Adhesive mortars properties: Squeeze Flow and Contact Visualization

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Outline

- Context
- Objective
- Previous Research Summary
  - MRI
  - Interfacial rheology
- Methods and Results
  - Squeeze flow
  - Microtomography
  - Optical microscopy
- Conclusion

Context

Adhesive mortars

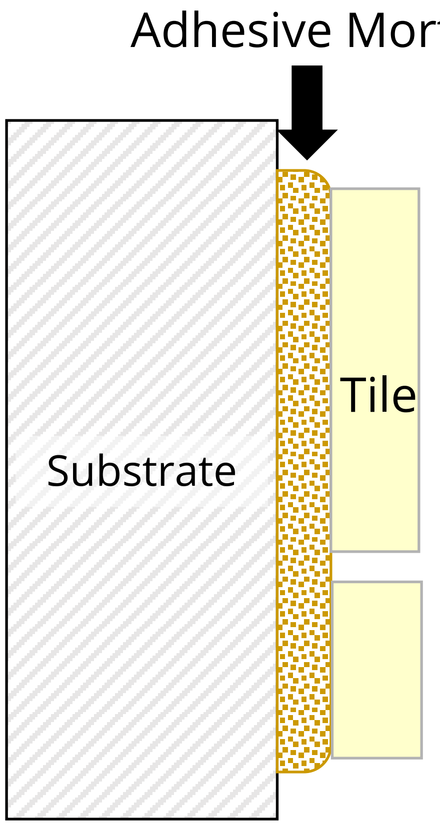
Facades

Swimming Pools

Bathrooms

Context

Adhesive mortars



To accomplish a good performance different fresh state properties are required

Context

System application steps:

1. **Mortar application to a substrate**

   Spreading: Easiness to apply - good squeeze flow properties

   Plasticity: Form clear ribs when troweled with a toothed comb

   Water retention: Retaining water from the substrate

2. **Tile Placement**

   Easiness to squeeze the ribs: The tiles can be easily placed

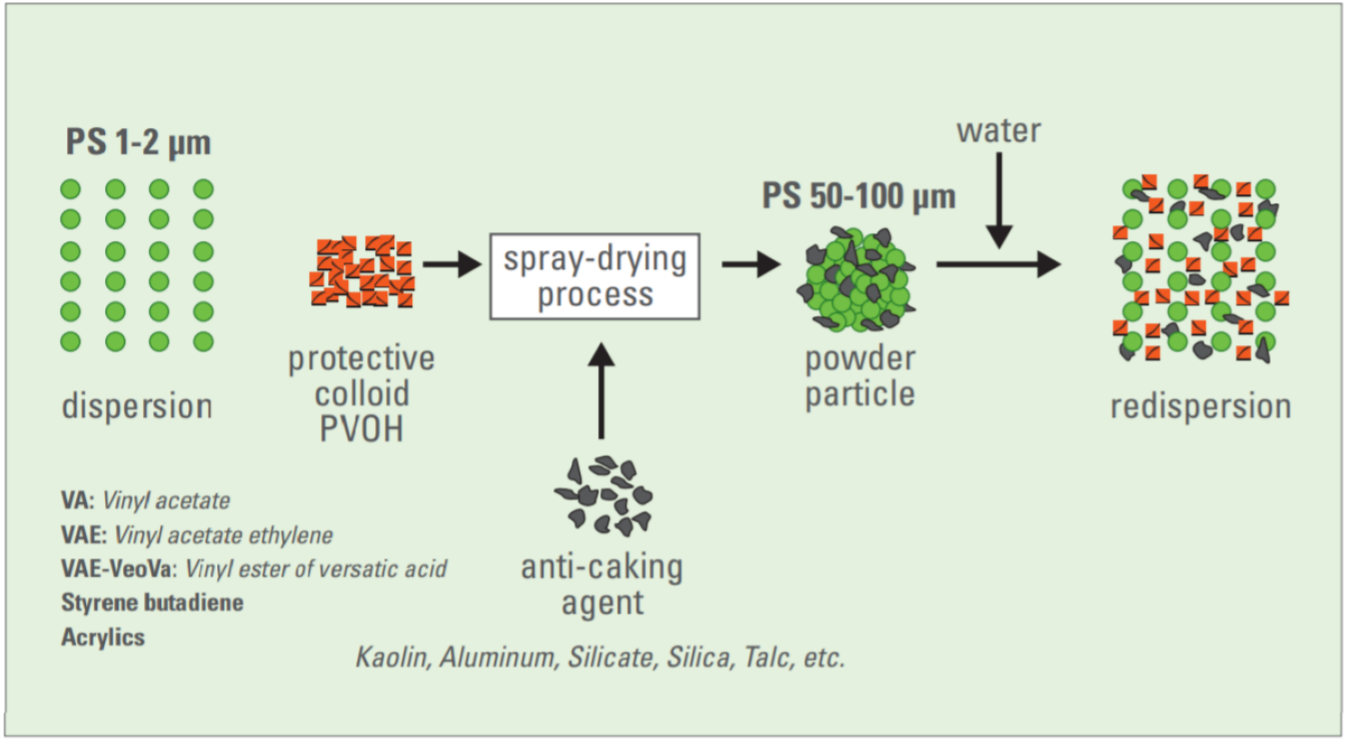
   Wetting: The ability to wet the tile and form a good adhesion with tile

3. **Hardening**

   Creep: Maintain the tiles attached during hardening

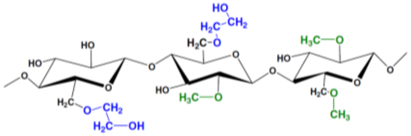
Context

Redispersible polymer powder (RPP): Redispersible form of a polymeric colloidal suspension which improves flexibility, adhesion, tension properties



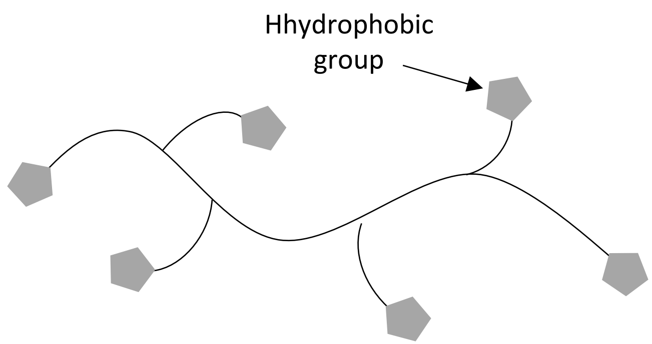
Dow, 2012

Context

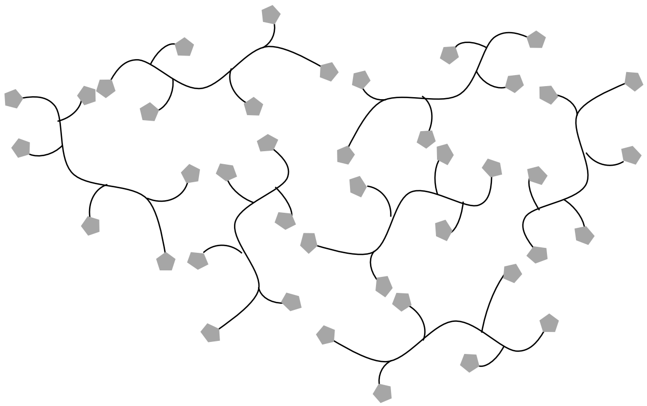


Cellulose ether: associative polymer mainly used as a thickening agent and to improve water retention.

Surfactant



Associative polymer



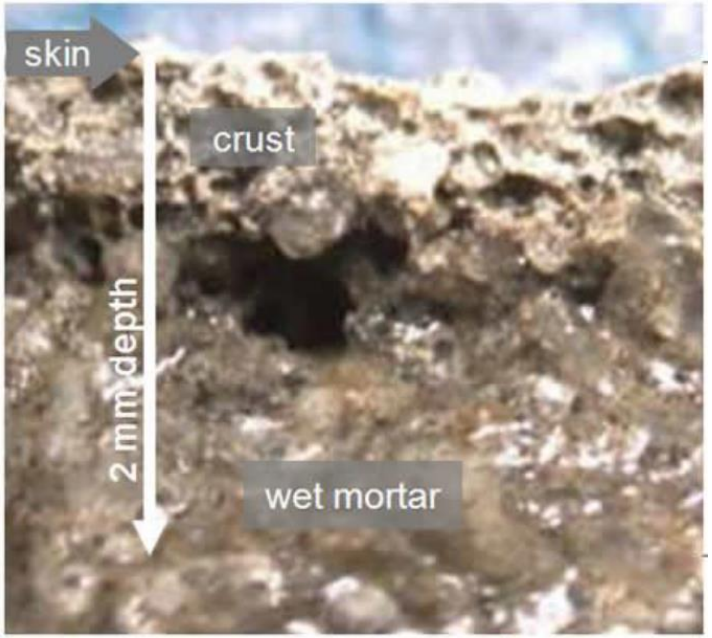
Network through hydrophobic associations

Cellulose Ether in the formulation → Undesired side effect (skin formation)

Context

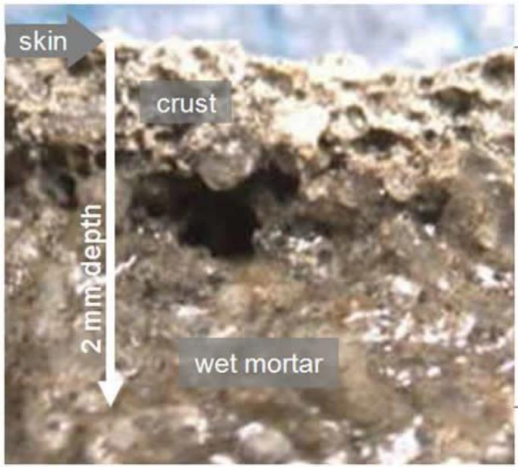
Skin formation: it is a surface layer with different properties compared to the body underneath

- Early drying
- carbonation
- polymer film formation
- ...



[Zurbriggen,2013]

Context



PINI, 2016

How the skin rheological properties
affect adhesive properties?

Objective

Characterize the rheological properties of the skin and understand its influence on adhesive properties.

Scope of the study

- Oscillatory Rheometry
- MRI
- Interfacial Rheology

Previous research on skin characterization

- Squeeze Flow
- Tomography
- Optical Microscopy

Squeeze behavior and Microsctructure/Contact generation and adhesion

Formulations

Portland
Cement (30%)

RPP Latex (2.5%)
VA/VeoVa

Silica Sand (67.5%)

Cellulose Ether (CE)
MHEC

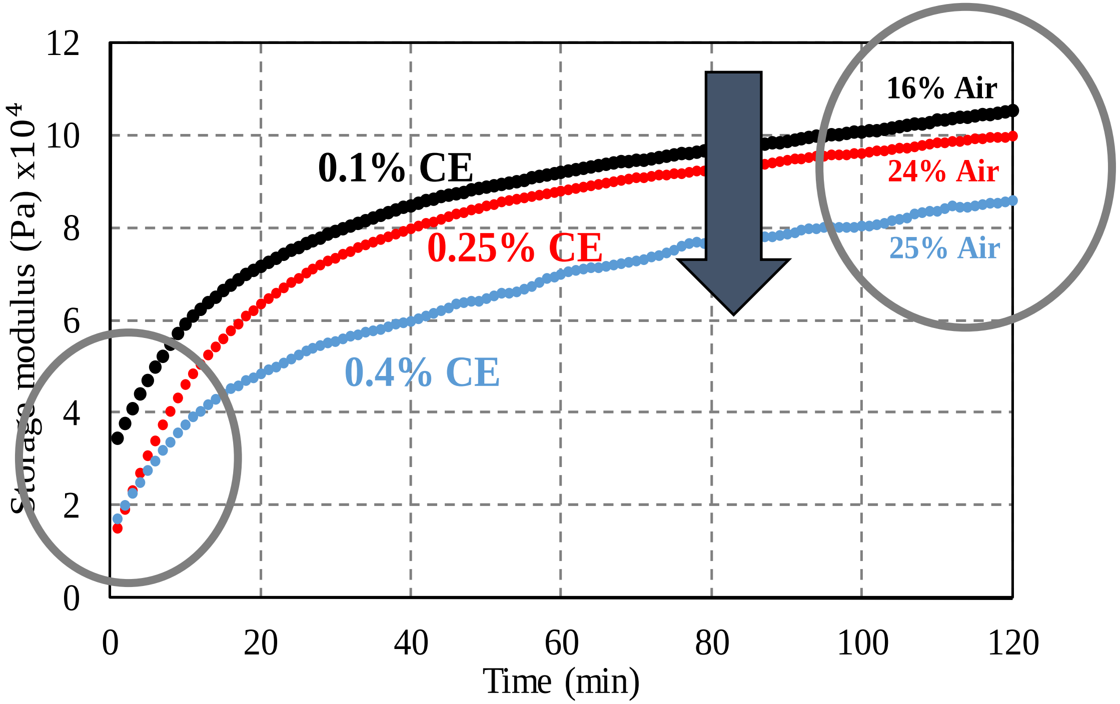
0.1% CE

0.25% CE

0.4% CE

Bulk Properties - Oscillatory Rheometry

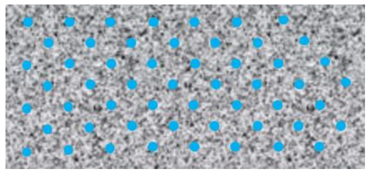
Effect of CE content of storage modulus (G') evolution over time

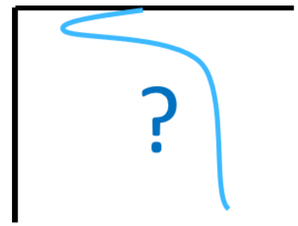


As CE content is increased, the initial values of G' firstly decrease due to entrained air and then increased by CE's thickening effect. Over time,the tendency is that for higher CE content, the lower the G' du to the CE's ability to delay structuring of cement particles.

MRI (Magnetic resonance imaging)

MRI tests were performed in order to observe water distribution and characterize the interface

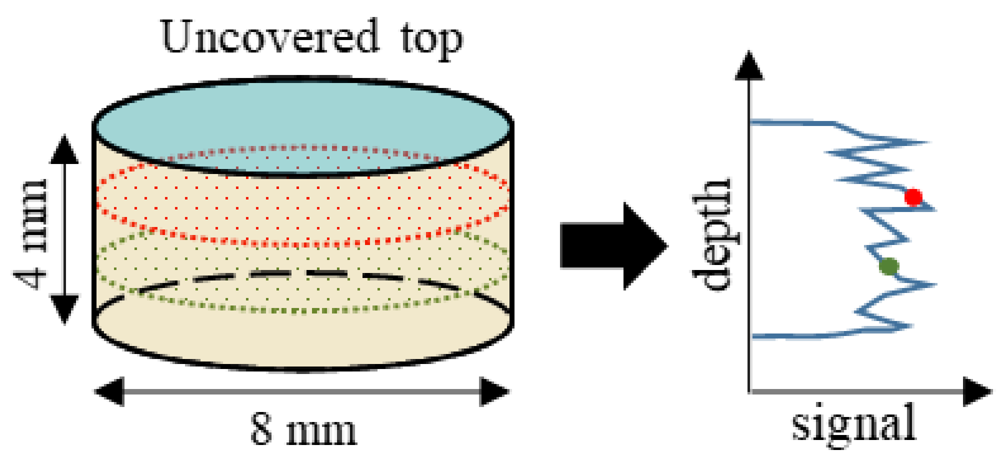




Skin depth and distribution

CNRS - Orléans

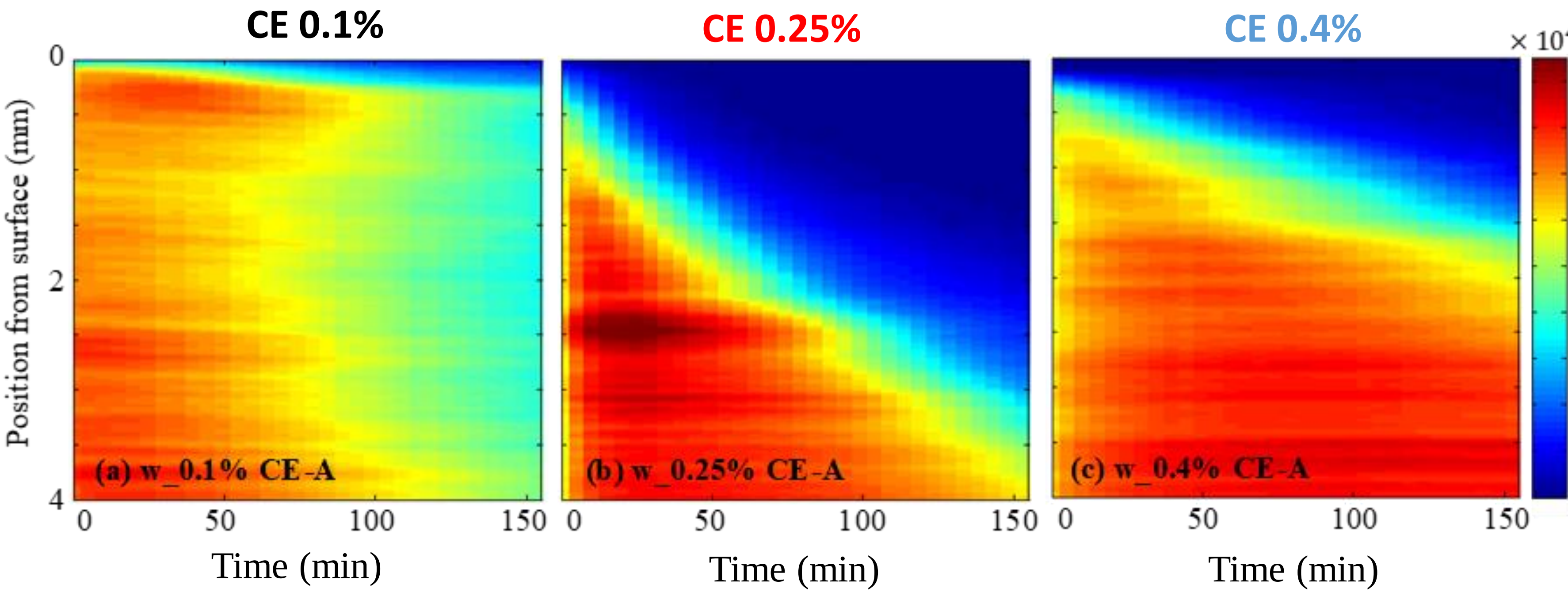
MRI was used to obtain
« mobile »water distribution



MRI (Magnetic resonance imaging)

MRI visualization over time – Different CE content

Blue = low signal = dry
Red = high signal = moisture



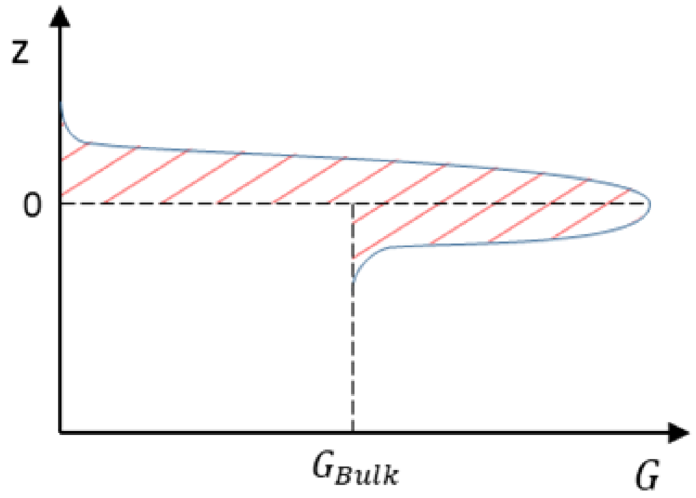
Signal distribution remains homogeneous over the depth, but higher signal loss

Dryer layer linearly growing, while a moisturez layer underneath

Dryer layer linearly growing in a lower rate

Interfacial Rheology

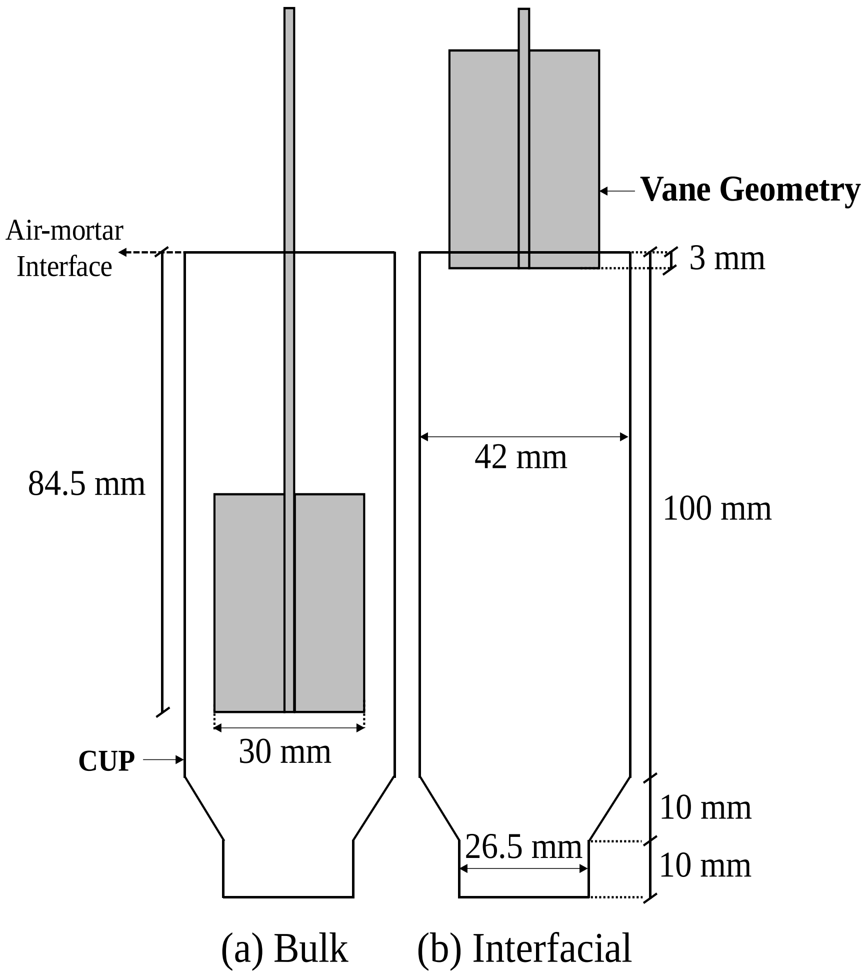
The interfacial measurements are based on Gibbs definition of surface, the "excess property"



$$G_{interface} = \int_{-\infty}^{0} [G(z) - G_{bulk}]dz - \int_{0}^{+\infty} [G(z)]dz$$

$G'_{interface}$ = Average G' including interface – Average G' without interface

Interfacial Rheology



Two measurements:

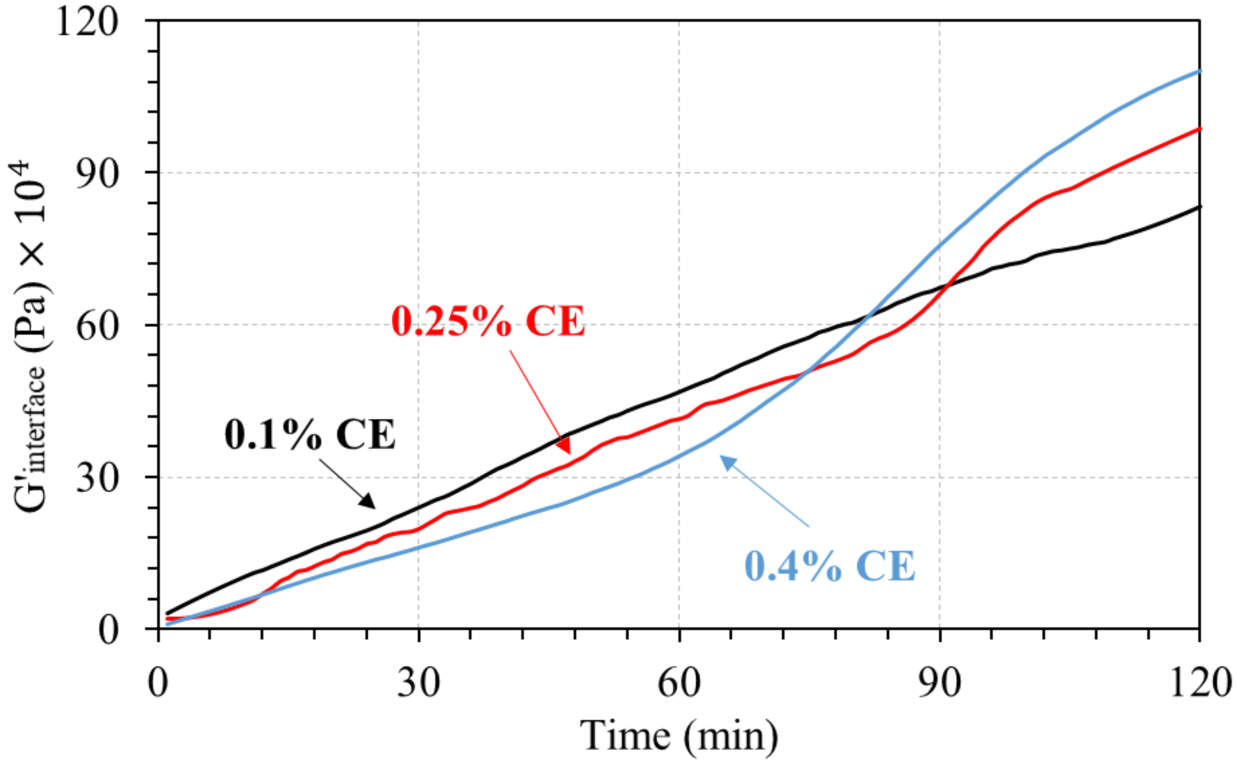
- Including the Interface
- Bulk

$$G'_{interface} = G'_{\text{(including interface)}} - G'_{\text{(bulk)}}$$

$$B_0 = \eta_s / \eta_{subph} L$$

Interfacial Rheology: Gray PC

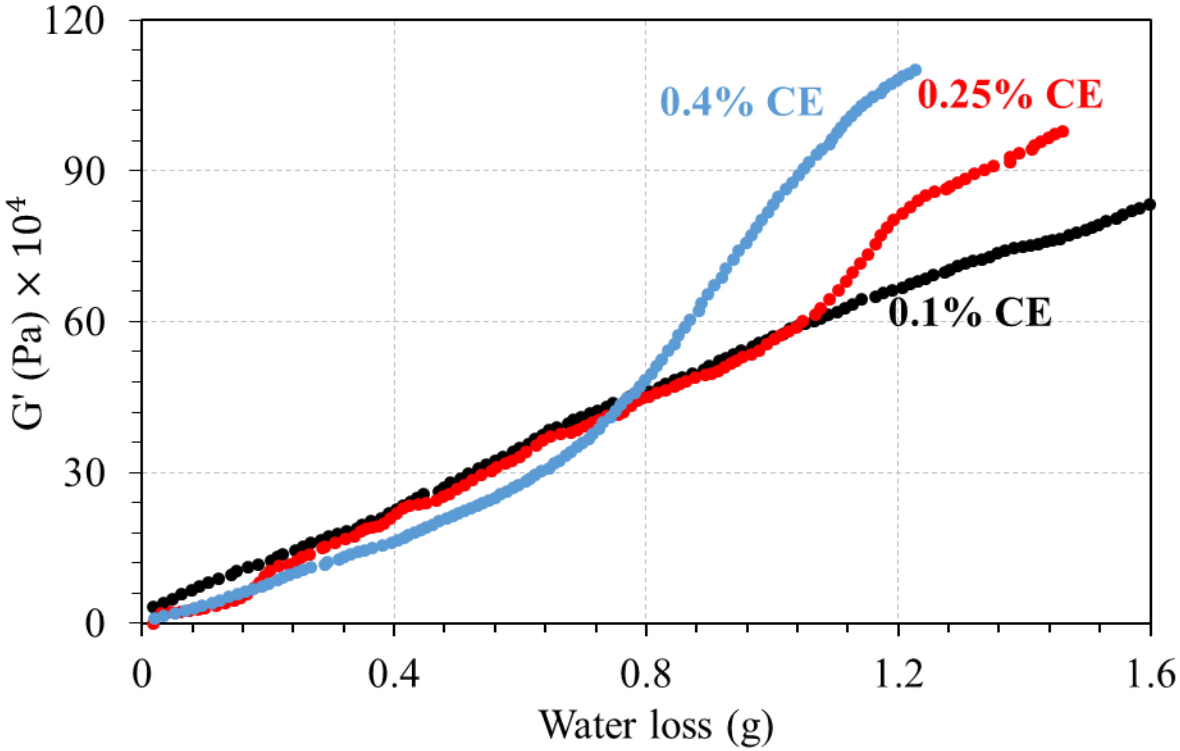
G’(interface) results for mortar with different CE content and gray cement



Similar results to white cement are observed with an initial reduction of G’(interface) as CE content is increased, and then an inversion occurs where higher CE content result in higher G’(interface). CE increase water retention.

Interfacial Rheology: Gray PC

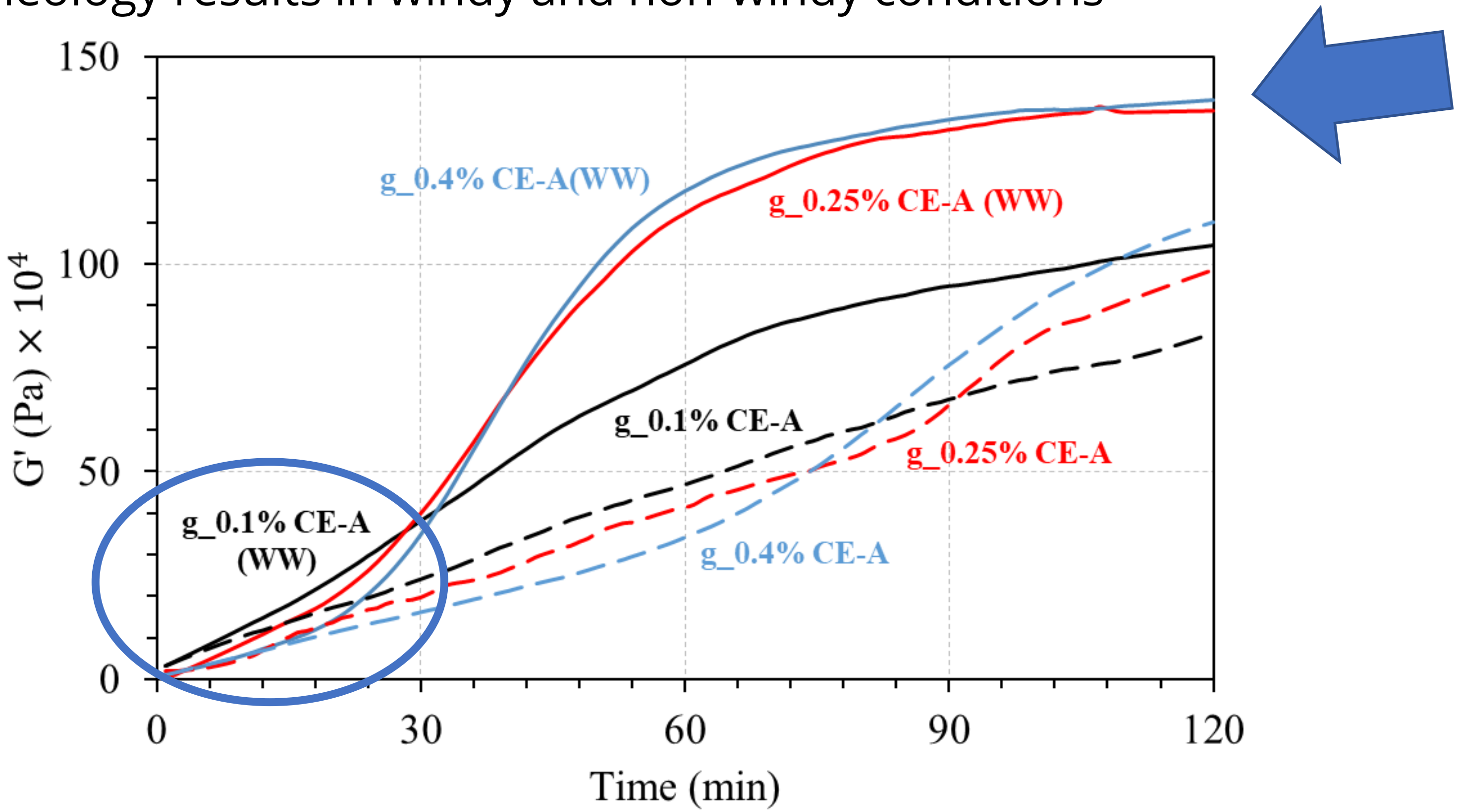
Interfacial rheology vs. water loss for mortar with different CE content and white cement



Similar results are observed for gray cement, reinforcing the water/solids ratio dominannce, followed by a polymer properties dominance at the interface

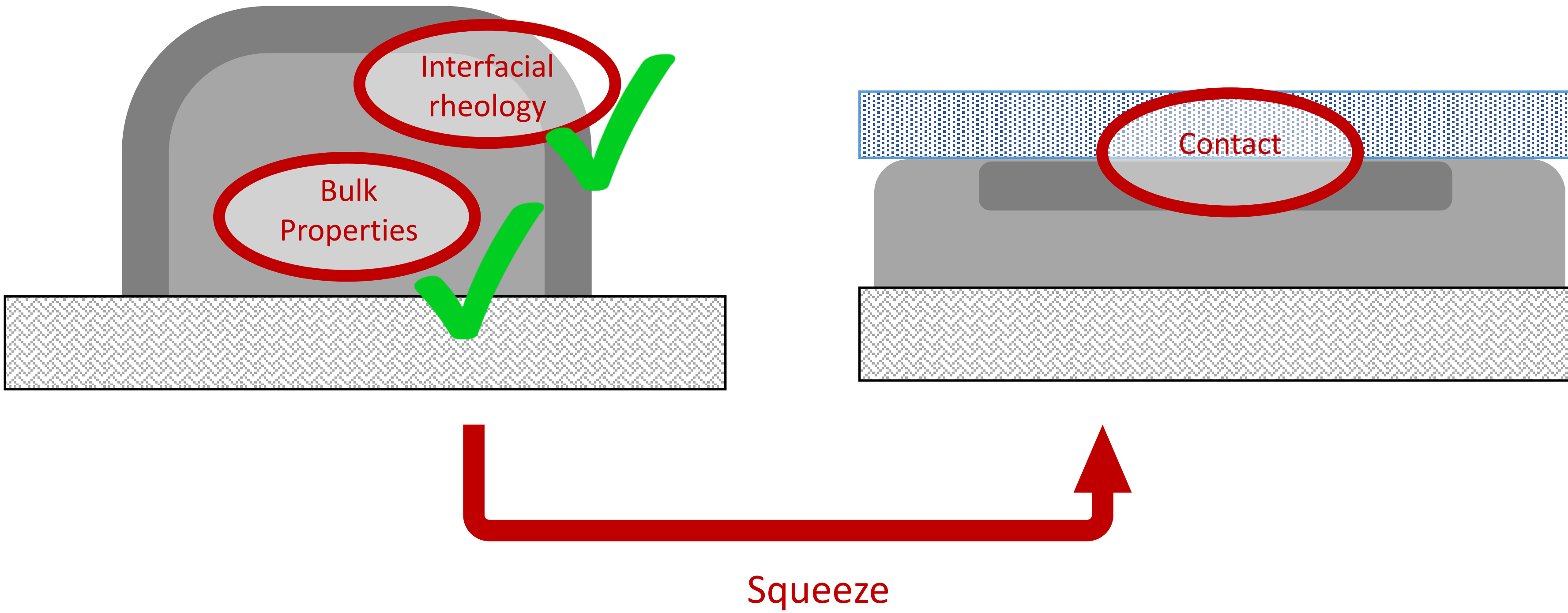
Interfacial Rheology: Impact of wind

Interfacial rheology results in windy and non-windy conditions



The same interfacial measurements were perfomed in windy conditions, which show very similar behavior, but acelerated. The initial decrease of G' evolution, followed by an inversion.

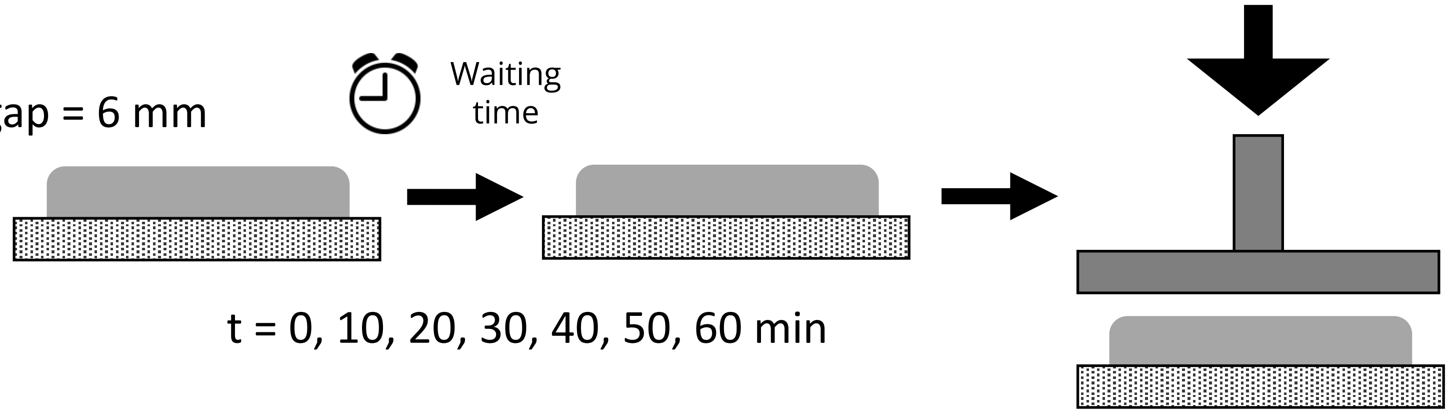
Skin Properties → Adhesion and Contact Generation



Squeeze flow - Procedure

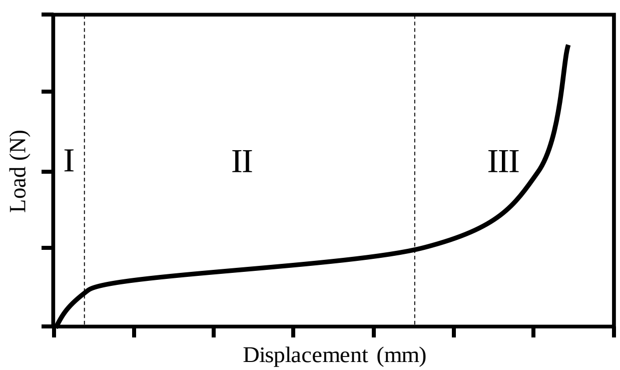
Squeeze flow procedure: samples were prepared and after the different waiting time, the squeeze flow test was perfomed

Initial gap = 6 mm



t = 0, 10, 20, 30, 40, 50, 60 min

Final gap = 3 mm

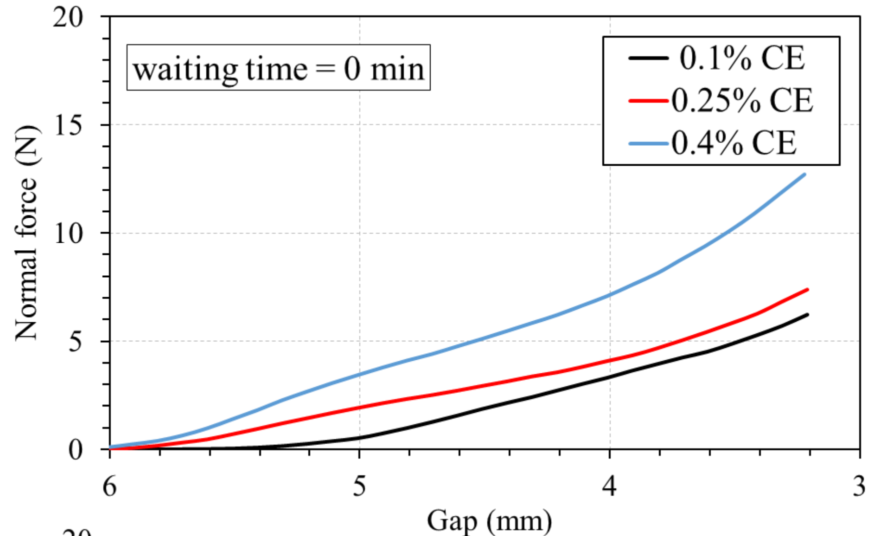


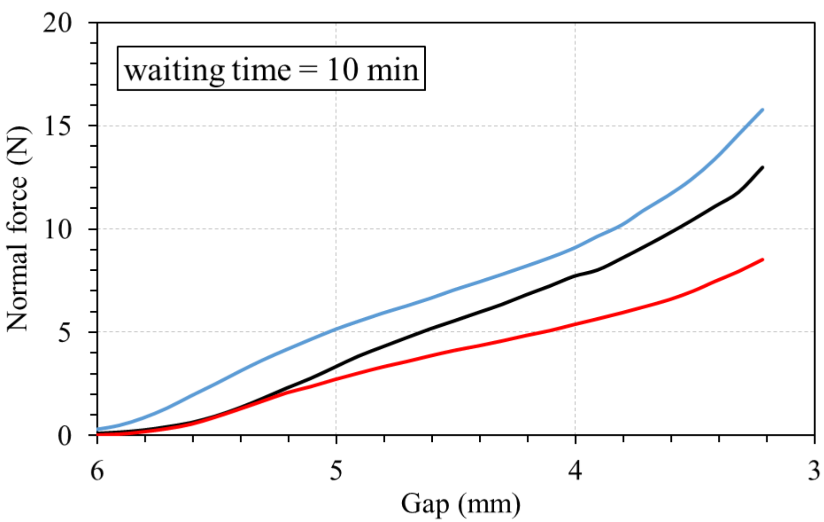
Typical load vs. displacement curve of a displacement-controlled squeeze flow test [Cardoso, 2009]

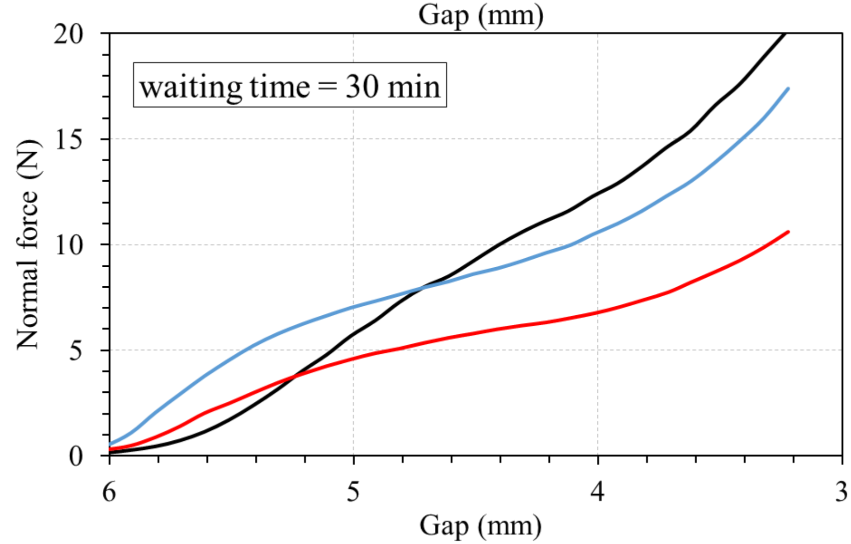
Squeeze flow: Impact of CE content

CE content influence on Squeeze flow

- Initial result show the increase of viscosity as CE is increased
- As the waiting times evolve, the formulation with 0.1% CE final force start to evolve, overcoming the other formulations

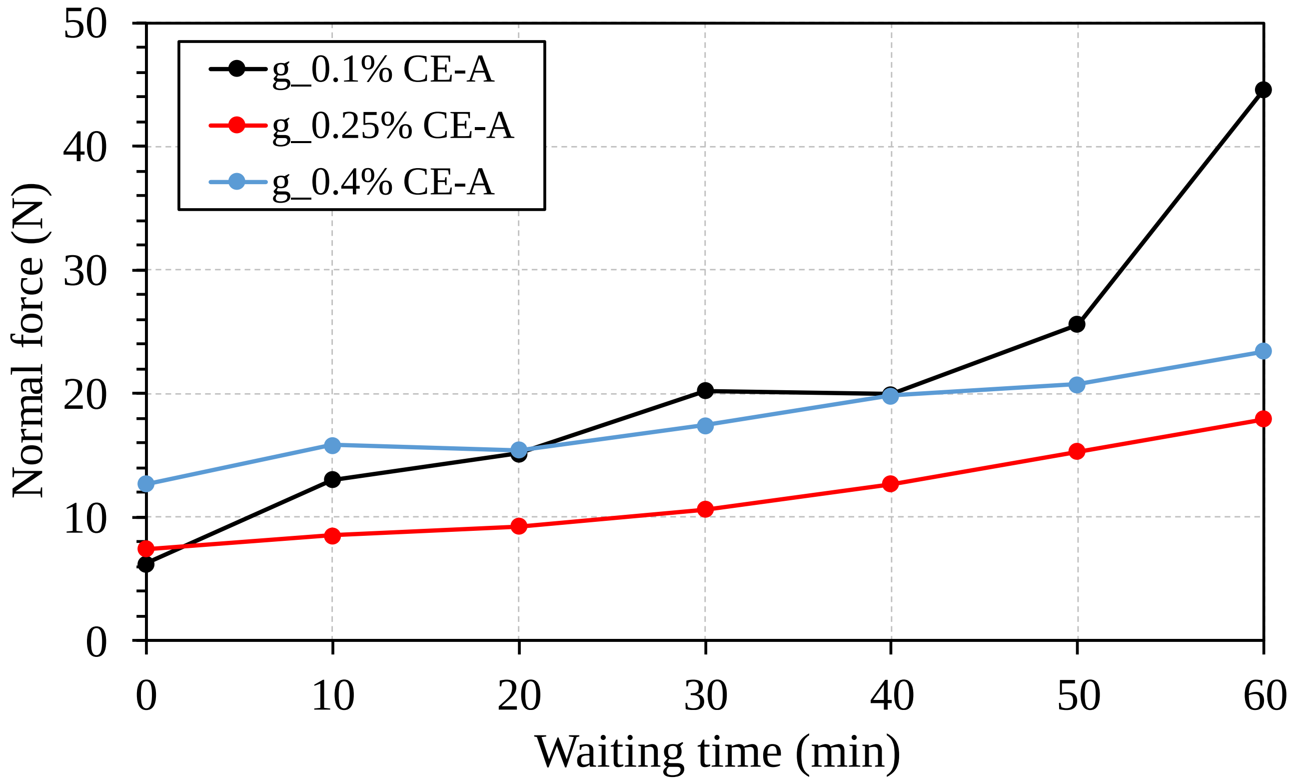






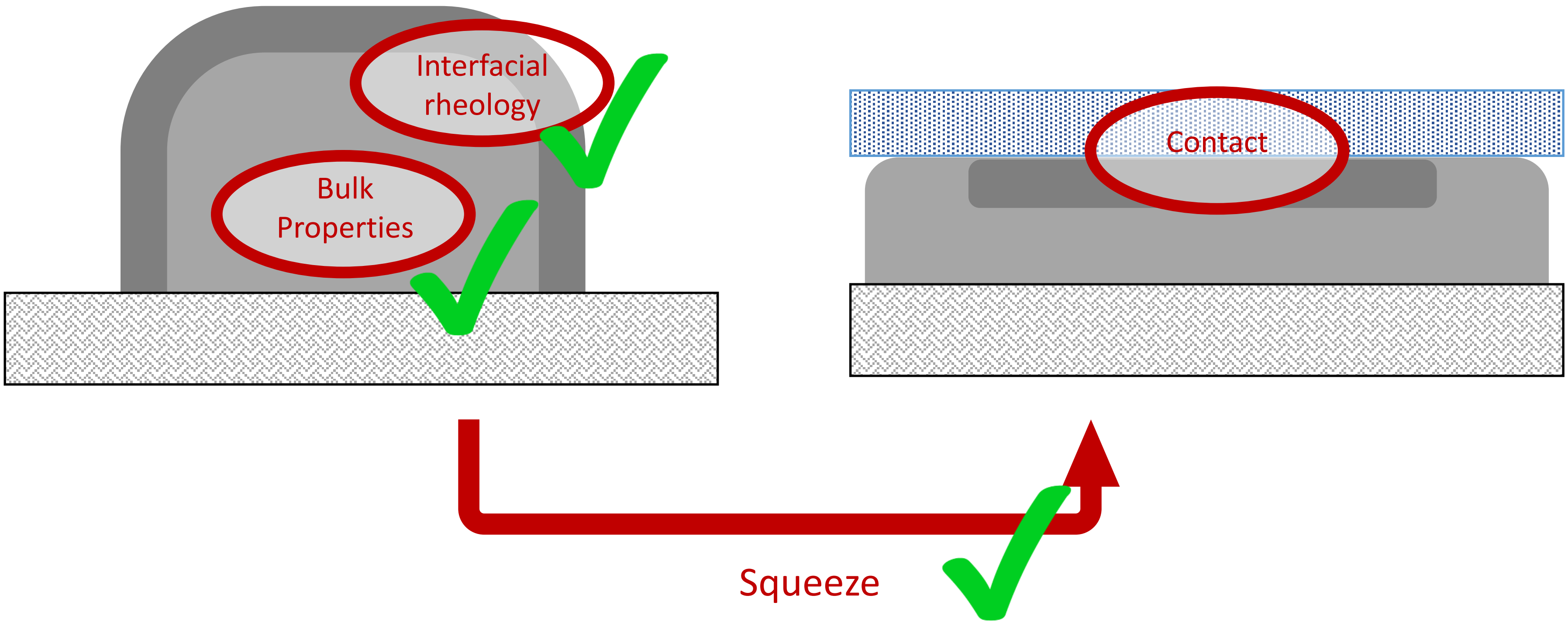
Squeeze flow: Impact of CE content

Final normal force of squeeze flow measurements



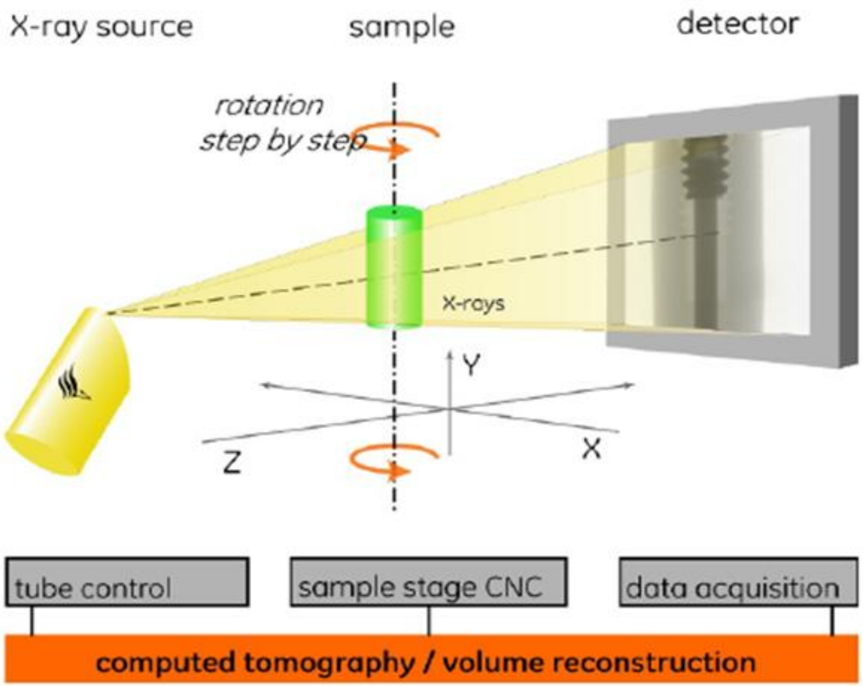
Delayed structure building of CE and less evaporation of the formulations with higher CE content

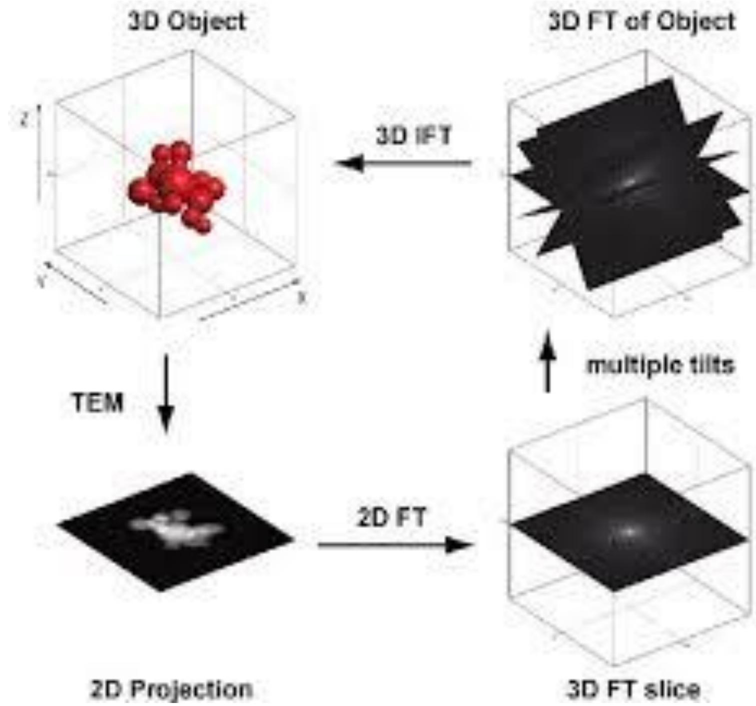
Skin Properties → Adhesion and Contact Generation



Microtomography

Tomography is imaging technique based on computation reconstruction (CT) of X-ray images.

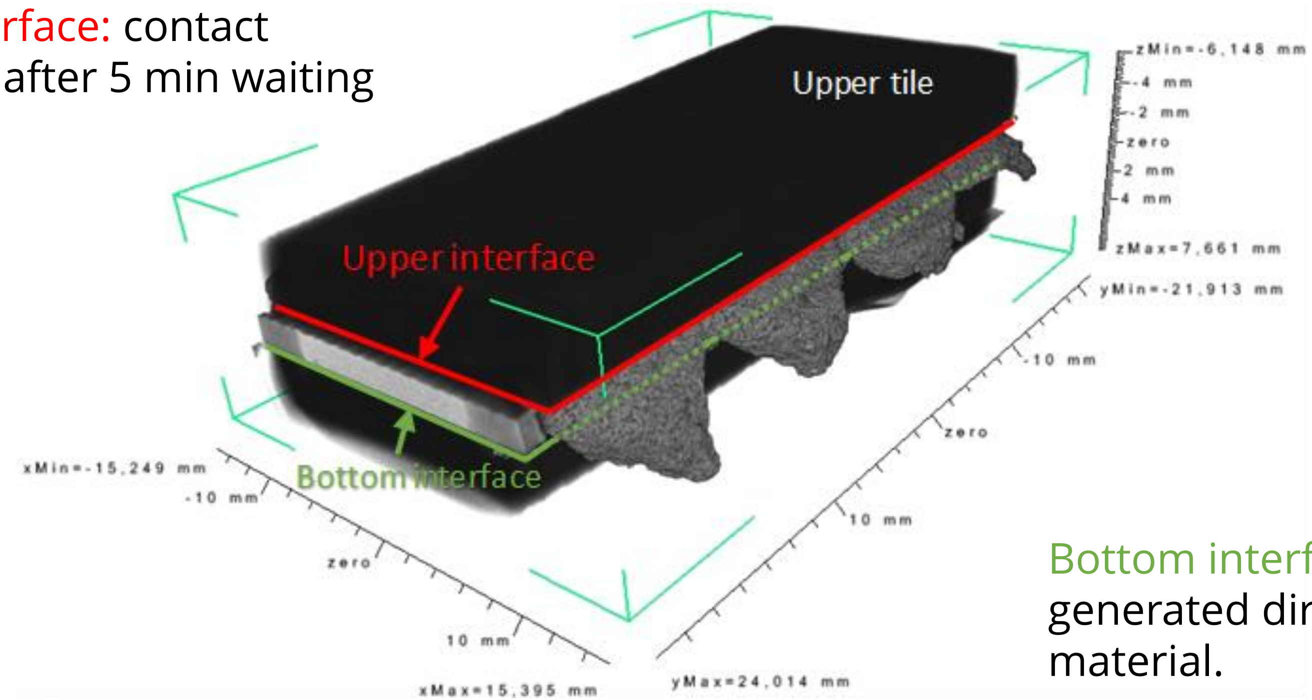




Microtomography: Contact

Contact of adhesive mortar and tile

Upper interface: contact generated after 5 min waiting

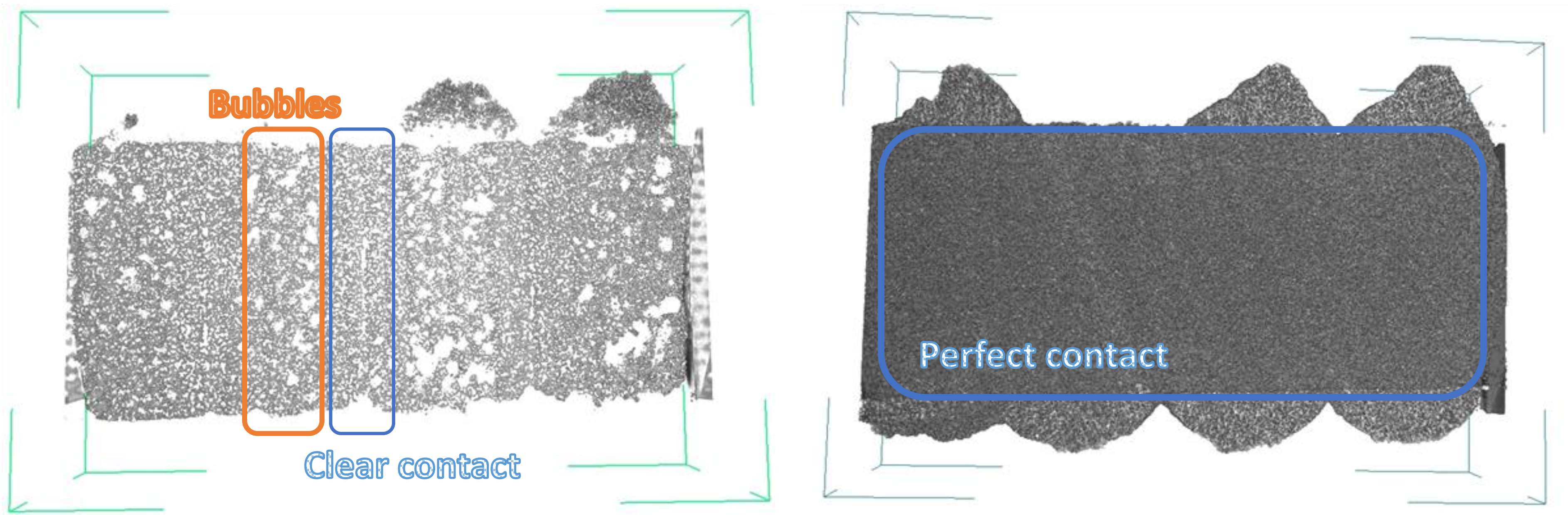


Bottom interface: contact generated directly of fresh material.

The tomographic 3D images were used to observe the contact at upper interface and bottom interface

Microtomography: Contact

Contact of adhesive mortar and tile



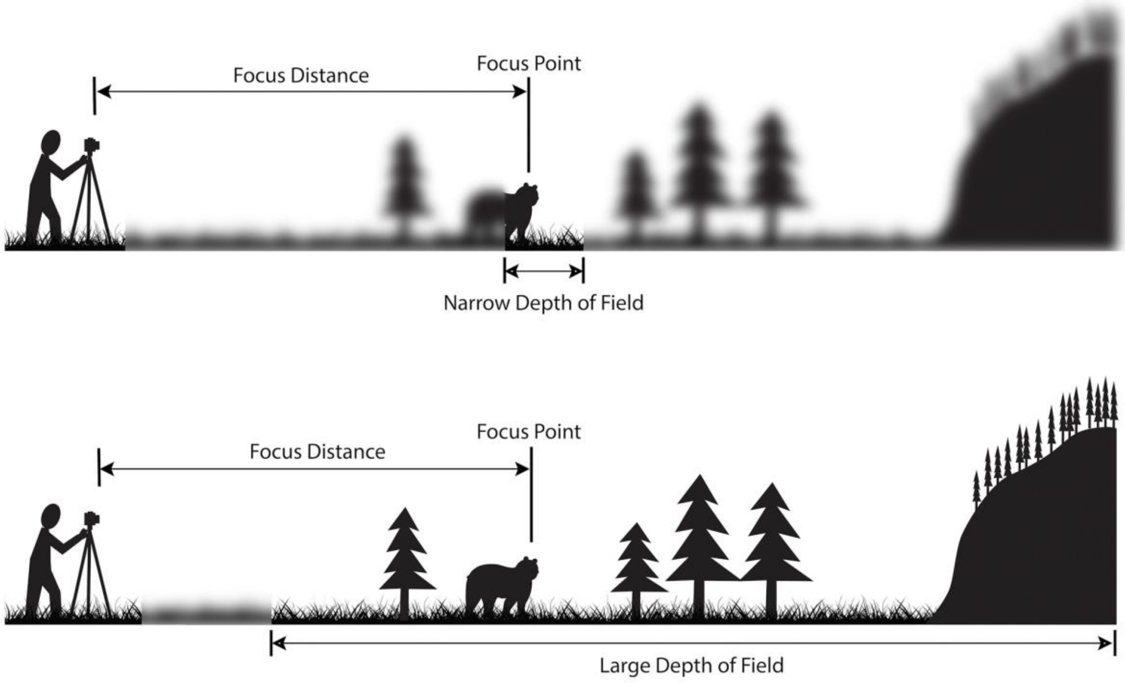
(a) Upper interface

(b) Bottom interface

The images show a clear differente between the upper and bottom interface. In the upper interface, the region of the strips there are bubbles/poor contact and a better contact between the strips. In the bottom interface, the contact was perfect.

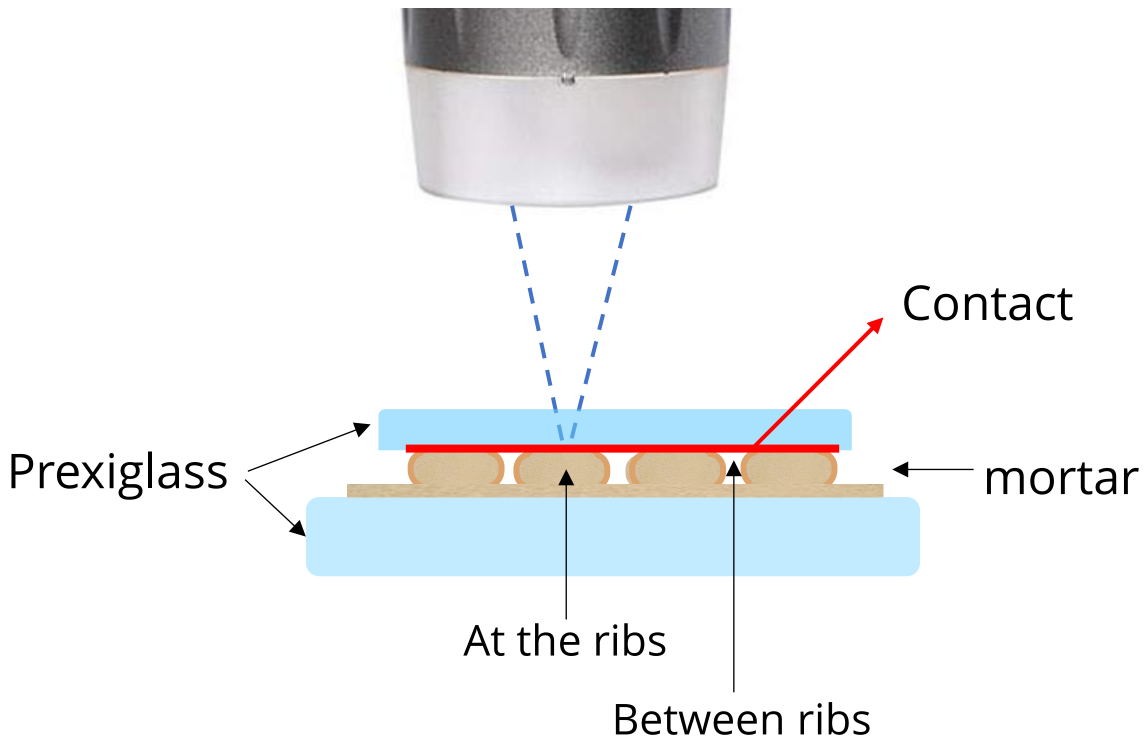
Optical Microscopy

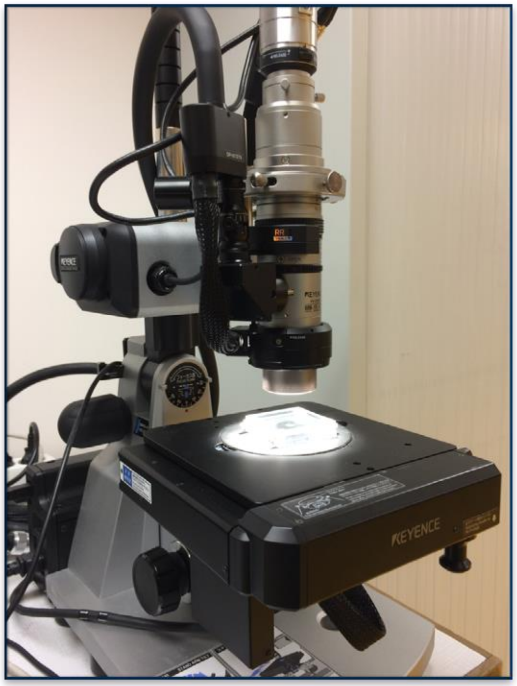
Narrow depth of field: Narrow distance where the image is focalized



Optical Microscopy

<u>Narrow depth-of-field</u> microcopy: visualize the interface

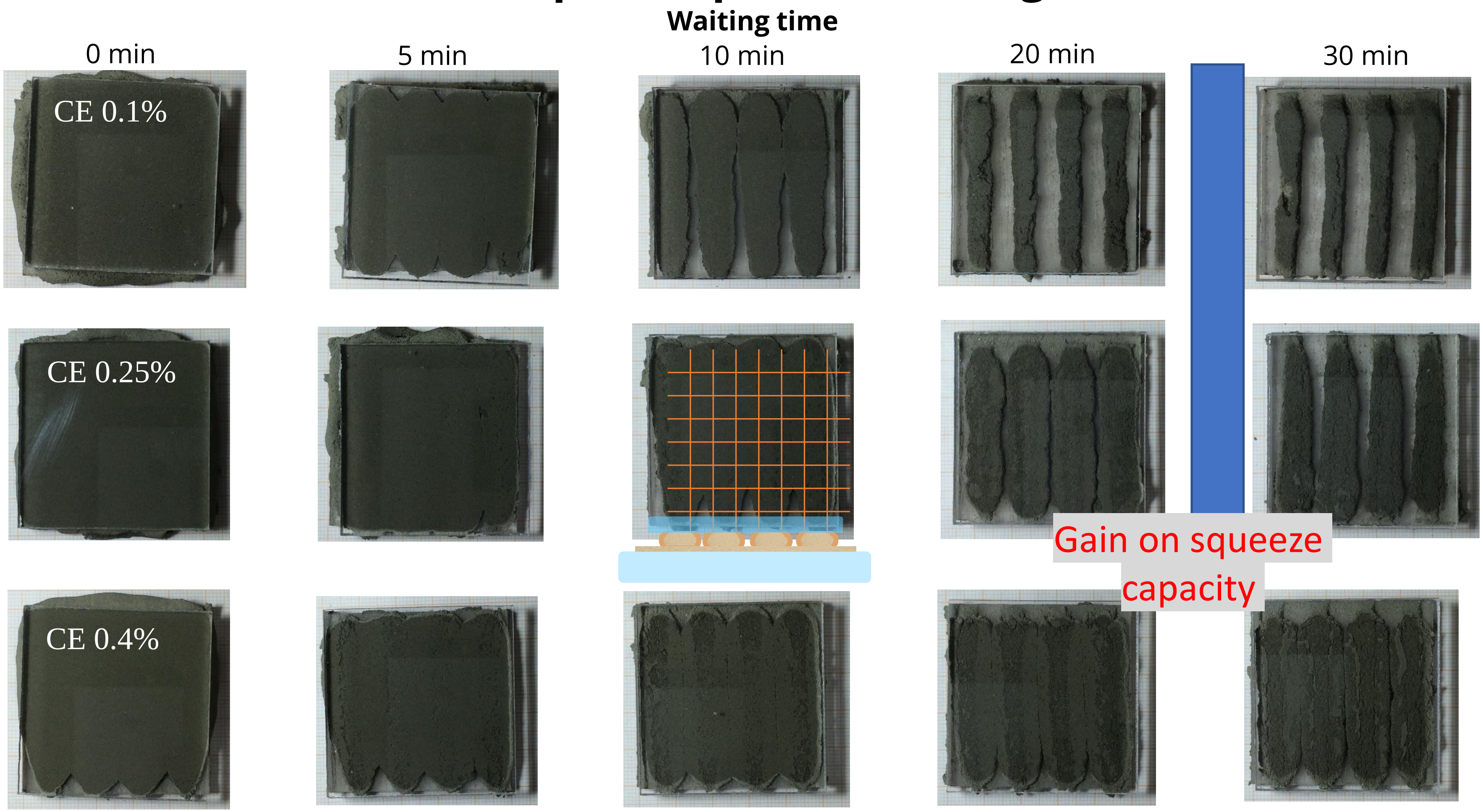




Only the interface between the mortar and the tile will be focalized

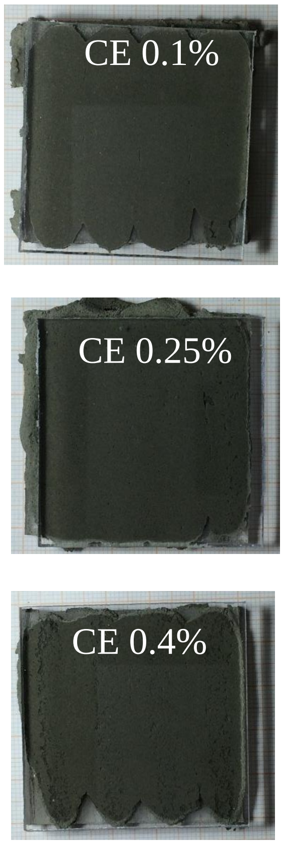
Optical Microscopy

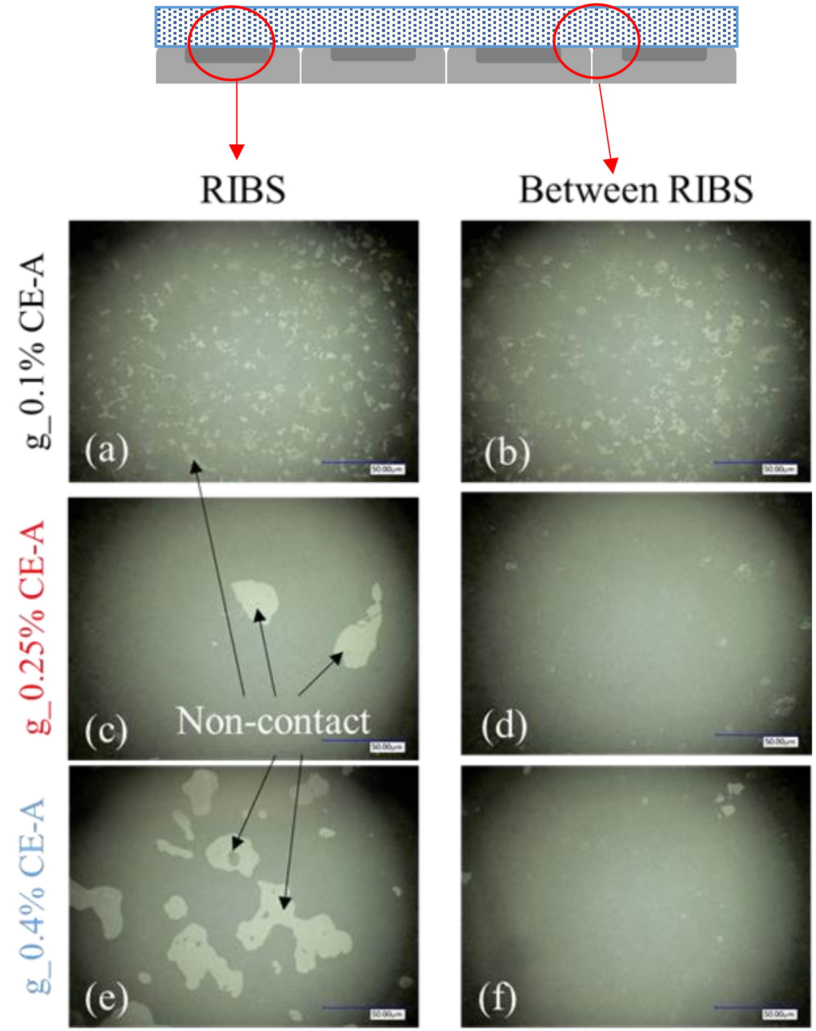
Effect of CE content – samples (equal force – 2kg/30s)



Optical Microscopy

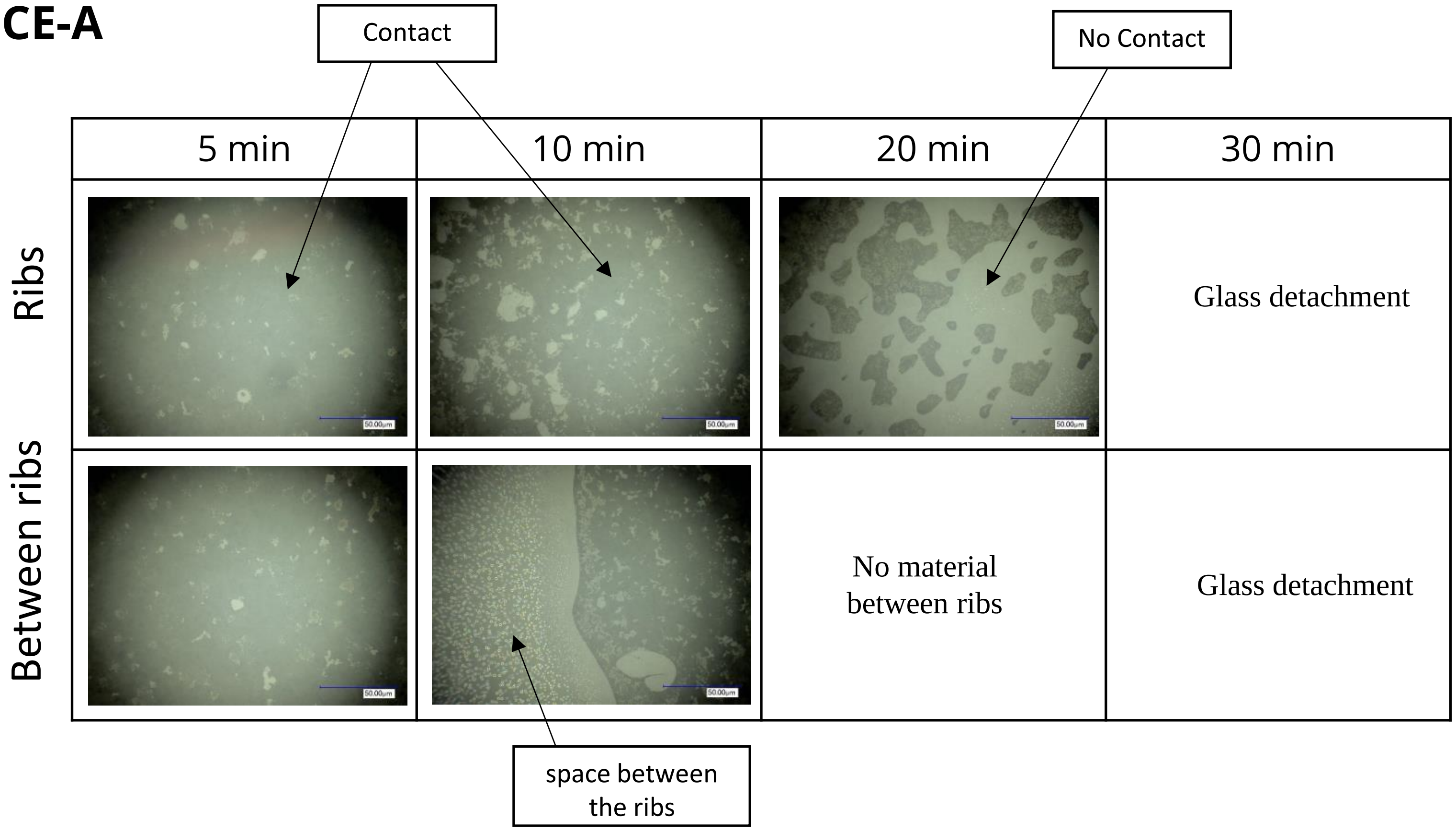
Waiting time = 5 min





Optical Microscopy

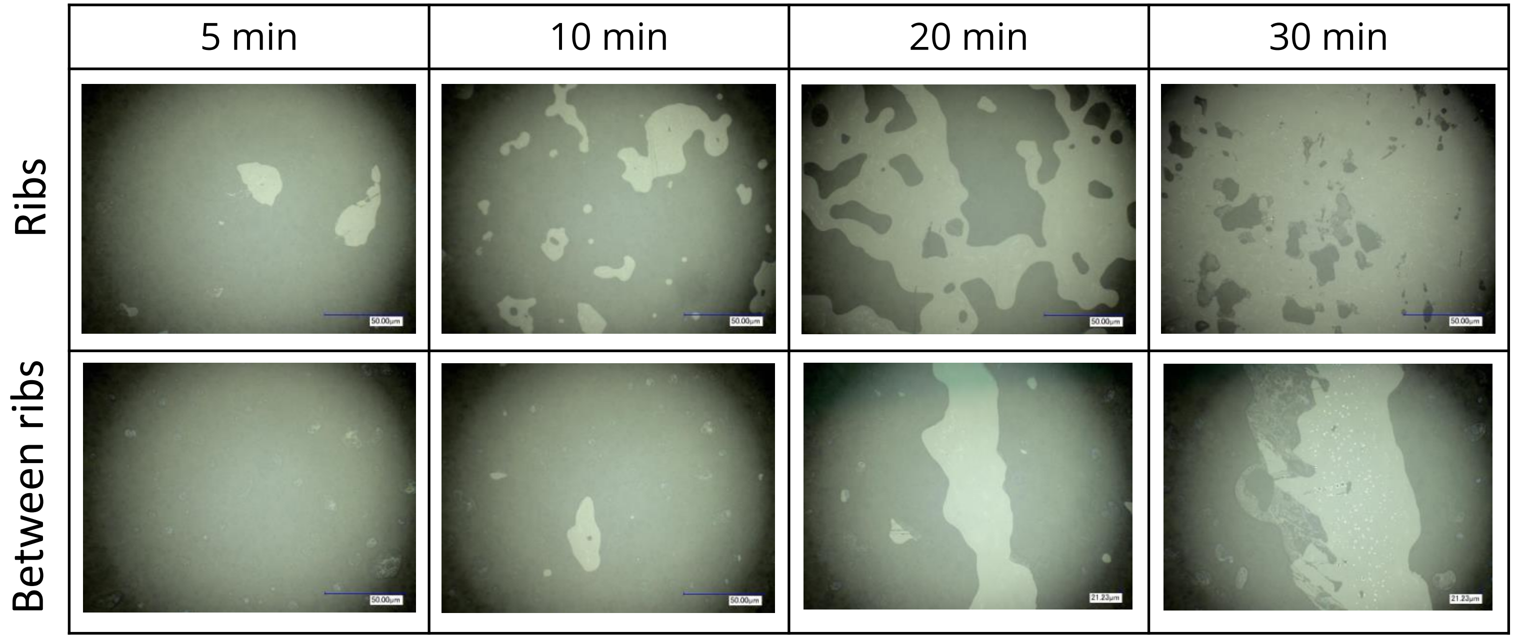
0.1% CE-A



The formulation with low CE content, rapdly loses it ability to deform, not being fully squeezed and generating space between the ribs. After 20 min the glass detaches.

Optical Microscopy

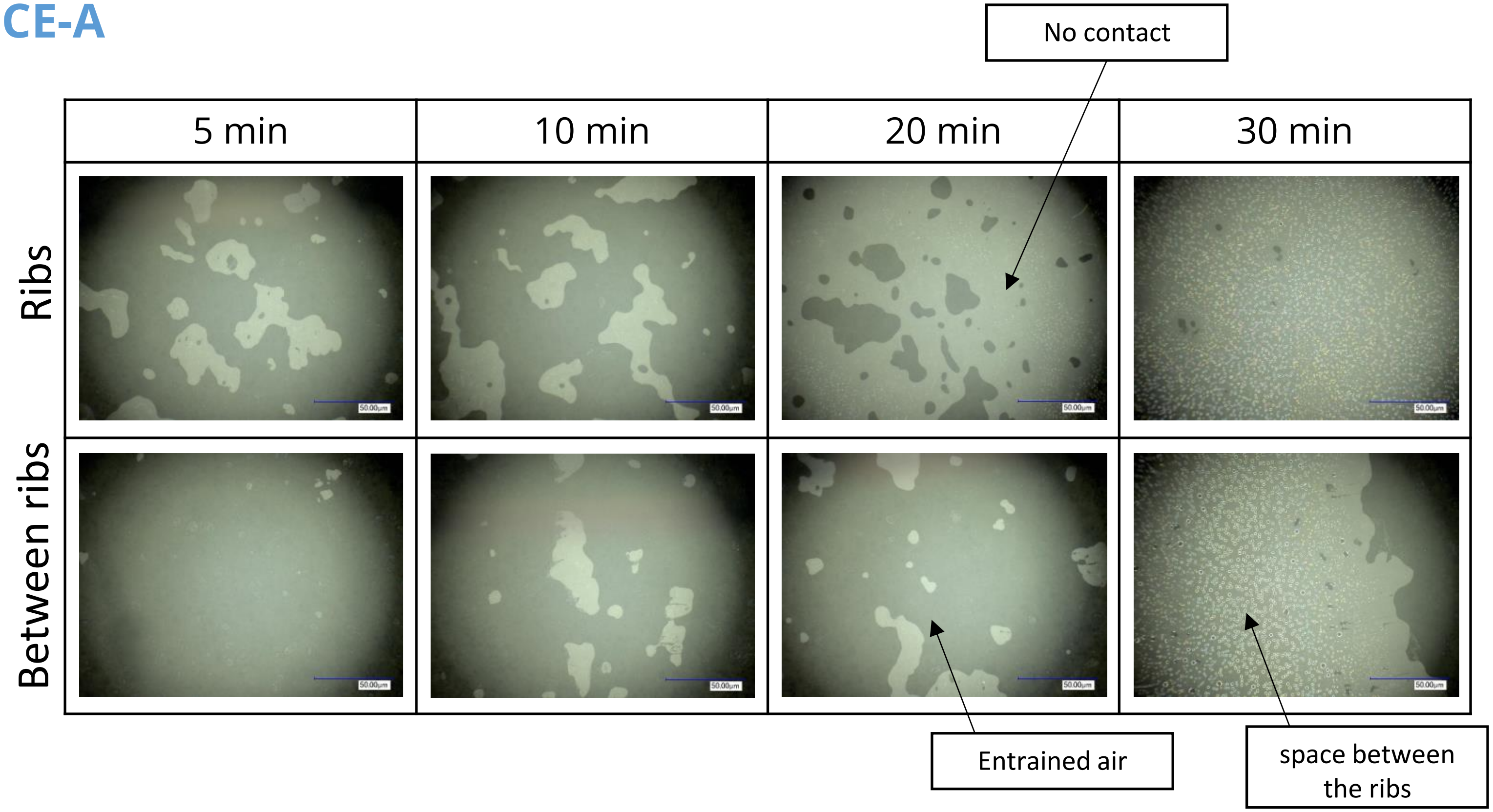
0.25% CE-A



The formulation with 0.25% CE loses contact at the ribs zones over time, but the contact in the zone between the ribs was able to maintain a good contact for a longer period

Optical Microscopy

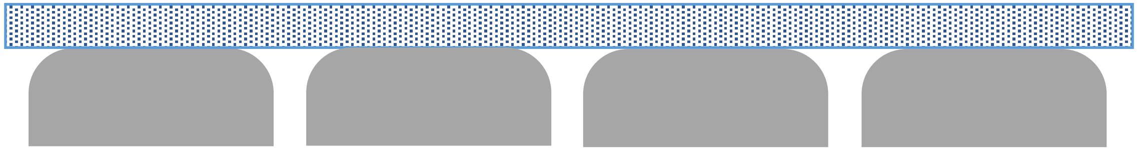
0.4% CE-A



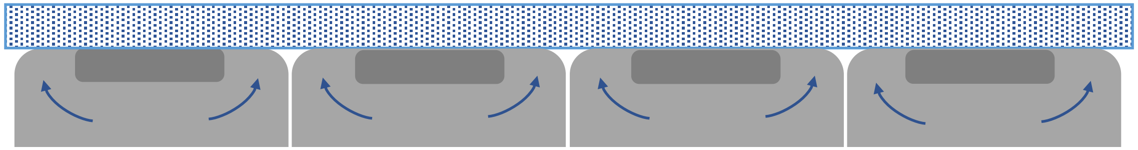
The formulation with 0.4% CE loses contact at the ribs zones over time faster, but the contact in the zone between the ribs was able to maintain a good contact for a longer period than the other formulations

Optical Microscopy

Formulations with lower CE content do not form a skin, but have worse squeeze flow properties, resulting in lower deformation and poor contact generation

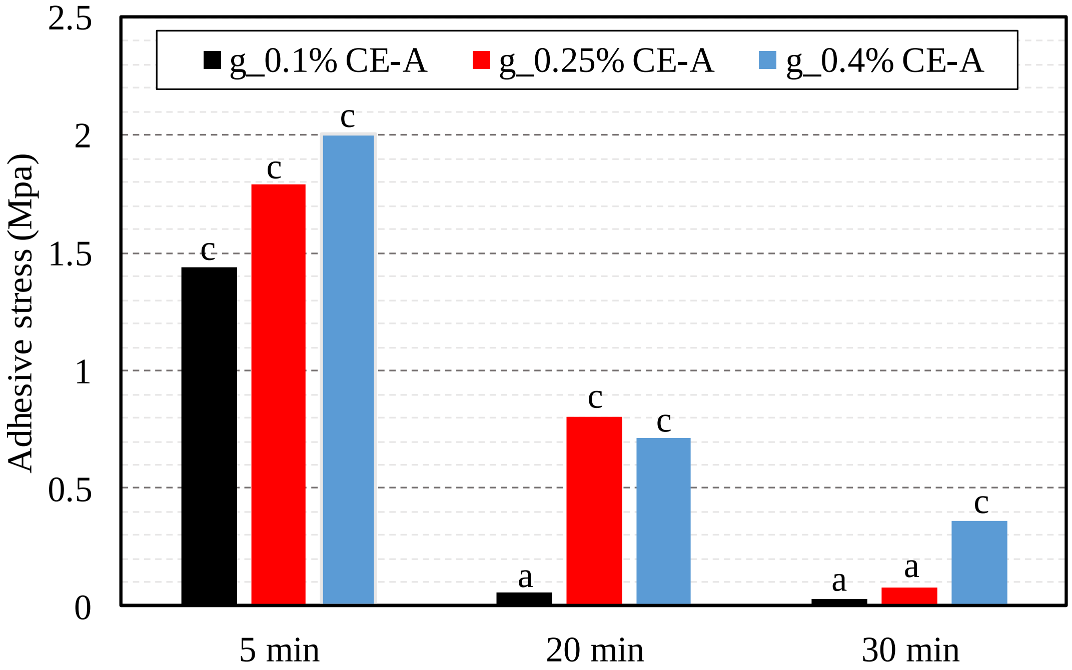


Formulations with **higher CE content**, despite the skin formation, fresh material is able to flow and form good contact with the tile.



Adhesive stress

Adhesive stress at 28 days



The first 5 min, the higher stress is probably related to the higher water retention due to higher CE content. For 20 and 30 min, the loss of adhesive stress is related to the squeeze flow ability.

Conclusions and Perspectives

- **Squeeze flow:** for low CE content - increase of stress forces occur over the waiting time
- **Micro-tomography:** Indication of contact generation impact was observed
- **Narrow depth-of-field for contact visualization:** despite the skin formation obseved for higher CE content, the fresh material inside the skin is able to generate good contact with the tile.

Finally, further comprehension on skin formation was achieved in this study and new questions and perspective possibilities were opened.

- The presence of the skin itself does not represent an issue if it is able to break and release fresh material
- Techniques and analysis of this study could be helpful to formulation engineering
- The impact of other polymer additives and the synergical impact on adhesive properties
- Other mortar's interfacial properties could have an influence on adhesion, such as extensional behavior of the skin

Thank you very much!