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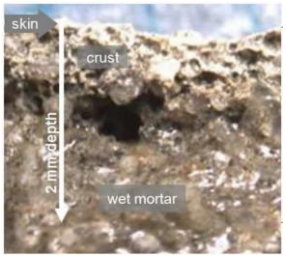
PhD – Research summary

Adhesive Properties and Interfacial Rheology of Adhesive Mortars

Adhesive mortar is a mansonary element which is used to glue tiles to walls and floors. To achieve best performance, a combination of different properties of the material in its fresh state is required. One of the main additives used to improve its properties is cellulose ether (CE), but an undesirable side effect of this indispensable admixture to this type of material is the formation of a dry skin on its surface. The presence of this layer at the mortar-air interface can interfere in the mortar's wetting properties and its ability to form proper contact with the tile. Since the initial contact is the first step for the development of adhesive properties between the materials, comprehension of the rheological properties and contact generation is crucial.



[Zurbriggen,2013]

In this context, the objective of this research was to:

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

Throughout the application, different types of strains and stresses are imposed to the adhesive mortar, and one method is not enough to fully characterize its behavior. Thus, in this investigation various techniques were used to evaluate the mortar's fresh state properties.

This research is split in three main parts:

- Bulk properties characterization with various rheological analysis;
- Air-mortar interface characterization through MRI and Interfacial Rheology;
- Microstructure/Contact generation and Adhesion with micro-tomography and optical microscopy.

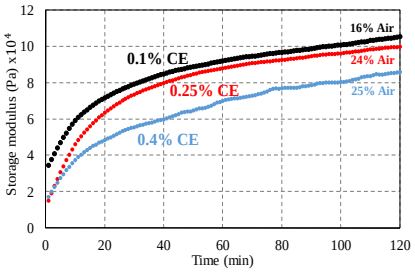
Model adhesive mortar's formulations with different CE content of MHEC were used:

Formulations	CEM I	Sand	Latex	CE	Water/powder
0.1% CE	30%	67.40%	2.5%	0.1%	
0.25% CE	30%	67.25%	2.5%	0.25%	0.34
0.4% CE	30%	67.10%	2.5%	0.4%	

Bulk Properties Evaluation

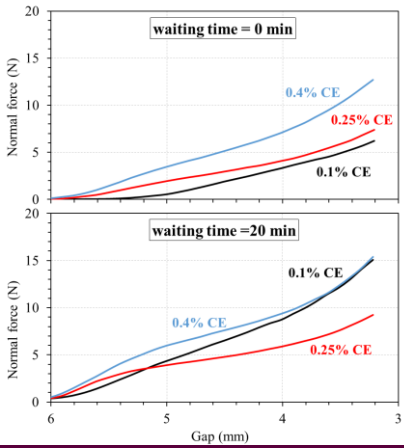
Small amplitude oscillatory measurements (SAOS)

Oscillatory measurement consists in applying a sinusoidal deformation to the mortar, and obtain a sinusoidal stress response. From this test, it is possible to obtain elastic and viscous properties of the material without affecting its structure. Results showed a delay in the structure building of cement particles as CE content was increased.



Squeeze Flow

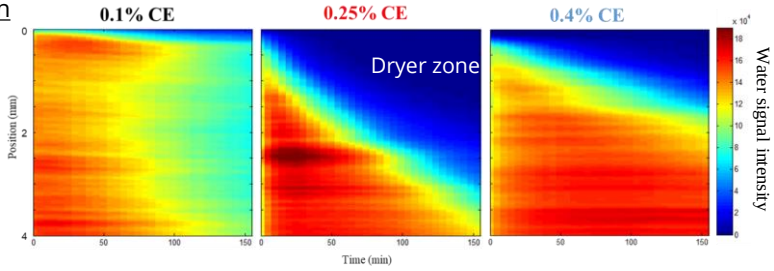
Squeeze flow consists of a test where a sample is compressed at constant velocity while the normal force is measured. The method was used to evaluate adhesive mortars formulations after different waiting times. Initial results show an increase of viscosity as CE is increased, but for longer wait times, the formulation with 0.1% CE final force starts to evolve, overcoming the other formulations. The tests results allowed to identify differences on the mortar's squeeze flow ability over time.



Air-Mortar Interface Characterization

Magnetic Resonance Imaging (MRI) Characterization

MRI was used to obtain the distribution of free water content at the air-mortar interface subjected to water evaporation. The evolution of the interface over time was characterized. For the formulations with higher CE content, a dryer surface layer formation with a moisturized zone underneath was observed.



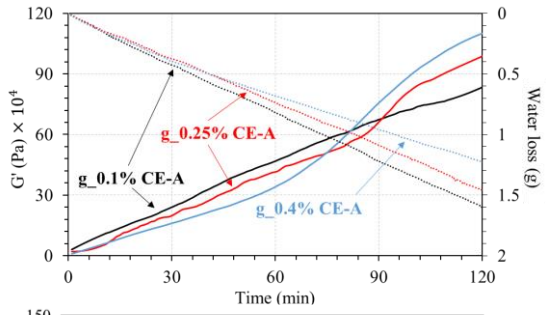
Interfacial Rheology

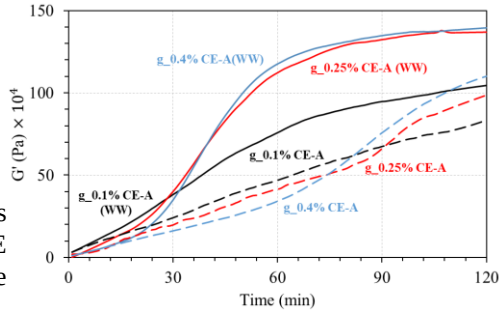
In this research, the concept of interfacial rheology was introduced to granular materials. The interfacial results are based on Gibb's definition of "excess property". The evolution of interfacial properties was obtained through two measurements: one in which the vane geometry is partially immersed (3mm) including the interface and another with the geometry fully immersed in the bulk. The excess property is obtained by the difference between the measurement at 3mm and at the bulk. For the measurements at 3mm, the same test parameters of the bulk measurements were used. The bulk properties subtraction allows a closer approach to the real interfacial properties. In this investigation, low amplitude storage modulus (G') was defined as:

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

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

Through the technique, it was possible to verify a reduction of $G'_{interface}$ as CE content is increased, and then an inversion occurs where higher CE content result in higher $G'_{interface}$. The behavior is accelerated when the test is performed in windy conditions.

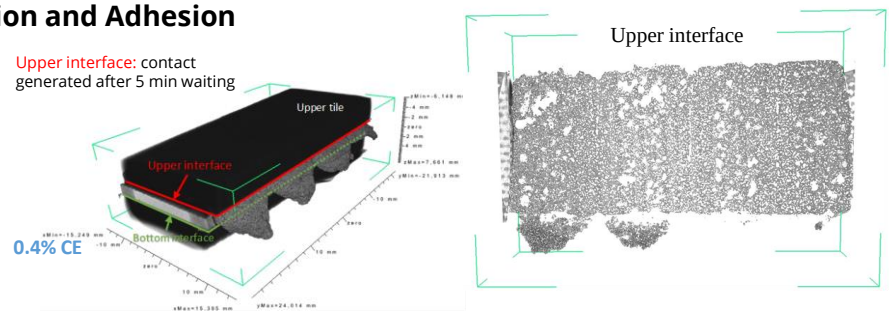




Microstructure/Contact generation and Adhesion

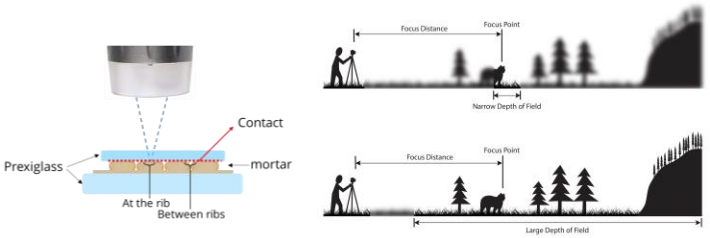
Micro-tomography

Micro-tomography technique was used to characterize the microsctructure of the adhesive mortar's system. With the technique, evidences of the microstructure of the interface between mortar-tile were observed. Two different zones were observed: Contact with bubbles and clear perfect contact.

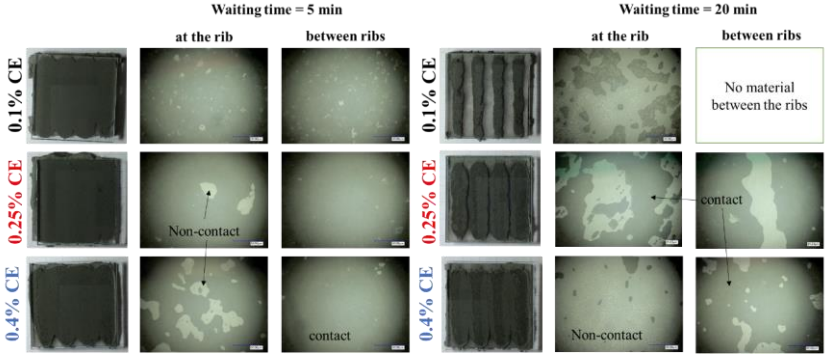


Optical Microscopy

Optical microscopy was used in order to obtain the contact generated between the tile and the mortars. This new technique focuses in the region of the contact with a narrow depth-of-field microscope and observe the wetted and unwetted regions. This method was used to evaluate the contact generated after different waiting times.



The results indicated that despite the dryer layer (skin) as CE content is increased, when squeezed, fresh material is released and therefore generates good contact between the ribs zone.



This research represents an original study in the field of construction materials, since it allows further comprehension on skin formation of adhesive mortars.