Institute for Building Materials Science

Leibniz Universität Hannover

Rheometric method for the quantification of micro sedimentation phenomena in cement suspensions

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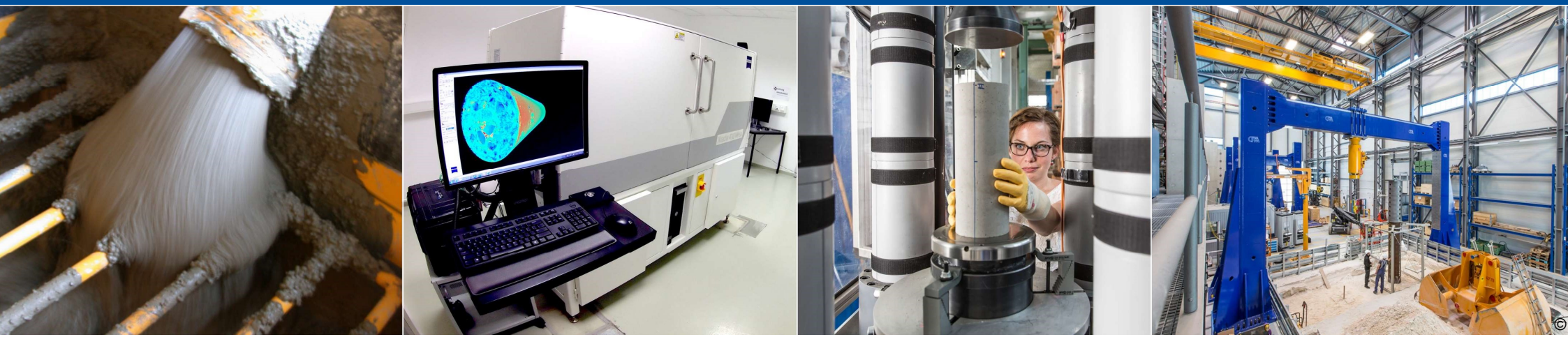
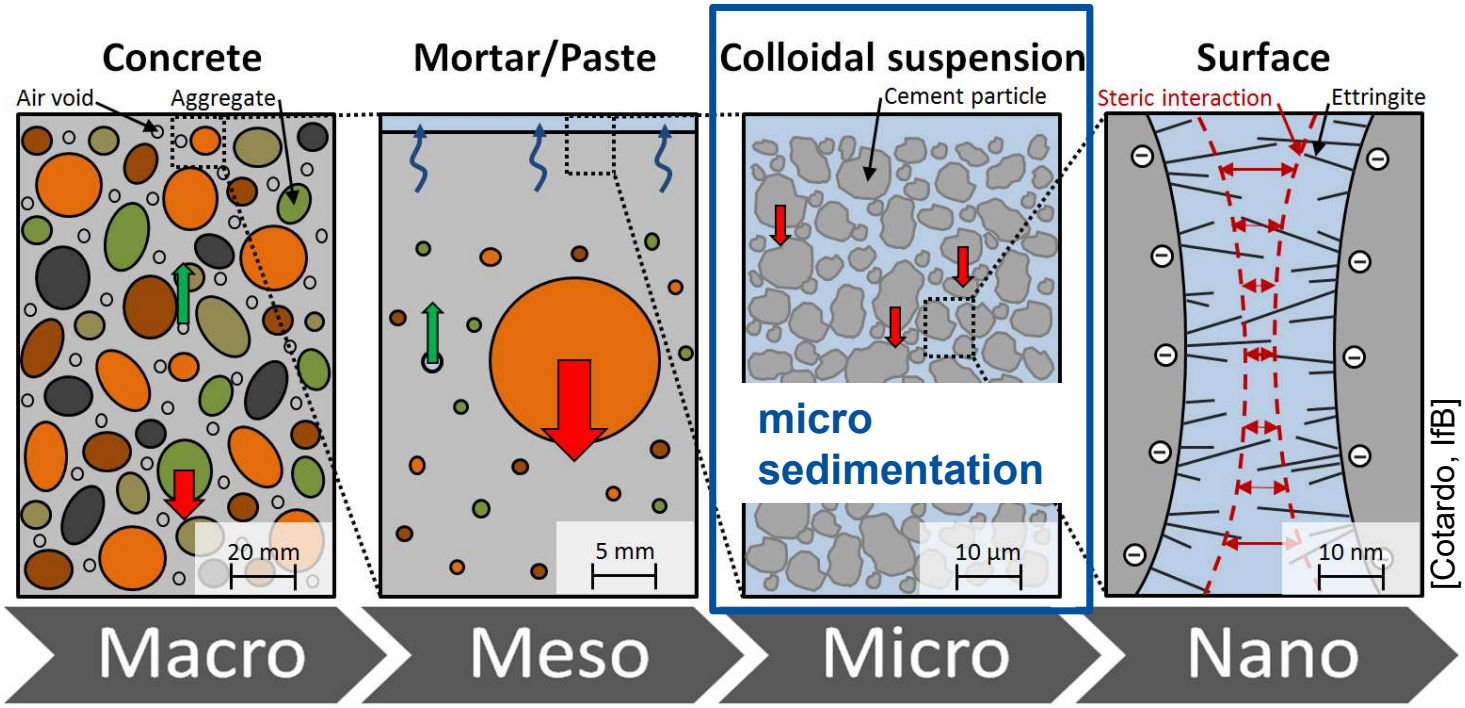


Motivation



The microstructure influences segregation phenomena of concrete.

Micro sedimentation always occurs.

Properties vary over the height.

Aim of investigations

The aim was to quantify the effect of micro sedimentation phenomena in cement suspensions by rheological measurements.

Basic requirements of rheological measurments:

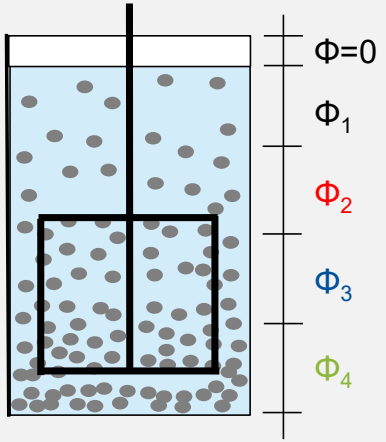
- laminar flow
- steady state flow
- wall adhesion
- **homogeneity** of the sample

➡ **Inhomogeneities** can be porpuseful used in order to measure micro sedimentation phenomena.

➡ In order to derive the relationship between micro sedimentation and changes in properties, the quantification of the rheological parameters is useful.

Rheological measurement

$\tau = f(\Phi)$

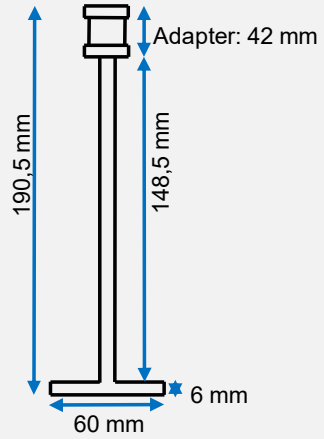


$\Phi_1 < \Phi_2 < \Phi_3 < \Phi_4$

The geometry of the measuring paddle prevents inhomogeneities.

Approach

Adaption of geometry

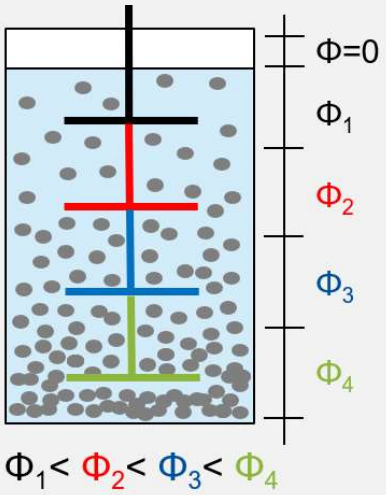


Geometry of measuring paddle: inverted „T“

- enables measurement on a defined sample height
- no mixing effect
- complete sample diameter is sheared

Defined measurements

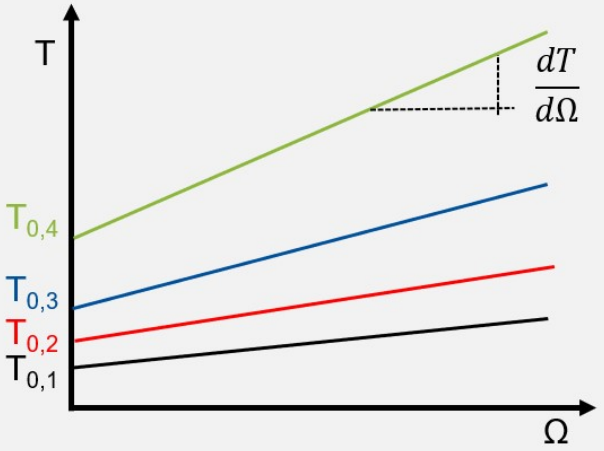
$\tau = f(\Phi)$



$\Phi_1 < \Phi_2 < \Phi_3 < \Phi_4$

- significant number of positions depending on the sample height
- definition of positions

Quantification



- representation of flow curves
- determination of yield torque and slope of flow curve

Description of the measurement method

Experimental set-up of sedimentation-dependent rheological measurements

- Thermo Scientific™ HAAKE™ MARS™ 60 rheometer
  - SEARL-type rheometer for low shear conditions
- circulation thermostat (Temp. 20°C)
- measuring cell for building materials
- self-engineered T-stirrer + universal adapter

- the sample was filled into the measuring cell after homogenization (13 min p. init. water-cement contact)
- covered with a glass plate between the measurments in order to prevent water evaporation

[von Bronk, IfB]

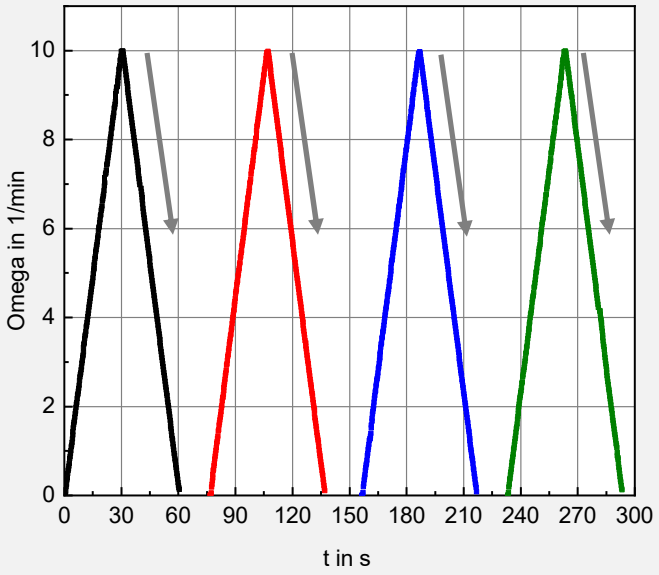
Description of the measurement method

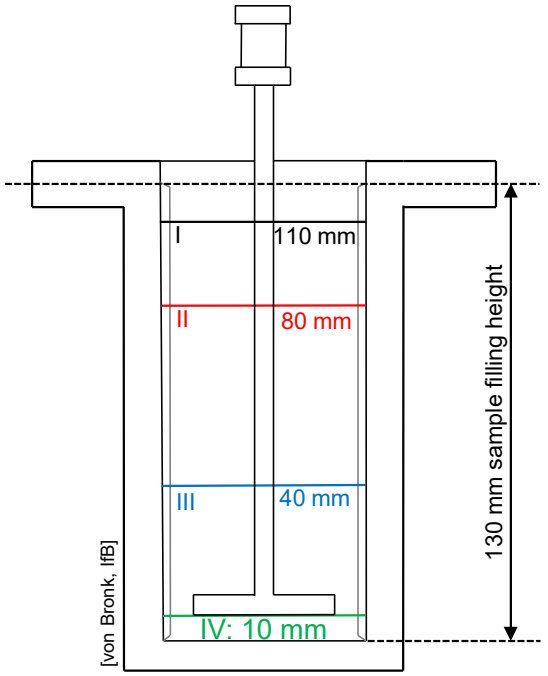
Experimental set-up of sedimentation-dependent rheological measurements

measurement job:

- four measuring heights (I-IV): 110, 80, 40 and 10 mm
- four measuring times: 15, 45, 90 and 180 min

downward ramp used for data evaluation

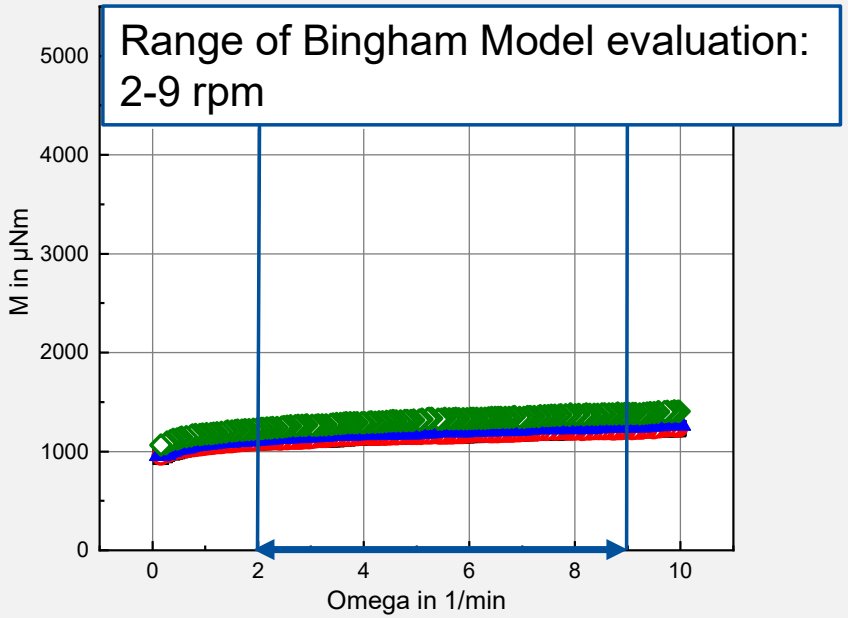




Rheological sedimentation behavior: flow curves

HeidelbergerCement OPC/ CEM I 42,5 R (20°C), demineralised water (20°C): w/c-ratio=0.50

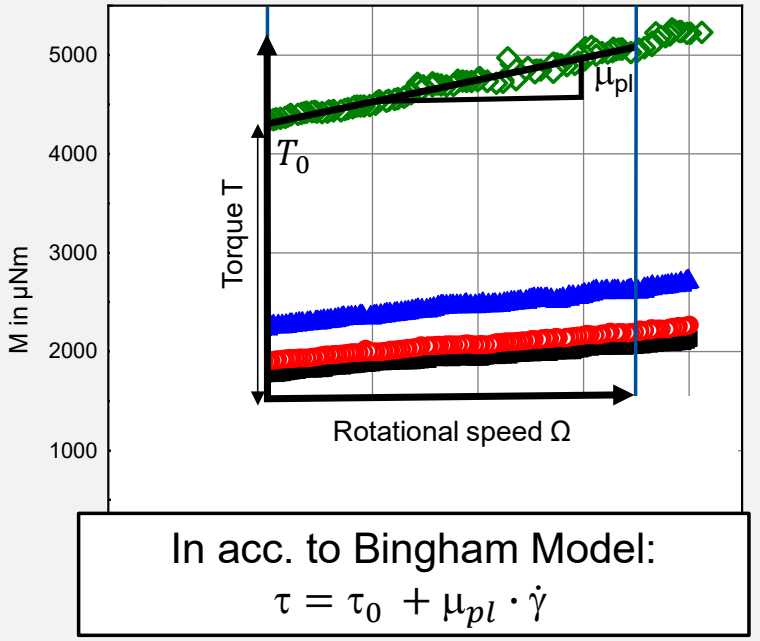
15 min after initial water-cement contact



- the torque increases with decreasing measuring height

Depending on the sample height, the sample does not show the same rheological behavior. Is the composition the same?

90 min after initial water-cement contact



In acc. to Bingham Model:

$$\tau = \tau_0 + \mu_{pl} \cdot \dot{\gamma}$$

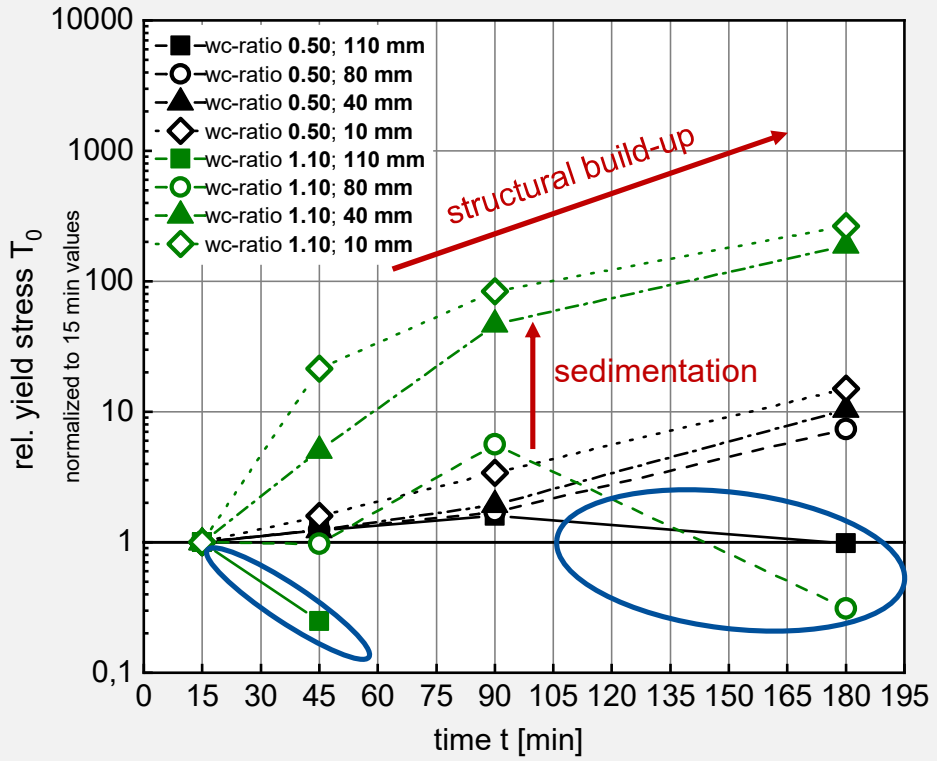
- the torque and the slope of the flow curves increases with increasing time

Two overlapping effects: ***sedimentation and structural build-up***

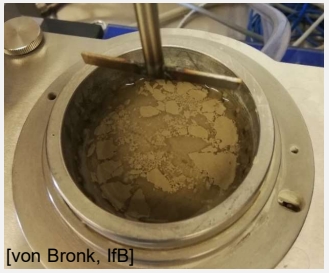
Rheological sedimentation behavior: yield stress

HeidelbergerCement OPC/ CEM I 42,5 R (20°C), demineralized water (20°C): w/c-ratio=0.50, w/c-ratio=1.10

Rel. yield stress T_0 normalized to 15 min values

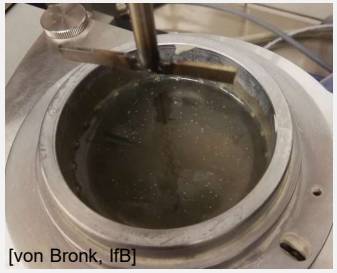


- time-dependent increase in the rel. yield stress T_0 (structural build-up)
- time-dependent increase in the difference ot the measured rel. yield stress T_0 depending on the sample height (sedimentation)
- decrease of the rel. yield stress T_0 in the upper part of the sample due to bleeding phenomena
  - measurement is partly or completely in the bleeding water layer



sample (0.50) after 178 min

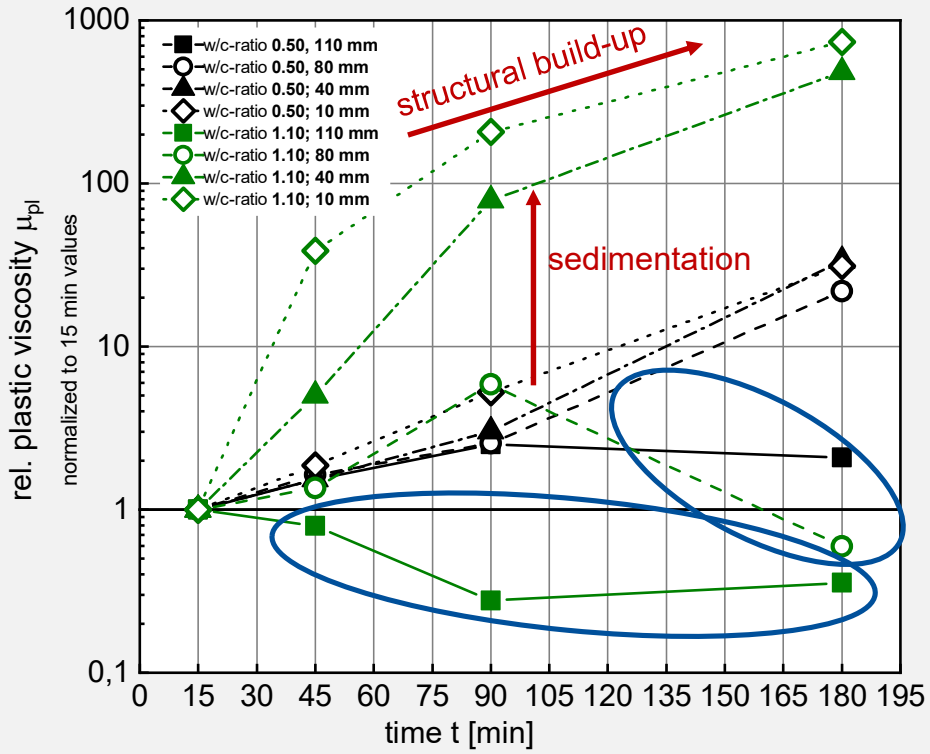
sample (1.20) after 178 min



Rheological sedimentation behavior: viscosity

HeidelbergerCement OPC/ CEM I 42,5 R (20°C), demineralized water (20°C): w/c-ratio=0.50, w/c-ratio=1.10

Rel. plastic viscosity μ_{pl} normalized to 15 min values

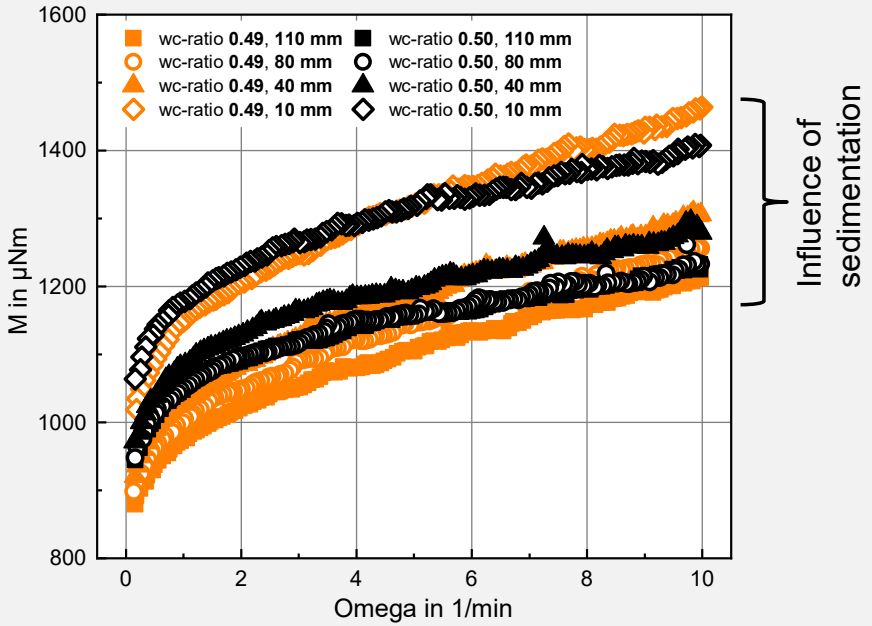


- time-dependent increase in rel. plastic viscosity μ_{pl} (structural build-up)
- difference in rel. plastic viscosity μ_{pl} depending on the sample height
  - sample w/c-ratio 0.50: slight sedimentation
  - sample w/c-ratio 1.10: great sedimentation
- decrease in rel. plastic viscosity μ_{pl} in the upper part of the sample due to bleeding phenomena
  - measurement is partly or completely in the bleeding water layer

Rheological sedimentation behavior: time effect

HeidelbergerCement OPC/ CEM I 42,5 R (20°C), demineralised water (20°C): w/c-ratio 0.50, w/c-ratio 0.49

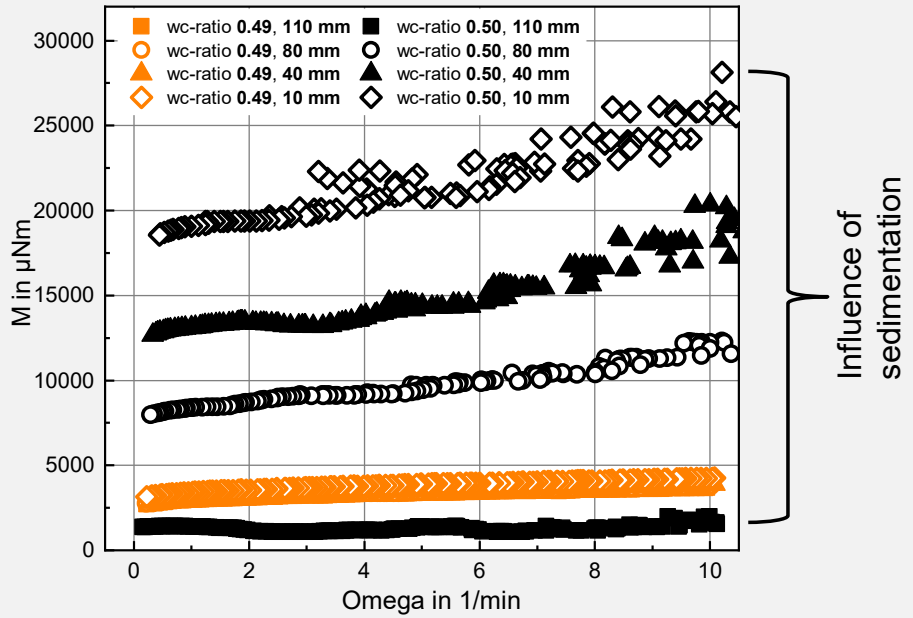
15 min after initial water-cement contact



homogenization of samples: 13 min p. init. water-cement contact

- correlation of the flow curves of the considered samples
- slight sedimentation phenomena visible in both samples (comparable)

180 min after initial water-cement contact

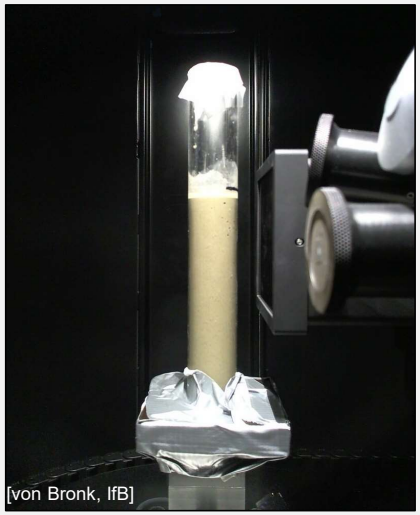


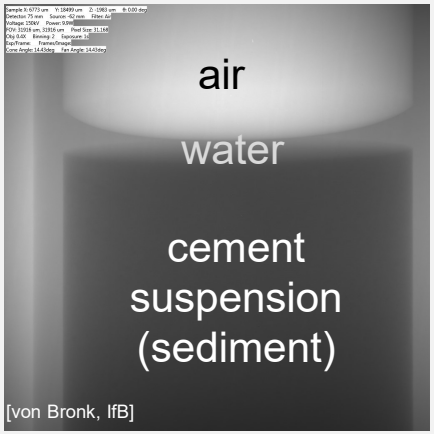
homogenization of sample 0.49: 178 min p. init. water-cement contact

- torque increase for both samples due to structural build-up
- significant time-dependent **micro sedimentation effect** for non-homogenized sample (w/c-ratio 0.50)

X-ray imaging of sedimentation

experimental set-up

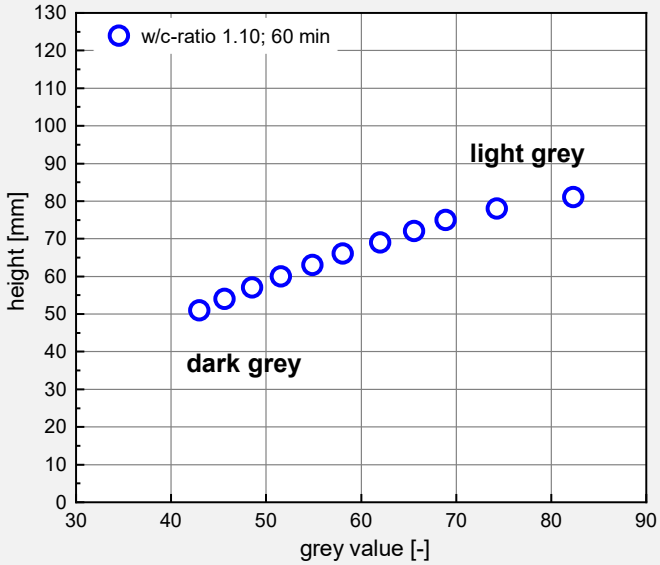

[von Bronk, IfB]



[von Bronk, IfB]

- x-ray microscope ZEISS Xradia 410 Versa
- acrylic glass (26 mm inside diameter) cylinder with formwork panel foot, sealed with silicone
- 130 mm sample filling height
- sample covered with Parafilm®

grayscale evaluation with Image J



- good correlation of rheological sedimentation to grey value gradient over height (as an indication for density (w/c-ratio))
- open issues:
  - detection of the total sample height
  - simplification of the measurement method

Conclusions

Conclusions

- micro sedimentation always takes place in cementitious suspensions
- inhomogeneities are unavoidable in cementitious suspensions and should be considered for rheological measurements
- micro sedimentation is measurable and quantifiable by rheological measurements
- x-ray method seems to be useful for the detection of micro sedimentation
- further development and simplification of the X-ray method for detecting micro sedimentation
  - to provide evidence that the difference in rheological properties over height is due to the change in composition

Acknowledgement

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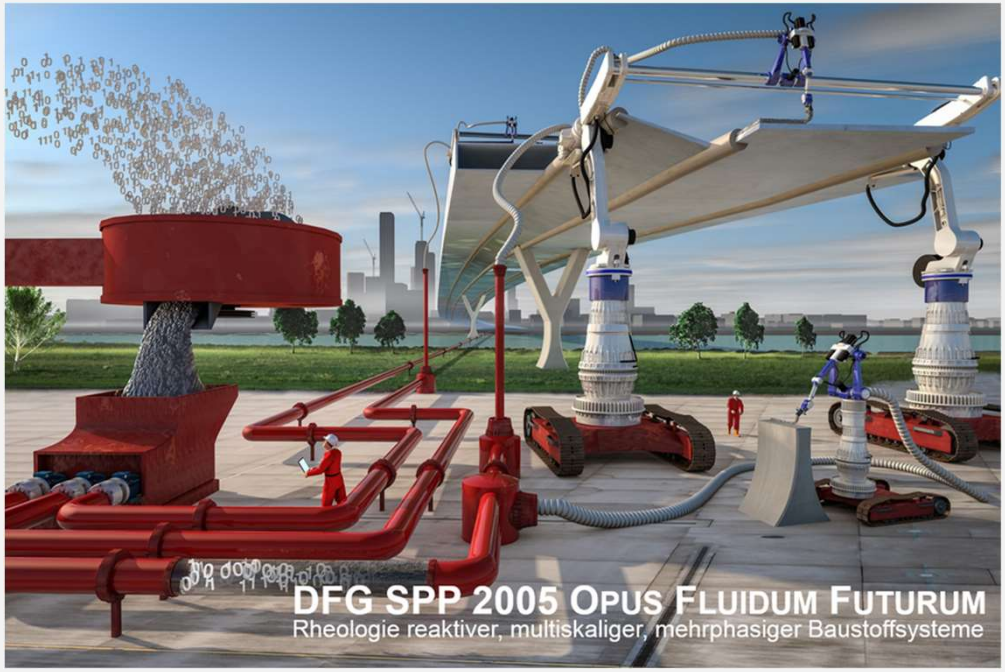

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audience

Thank you for your attention!