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SLOVENIAN NATIONAL BUILDING AND CIVIL ENGINEERING INSTITUTE

Challenges of oscillatory measurements - upgrade of classical rotational measurements to cementitious materials

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Rheology of Building Materials
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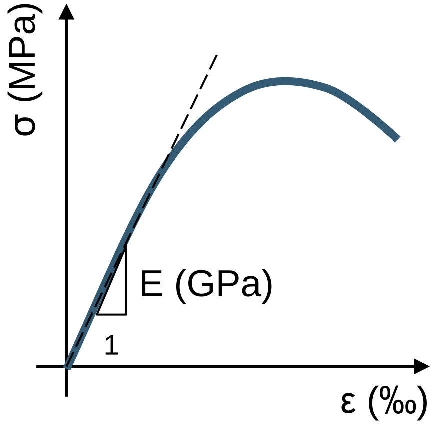
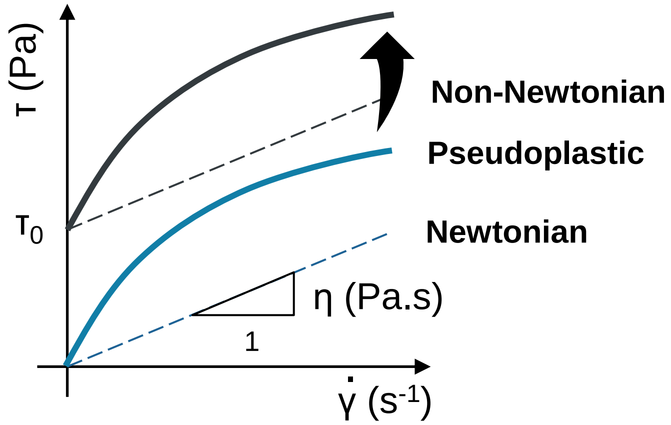




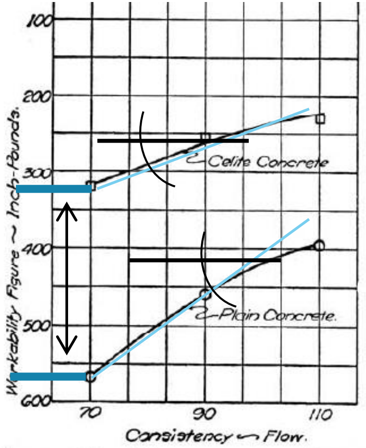
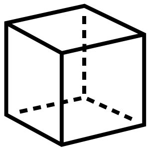


FIGURE 10.—WORKABILITY OF PLAIN AND DIATOMACEOUS CONCRETES

Calvert, R. **1930**. Diatomaceous earth. Journal of chemical education, Vol. 7, No. 12.

solid
(hardened concrete)



fluid
(water)

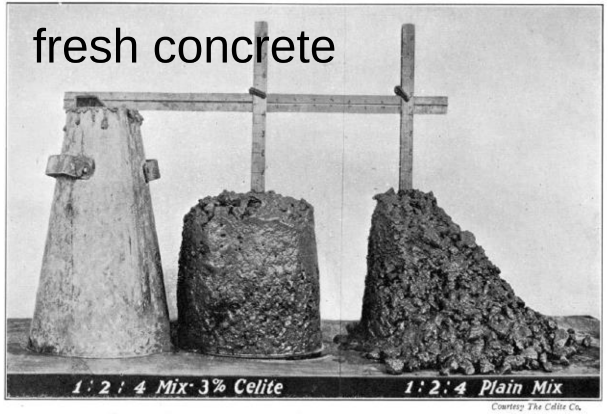
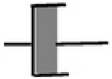


FIGURE 9.—EFFECT OF DIATOMACEOUS EARTH ON THE WORKABILITY OF CONCRETE

E

η



ELASTICITY

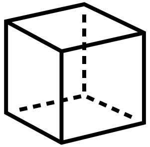
VISCOSITY

VISCOELASTICITY

VISCOELASTICITY

what

- elastic (solid-like) behaviour
- viscous (fluid-like) behaviour



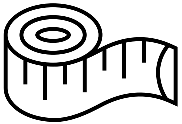
vs.

why

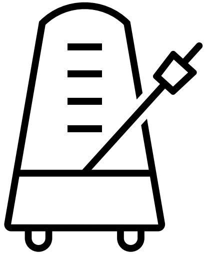
- decreased workability
- better stability
- shape retention
- degree of flucculation of particles,
- impact of admixtures (mineral or chemical)

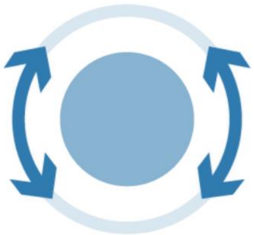
how

- small shear deformations (linear range)
- non-destructive testing (linear vicoelastic response):
  - dynamic tests (**oscillatory shear**)
  - static tests (creep-recovery)



OSCILLATORY SHEAR





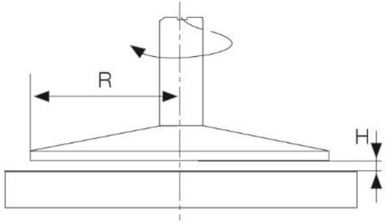
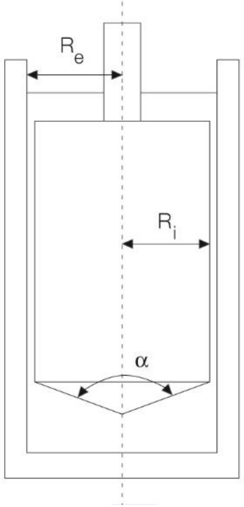
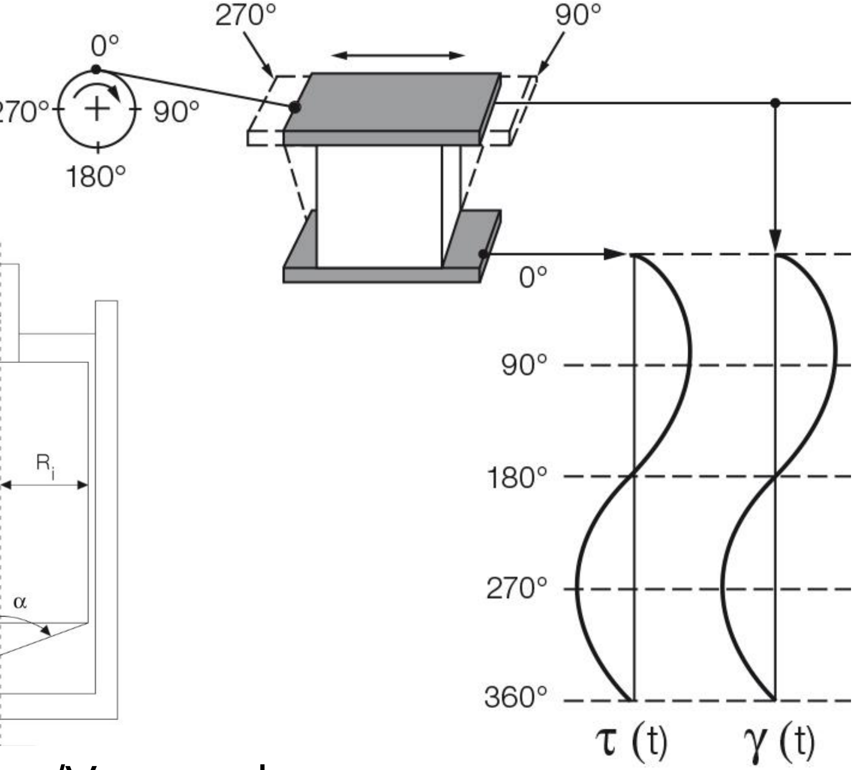


Plate-plate measuring system



Coaxial cylinders/Vane probe measuring system



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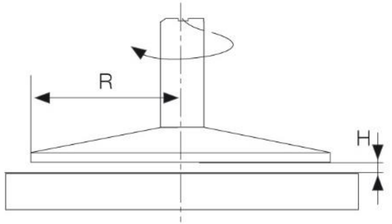

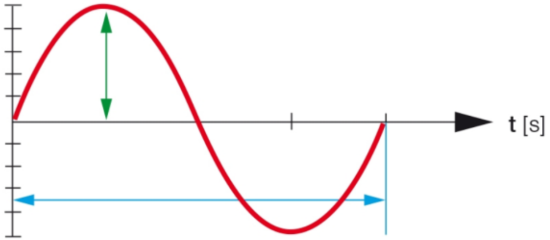

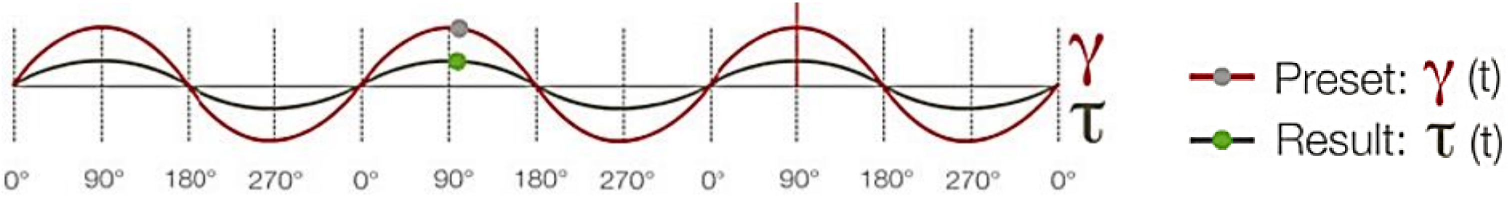

shear strain
shear stress

- Sample is sheared while compressed between two plates (upper plate oscillating)
- Constant rotational speed – constant oscillating frequency
- The deflection path of the upper plate is measured and evaluated as strain
- Parameters for oscillatory tests are usually preset in the form of a sine curve
    - defined by its **amplitude – maximum deflection** and its
    - **oscillatory period**

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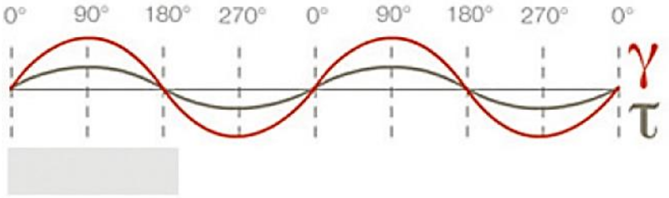
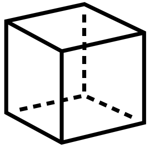
For the solid, gel-like state, δ is between 0° and 45°: i.e., **45° > δ ≥ 0°.** In this case, the material at rest is solid, such as pastes, gels, or other stiff, solid matter.

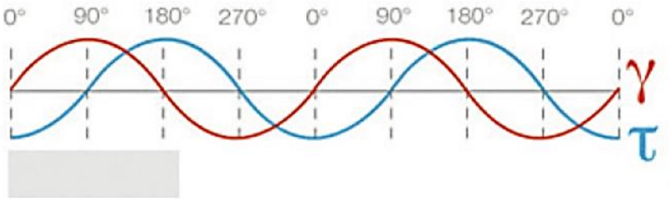


δ = 0

Ideally elastic (solid-like) behaviour



PHASE SHIFT δ



Ideally viscous (fluid-like) behaviour

δ = 90°

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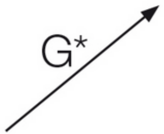
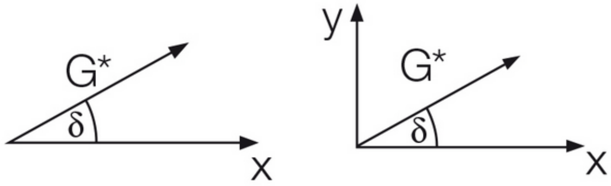


$$G^* = \tau_A \, / \, \gamma_A$$

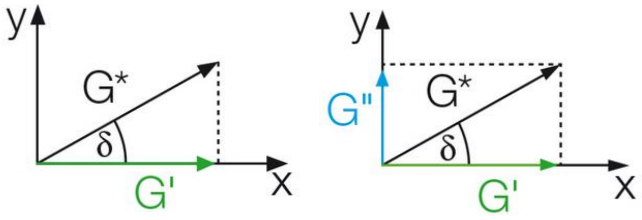
Complex shear modulus G* (Pa)

- ratio between shear-stress amplitude and strain amplitude



Phase shift δ

- time lag between the preset and the resulting sinusoidal oscillation



The part of the G* value that runs along the x-axis is the **elastic portion G'**, the part of the G* vector that is projected onto the y-axis is the **viscous portion G"**.

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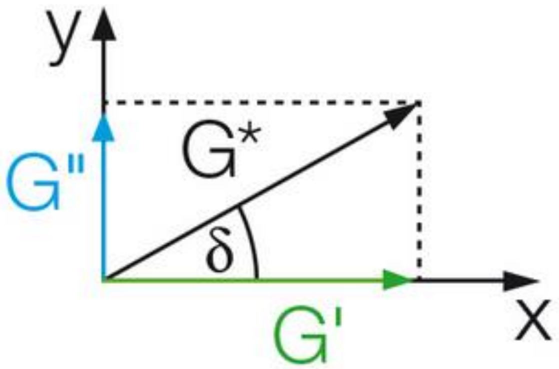

G‘ – STORAGE MODULUS
G“ – LOSS MODULUS

Storage modulus G' represents the stored deformation energy (within deformed material) and **loss modulus G''** characterizes the deformation energy lost (dissipated) through internal friction when flowing.

Viscoelastic solids with **G' > G''** have a higher storage modulus than loss modulus due to links inside the material, for example chemical bonds or physical-chemical interactions. For ideally elastic behavior δ = 0°. G'' = 0 and tan δ = G''/ G' = 0.

Viscoelastic liquids with **G'' > G'** have a higher loss modulus than storage modulus and there are no such strong bonds between the individual molecules. For ideally viscous behavior δ = 90°. G' = 0 and tan δ = G''/ G‘ = ∞.

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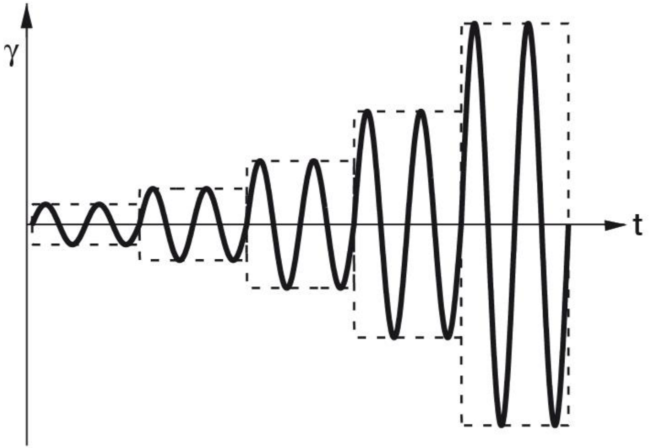

APLITUDE SWEEPs aim at describing the deformation behavior of samples in the **non-destructive deformation range** and at determining the **upper limit of this range**. Often, we use them to characterize behavior that occurs if this upper limit is exceeded with increasing deformation, when the inner structure

- gets softer,
- **starts to flow**, or
- breaks down in a brittle way.

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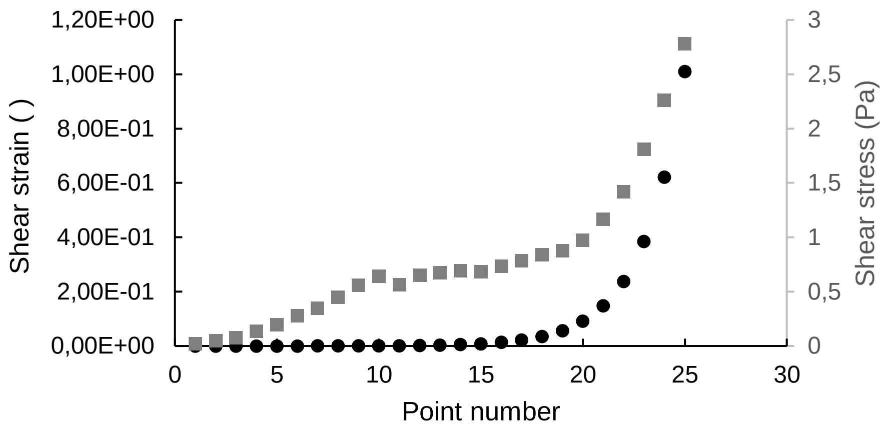
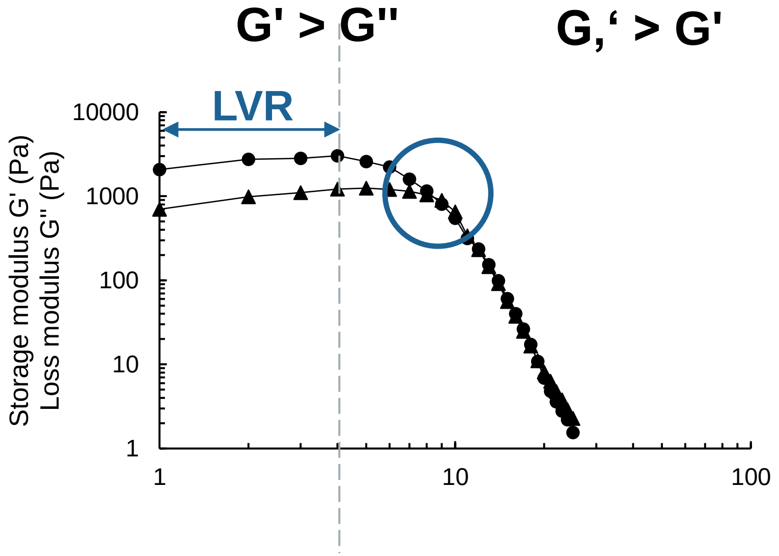
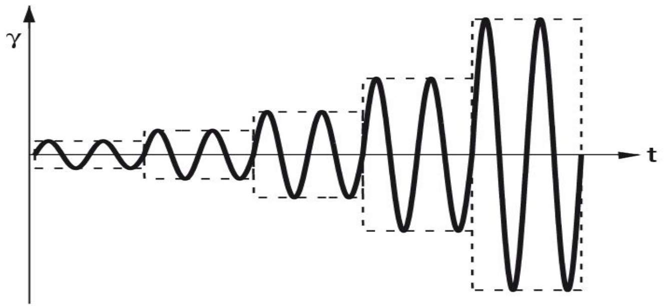
OSCILLATORY SHEAR - examples



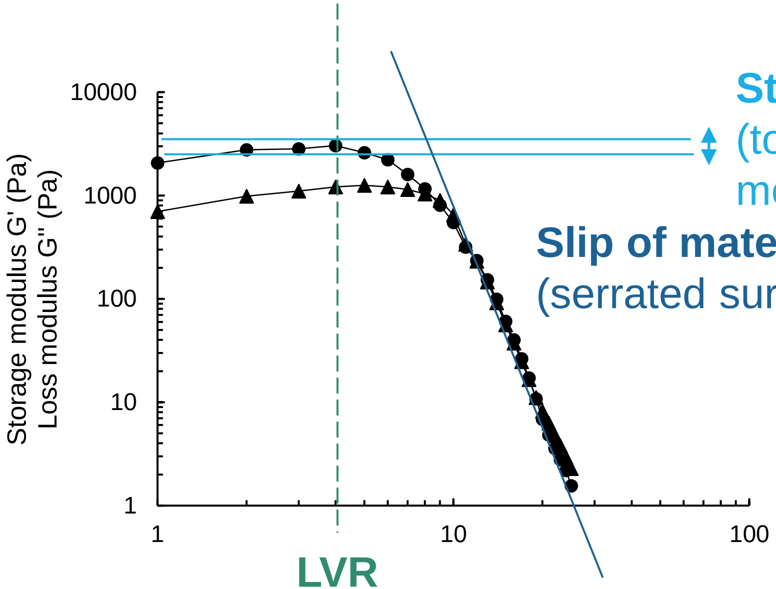




LVR = Linear viscosity range

CEMENT PASTE
Plate-plate geometry

OSCILLATORY SHEAR - examples



Structural build-up
(too short rest phase before perfromance of measurements)

Slip of material
(serrated surface for cementitious materials)

LVR
(not always possible to assess)

General reccomendations

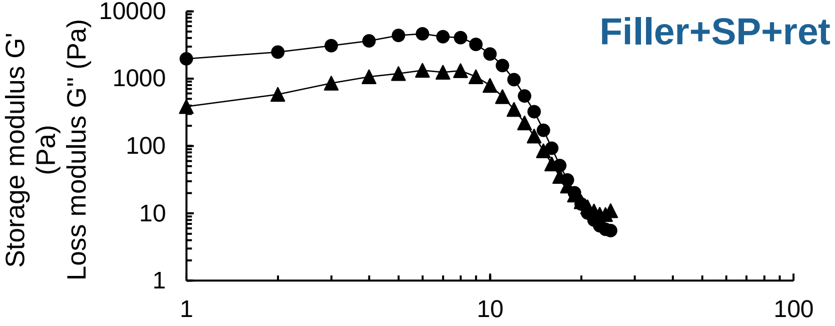
- **pre-shearing** included (really high shear rates)
- **rest time** neccessary (has to be determined individually – based on the packing density of the sample)

CEMENT PASTE
Plate-plate geometry

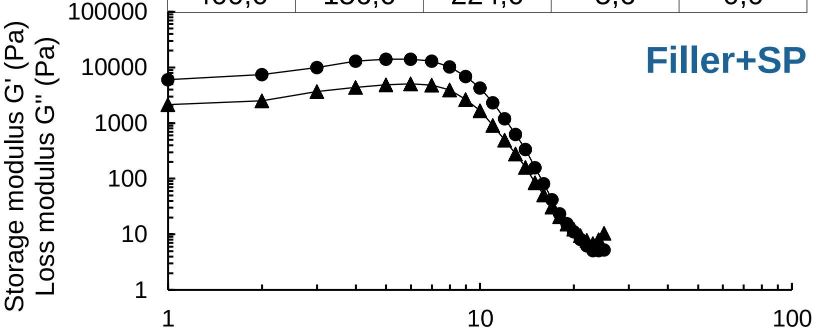
OSCILLATORY SHEAR - examples

CEMENT PASTE
Plate-plate geometry

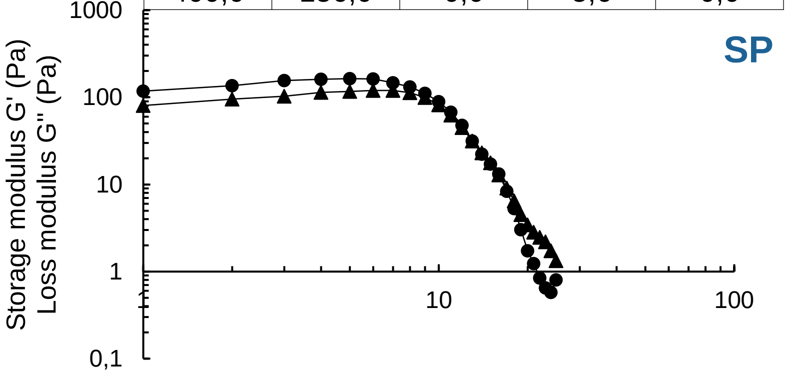
CEM I	water	filler	SP	retard
400,0	156,0	224,0	5,6	4,0



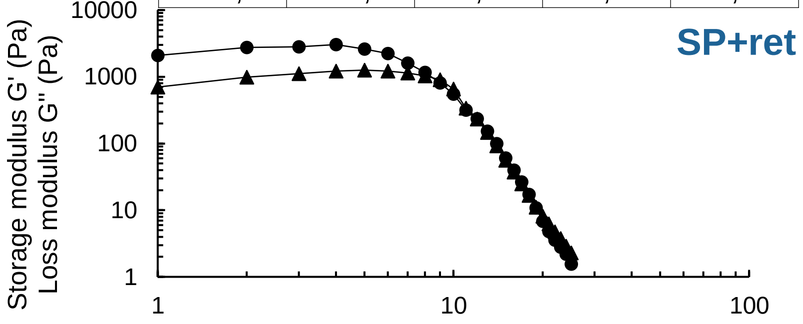
CEM I	water	filler	SP	retard
400,0	156,0	224,0	5,6	0,0



CEM I	water	filler	SP	retard
400,0	156,0	0,0	5,6	0,0

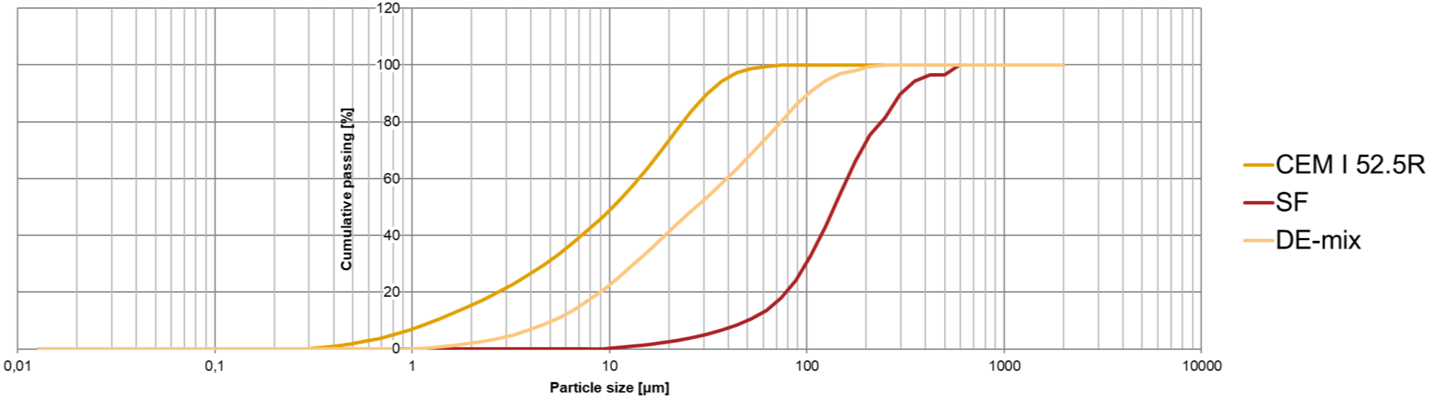


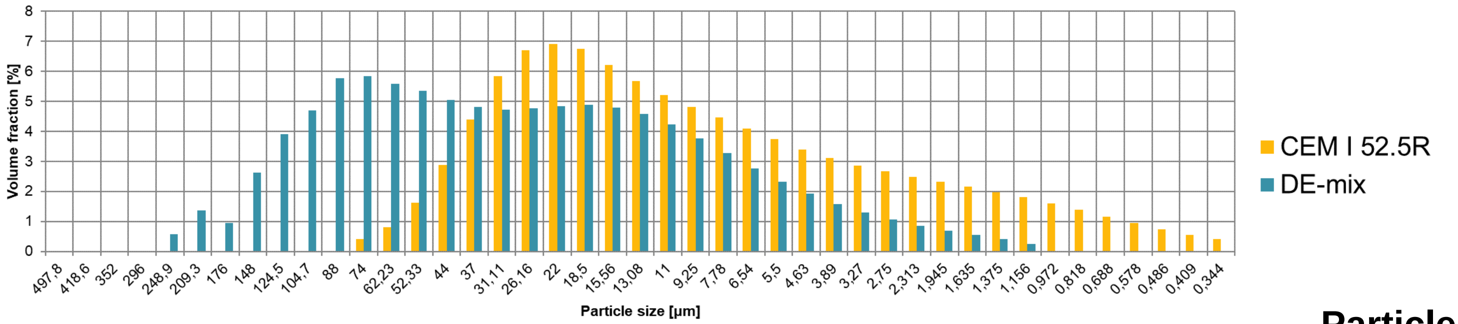
CEM I	water	filler	SP	retard
400,0	156,0	0,0	5,6	4,0



OSCILLATORY SHEAR - examples

CEMENT PASTE
Plate-plate geometry

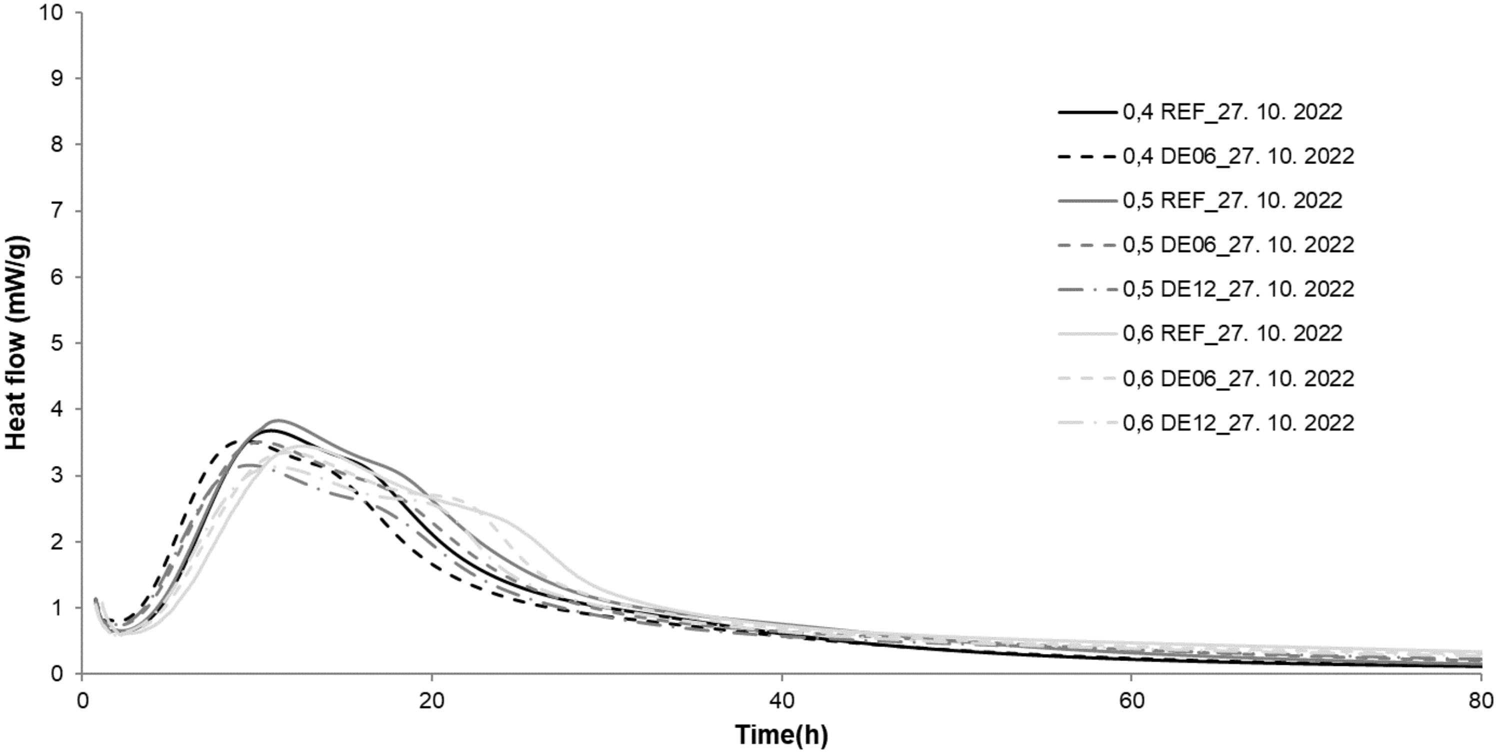




Particle size distribution

OSCILLATORY SHEAR - examples

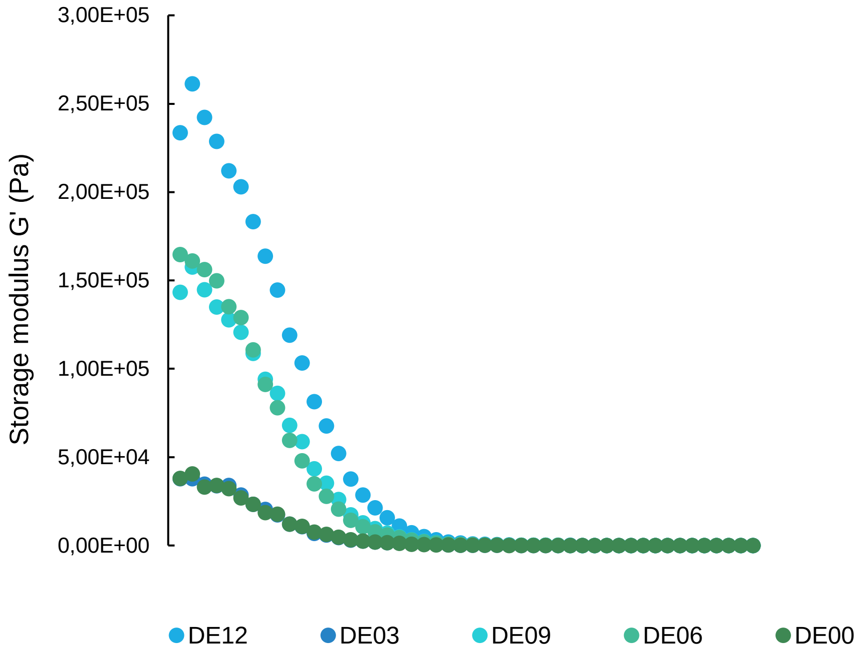
CEMENT PASTE
Plate-plate geometry

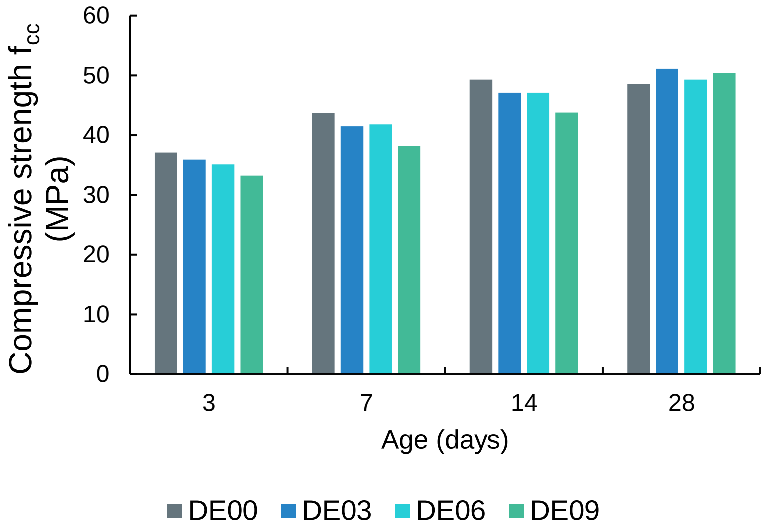


Isothermal calorimetry

OSCILLATORY SHEAR - examples

CEMENT PASTE
Plate-plate geometry





Small amplitude oscillatory shear (SAOS)
Compressive strength - maturity

Brunčič, Ana

CONCLUSIONS

1) Small amplitude oscillatory shear (SAOS) is a usefull measuring technique also for assessment of cementitious materials;
2) SAOS can help determine stability as well as thixotropy of a cementitous material (shear strain/stress causing behaviour transition), it is also useful tool for asessing impact of additions/admixtures;
3) SAOS, performed on cementitious system, can be correlated with macroscopical properties of concrete.

Aknowledgment

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Thank you for your attention.