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Effect of the mixing time on the rheological parameters of cement pastes

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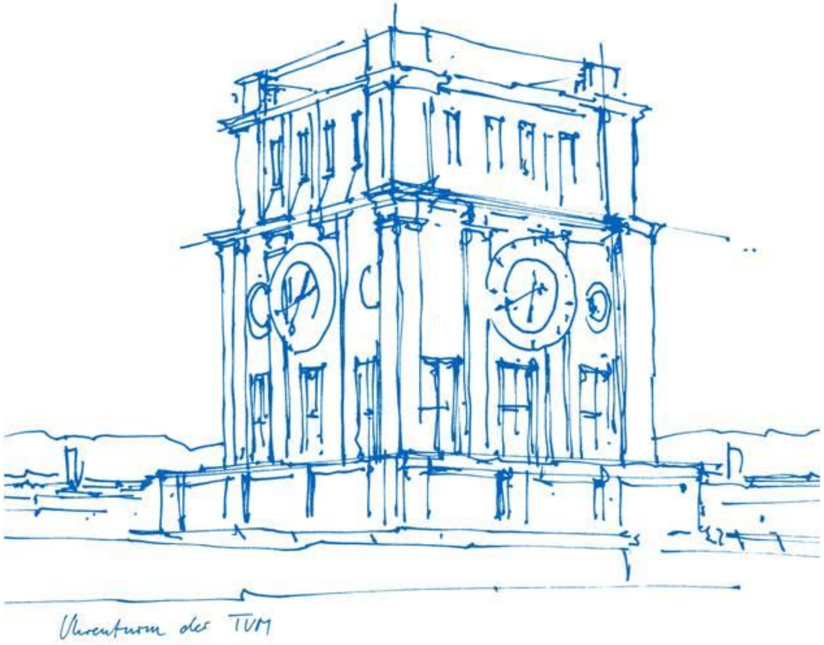
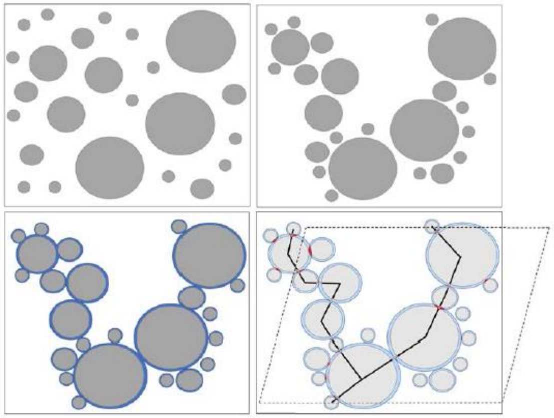


Content

- Rheology of colloidal suspensions
- Effect of the mixing time
- Experiental program
- Discussion
- Outlook

Motivation: Challenges in rheological measurements

- Different particle agglomeration and flocculation processes lead to different rheological parameters
- Change of interparticle networks due to different mixing time, energy and time for restructuring
- Strength of built networks responsible for viscosity and yield stress



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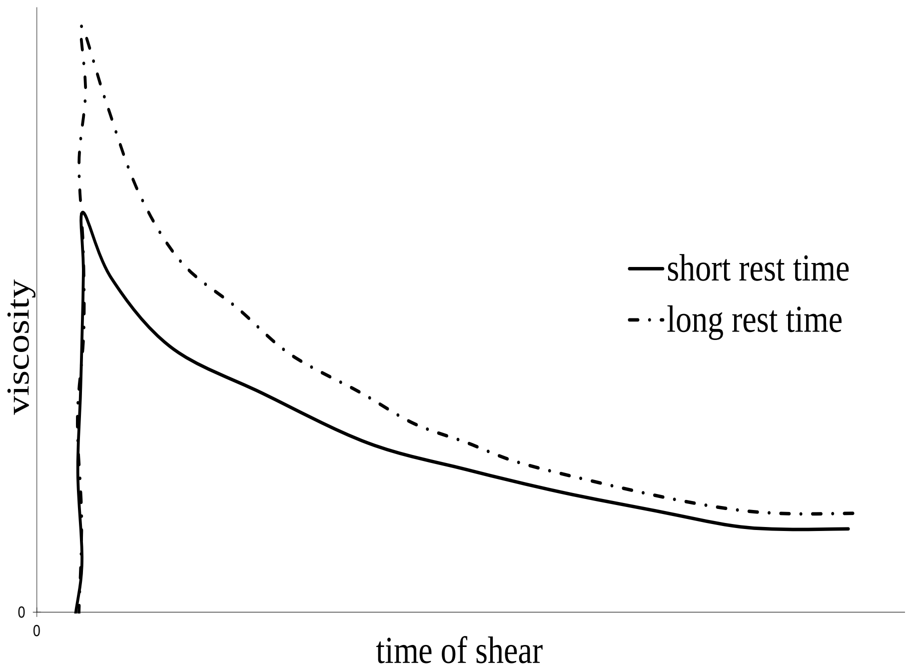
Motivation: Challenges in rheological measurements



Viscosity as a function of shearing and resting time before shear

BARNES, Howard A.: *Thixotropy—a review*. In: *Journal of Non-Newtonian Fluid Mechanics* 70 (1997), 1-2, S. 1–33

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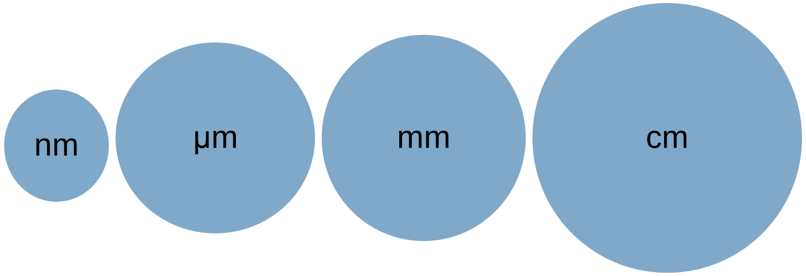
Suspension rheology

Colloidal suspension:

- Two-phase system with a liquid and a solid phase
- Solid phase: Colloids from 10^{-3} – 10^{2} µm [1]
  - ➢ Cement particles: Colloidal particles up to 10^{2} µm



[1] GENOVESE, Diego B.: *Shear rheology of hard-sphere, dispersed, and aggregated suspensions, and filler-matrix composites*. In: *Advances in colloid and interface science* 171-172 (2012), S. 1–16

Suspension rheology

Interparticle forces

- Colloidal interaction forces:
  - Brownian motion F_B
  - Van-der-Waals forces F_{VdW}
  - Electrostatic attraction F_{EA}
  - Electrostatic repulsion F_{ER}
  - Steric repulsion F_{SR}

Interaction forces:

$F_B + F_{VdW} + F_{EA} + F_{ER} + F_{SR}$

- Non-colloidal interactions
  - Hydrodynamic forces
  - Collisive forces

[1] GENOVESE, Diego B.: *Shear rheology of hard-sphere, dispersed, and aggregated suspensions, and filler-matrix composites*. In: *Advances in colloid and interface science* 171-172 (2012), S. 1–16

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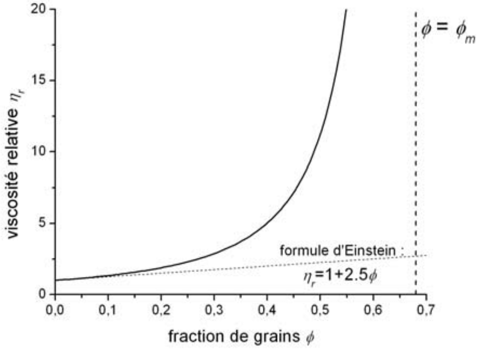
Suspension rheology

Influence parameters on rheological properties in suspensions like cement paste :

- Particle Size Distribution
- Fineness of Particles (d_{50}) (and shape) → physical particle interactions
- Chemical properties: Ion content, surface charges → chemical particle interactions
  - ➢ Agglomeration processes
- Max. Particle packing density ϕ_{max}
- Solid volume fraction; relative packing density ϕ_{rel} [1]
- Viscosity of the carrier liquid

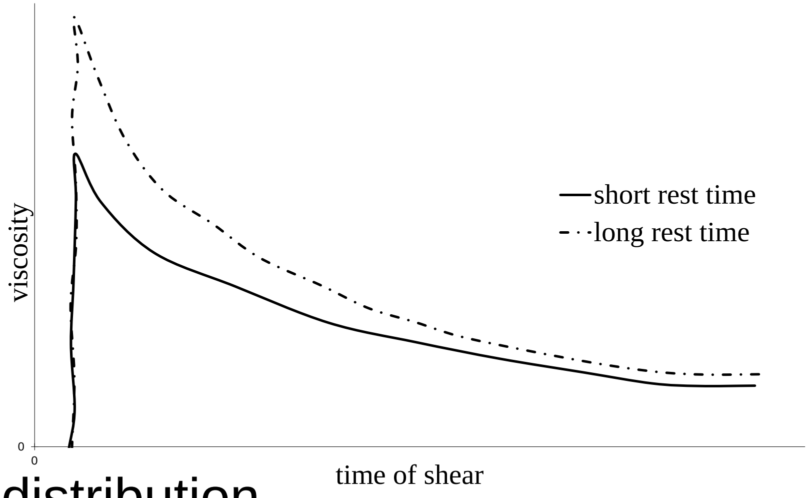


[1] Graphics: Dynamique et instabilités des interfaces grains/fluide dans les suspensions non-Browniennes, Christophe Chevalier

Rheological measurements

Influences of experimental procedures on rheological parameters

- Mixing regime: Induced shear and time to rest affect the apparent microstructure of the system



- ➢ Influence on effective particle size distribution
- ➢ With increasing agglomerates, effective d_{50} increases

Rheological measurements

Influences of experimental procedures on rheological parameters

- Most reasonable reference state of microstructure before rheological measurements should be found
- Ideal reference states: Completeley flocculated or completeley deflocculated

➢ **The effect of the mixing time on the microstructural change and thus the rheological parameters should be known**

Aim of the experimental program

- Investigation of the effect of mixing time on rheological parameters
- Estimation of a proper mixing procedure for upcoming experimental series
- Idea of agglomeration processes in colloidal suspensions

Experimental program

Used materials:

CEM I 42,5 R

Deionized water, adjusted for 20°C paste temperature

Experimental program

Mixing devices: Standard mortar mixer

Drilling machine with a propelling screw

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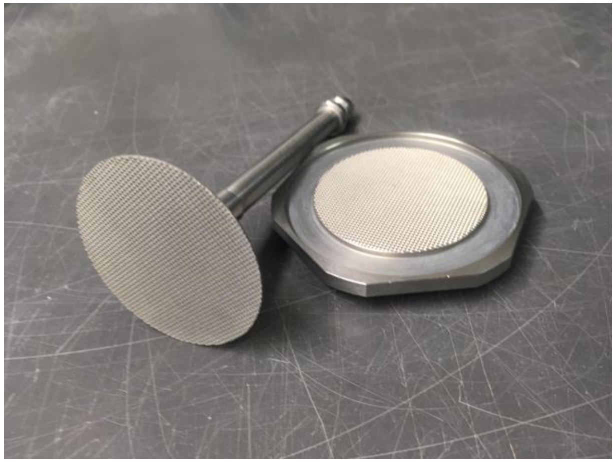
Experimental program

Measurements: Mini slump flow test acc. EN 1015-3

Rheometer – Anton Paar MCR 502

Test with serrated parallel plates

Gap: 1 mm between the plates



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Experimental program

Sample mixtures:

- Three experimental series
- Two different solid concentration and one variation with superplasticizer
- For each series, four different pre-shear times were tested

Mixture	w/c ratio	Solid concentration [-]	Cement [kg/m³]	Water [kg/m³]	PCE [wt.-% by cement]
0.42	0.42	0.434	1346.7	565.6	-
0.40	0.40	0.446	1383.9	553.6	-
0.40-SP	0.40	0.446	1383.9	551.3	0.25

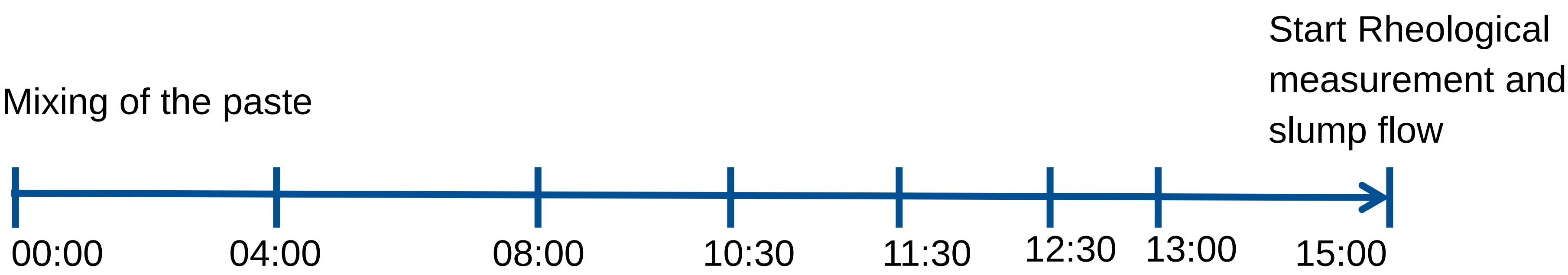
Experimental program

Mixing procedure:

- Mixing acc. To DIN EN 196 – 1 (4 min in total)
- Pre-shear for each series 30, 90, 150, 300 sec; respectively
- Time left at rest: 08:30 min (30 sec pre-shear) to 04:00 min (300 sec pre-shear)
- Slump flow measurement and rheological measurements 15:00 min after water addition

Experimental program

Mixing schedule:



08:30 min resting time I 30 sec pre-shear time

07:30 min resting time I 90 sec pre-shear time

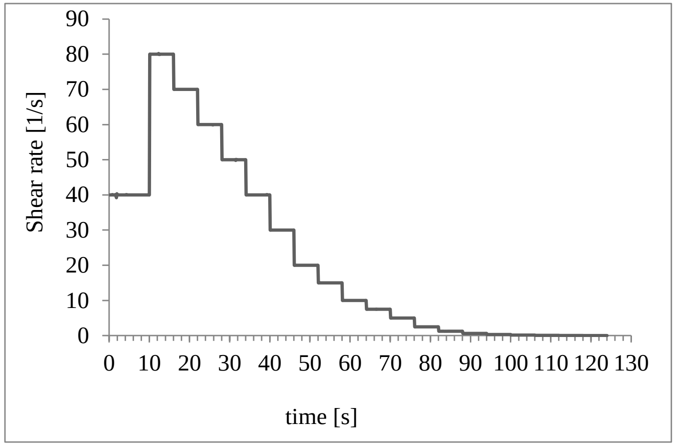
06:30 min resting time I 150 sec pre-shear time

04:00 min resting time I 300 sec pre-shear time

Experimental program

Rheological procedure:

- Pre-shear: 40 s^{-1} for structural break up
- 15 decrease steps from 80 s^{-1} to 40 s^{-1} ; duration: 6 sec
- Measurement of equilibrium torque and deformation
- Calculation of absolute parameters shear rate and shear stress
- Calculation of yield stress using the Hershel-Bulkley model
- Calculation of the Hershel-Bulkley viscosity at a shear rate of 10 s^{-1}



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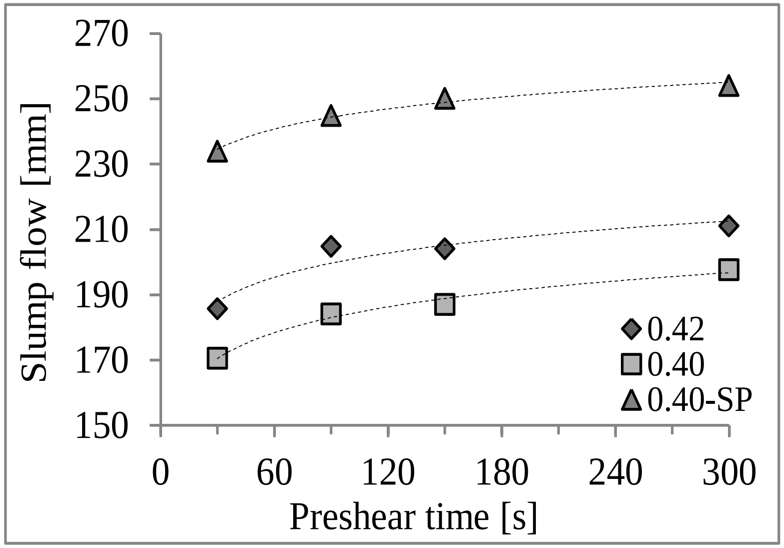
Results on slump flow



- With increasing mixing time, slump flow increases
- Increase of slump flow for different series equal
- Percentage increase of slump flow app. 16%

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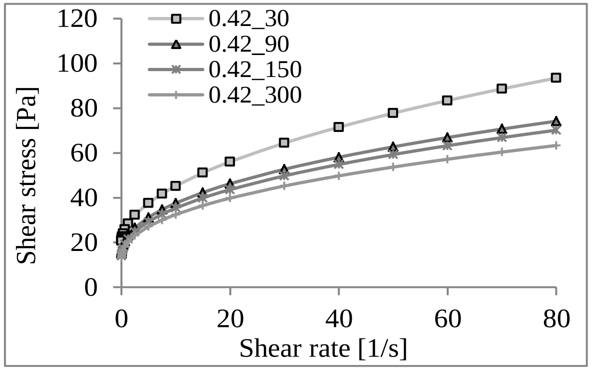
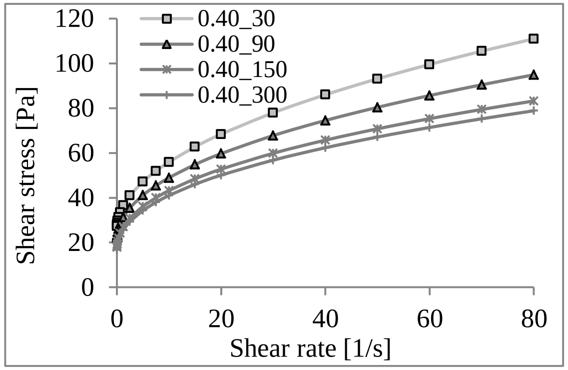
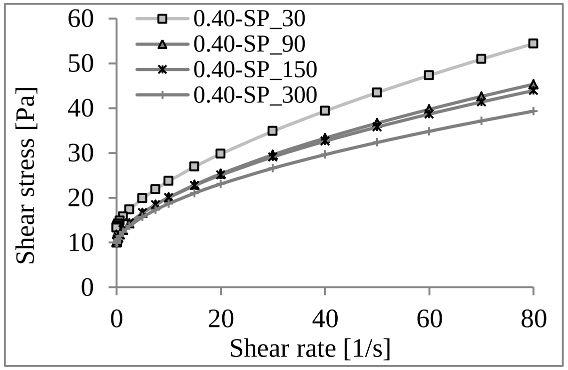
Results: Yield stress







- For each series, flow curves change with increasing pre-shear time
- The percentage difference between the different pre-shear times is similar for each series.
- Yield stress decreases with increasing pre-shear
- Decrease of percentage yield stress ranges from 33% (w/c = 0.42) to 40% (w/c = 0.40 and 0.40 – SP)

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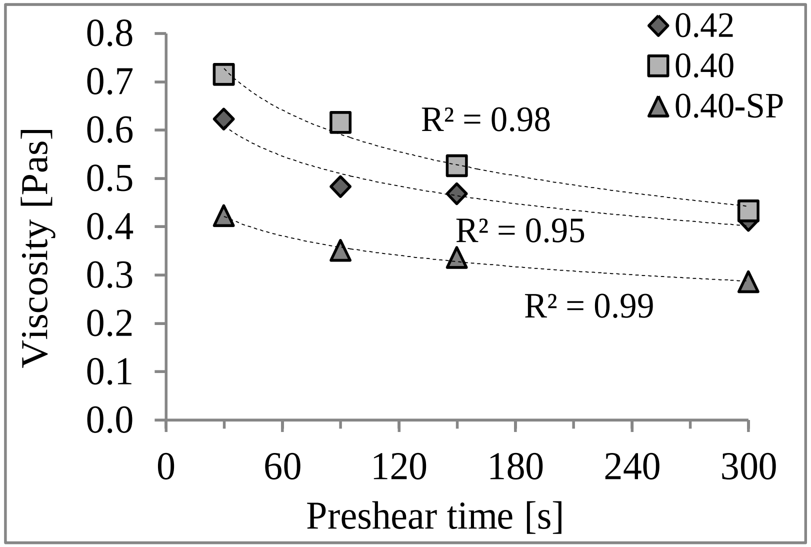
Results: Viscosity



- Decrease of viscosity with increasing pre-shear
- Most striking decrease in viscosity reached after 90 sec
- Equilibrium state can be assumed after 300 sec

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Conclusions

- With increasing shear time, yield stress and viscosity decrease due to a higher deflocculated state
- Results show good correlation to already existing theories regarding particle agglomeration
- Choice of preshear is crucial for measured rheological parameters
  - ➢ Most appropriate reference state should be clarified for rheological measurements

Conclusions

Former aims:

- Effect of mixing time was investigated, see results on the former slides
- An equilibrium state could be found after 300 sec. This is not appropriate for mixing procedures.
- No appropriate agglomeration investigation was possible. This is work for prospective research.

Outlook

- More detailed investigations regarding agglomeration status should be performed
- State of deflocculation has a decisive effect on structural build up
  - ➢ Dependence of thixotropy on preshear time is currently being investigated
- Use of the knowledge about particle agglomeration for the prediction of yield stress and viscosity on granulometry parameters

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Acknowledgements

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