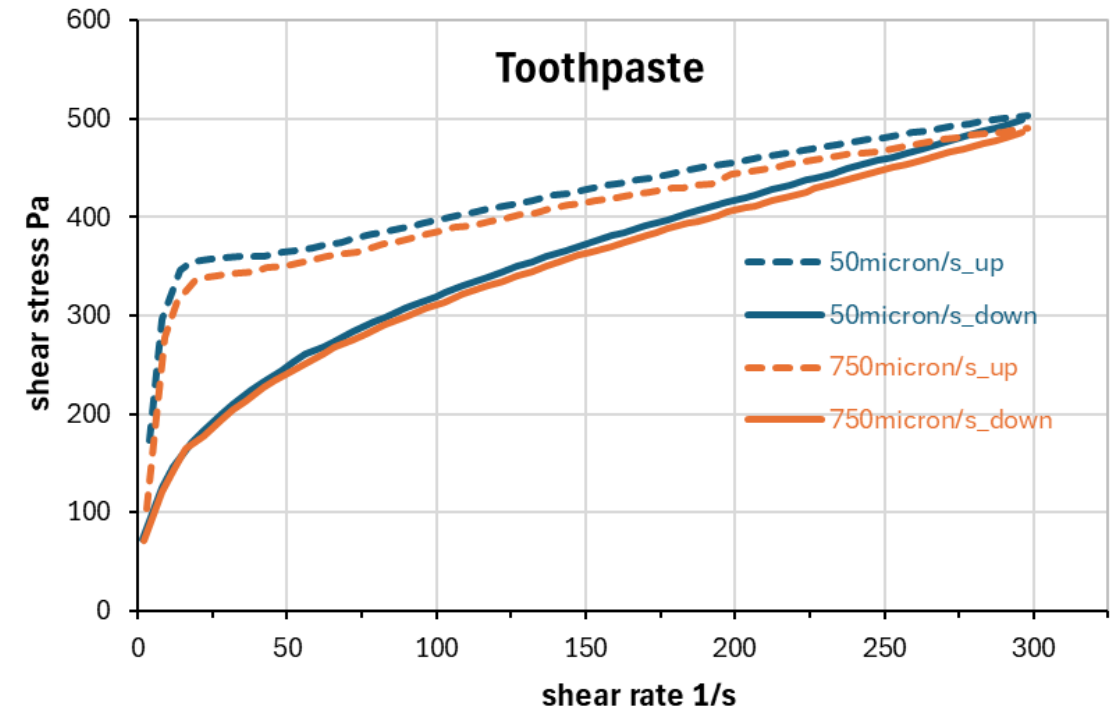
- A.M. Betioli, P.J.P. Gleize, V.M. John, R.G. Pileggi, Effect of EVA on the fresh properties of cement paste, Cem. Concr. Compos. 34 (2012) 255–260
- R.G. Pileggi, A.M. Betioli, F.A. Cardoso, V.M. John, Extended rheological characterization of cement pastes: squeeze flow plus rotational rheometry, Proc. 12th Int. Congr. Chem. Cem., Montreal — Canada, 2007

❖ This analysis suggests that the results published in both papers are not compromised qualitatively, but the differences between the rheological behaviour of the pure and of the modified pastes are probably bigger than the measured ones.

Squeeze-flow



Rotational shear-flow



- Squeeze-flow: no strain-hardening behavior = no phase separation. Higher velocity caused higher normal force = viscous effect.
- Toothpaste is a colloidal suspension with up to 2% of viscosity modifiers (thickeners) such as xantam gum, guar, CMC, HEMC, etc.
- Shear cycle: no substantial difference between the samples loaded with different velocities.

- ❑ Yield stress and plastic viscosity of the samples loaded in the automatic mode were, respectively, 30–50% and 40–65% higher than those of the paste samples squeezed at 750 $\mu\text{m/s}$.
- ❑ The gap positioning procedure should be considered when developing protocols for parallel-plate rheometry of concentrated suspensions, otherwise the results can be affected by phase separation, thus, leading to incorrect conclusions.
- ❑ Combining squeeze-flow and rotational rheometry can be useful as a more detailed rheological evaluation including phase separation likelihood under squeeze-flow and flow behavior under compression and geometric restrictions.

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Parallel-plate rotational rheometry of cement paste: Influence of the squeeze velocity during gap positioning



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ABSTRACT

This paper reports an experimental investigation regarding the influence of the squeeze velocity, during positioning of the parallel-plate gap, on the rotational shear flow behaviour of a cement paste. The descent of the upper plate was performed using diverse speeds while the normal force generated due to the compression of the paste was recorded. The slower the plate speed, the higher the resulting normal force. This behaviour was caused by liquid–solid separation, which is more likely to occur at slow squeeze velocities. Phase separation was confirmed by assessing, via microwave drying, the water contents of the trimmed portion of the paste sample and of the portion actually subjected to rotational shear cycles. Owing to the variation of water/cement ratio induced by liquid radial migration, paste's Bingham yield stress and plastic viscosity were significantly affected by squeeze speed, and both rheological parameters presented an inversely proportional relationship with this experimental variable.

Thank you for your attention!!

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