Elaboration and study of the functional properties of geopolymer foams

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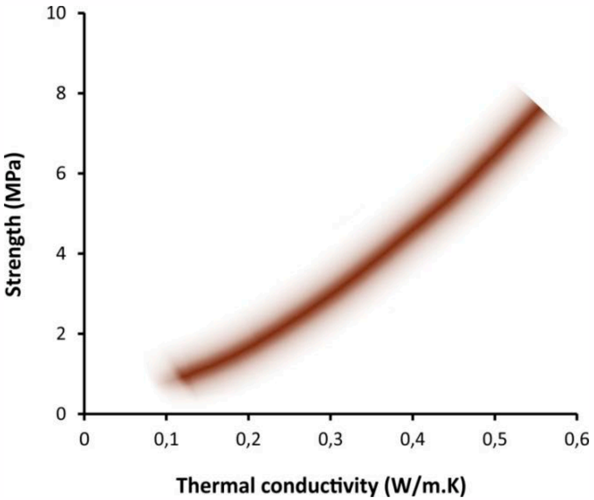
Geopolymer foams :

Foam concrete is a type of a lightweight concrete that consists of a cementitious binder with a high degree of void space.

Air incorporation enables to **lighten** the matter and to confer thermal and acoustic **insulation properties.**

Geopolymer foams can be used as **acoustic and thermal insulators**, **filters** or **light weight fire proof insulators**.

Objective :



Example of an aerated Portland cement mortar

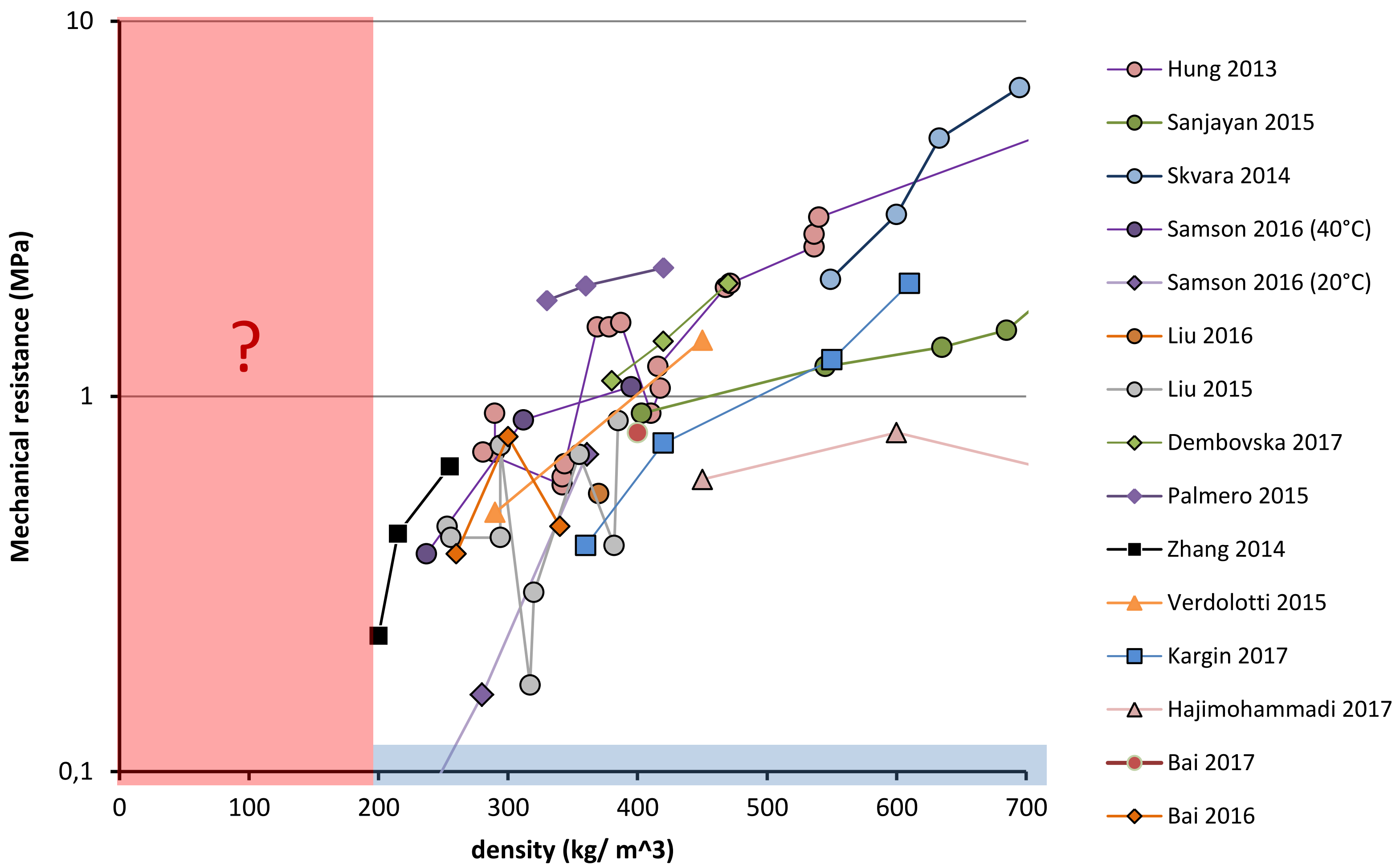
Conciliate mechanical resistance of geopolymer foams and their acoustic and thermal insulation properties

→ **Morphology optimization**

Controlling the **nature**, **size** and **distribution** of voids is the most critical step in the production of these foams since the voids **determine the density and strength** of the foam concrete.

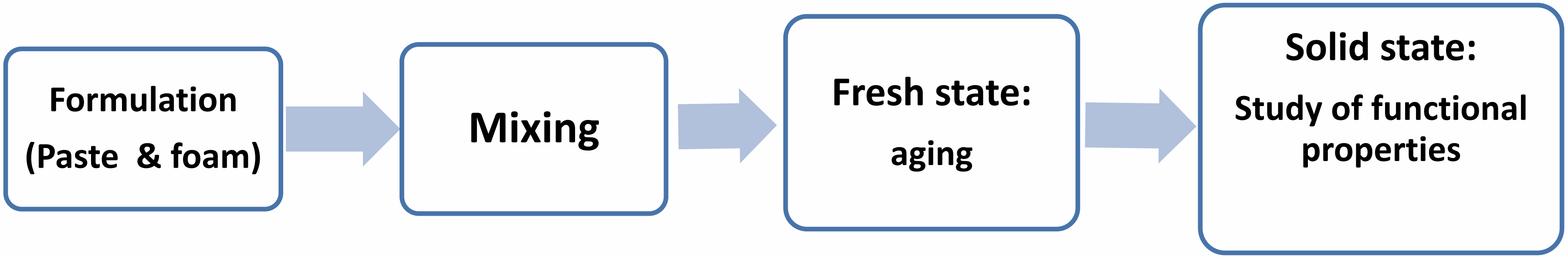
In the literature :

> The densities of Geopoymer foams made till now are over 200 Kg/m^3



Issue:

Major issue encountered during the optimization process: the morphology evolves strongly before setting



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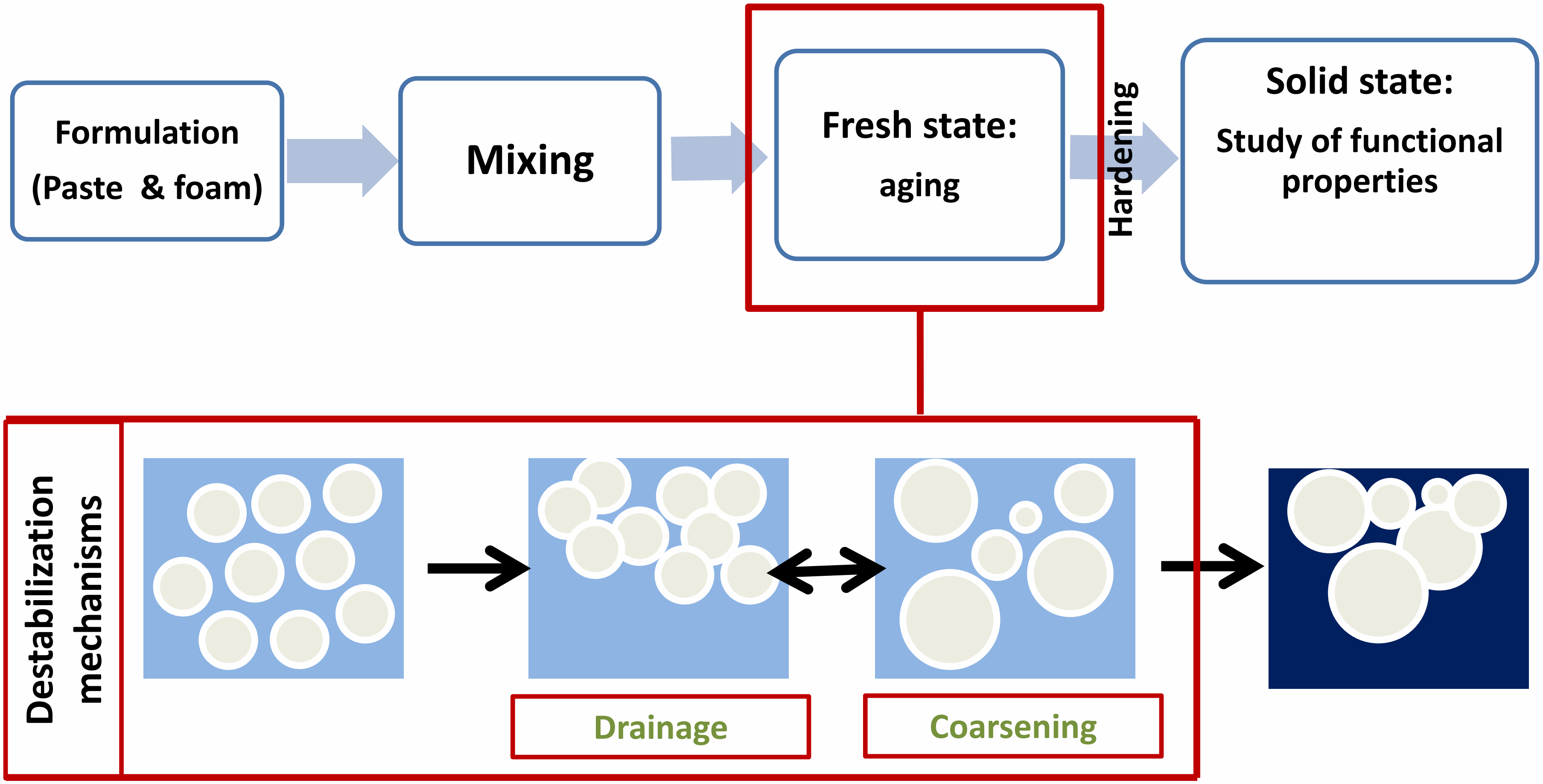

Issue:

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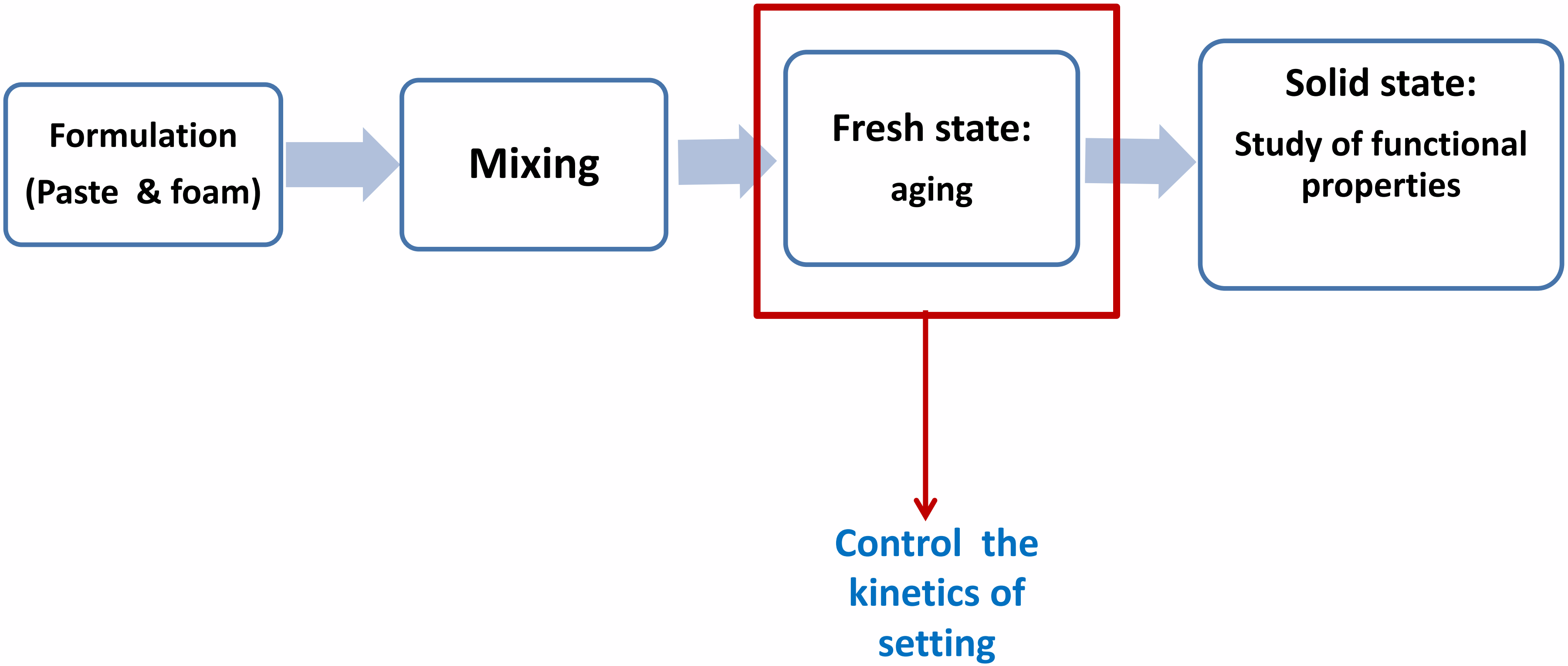
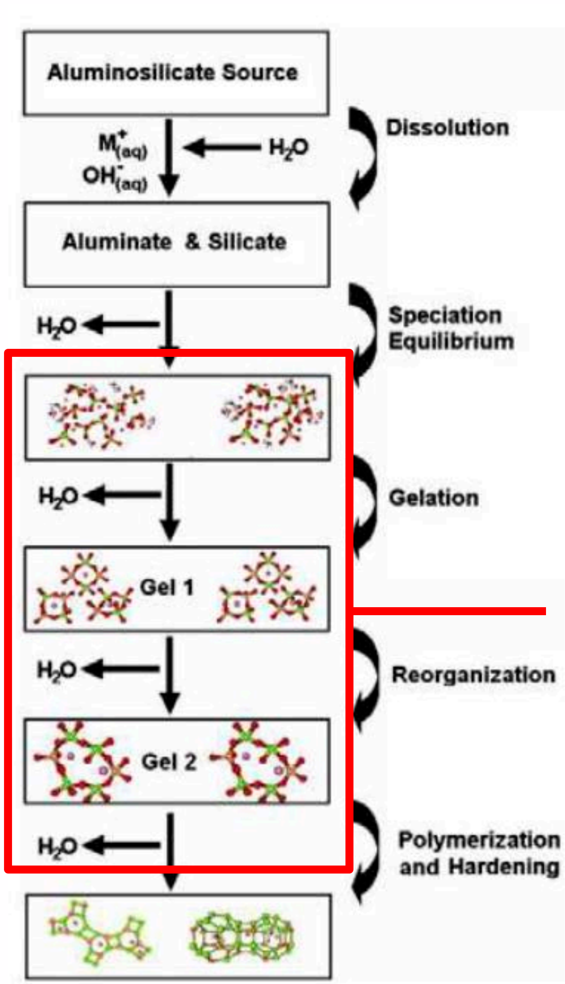
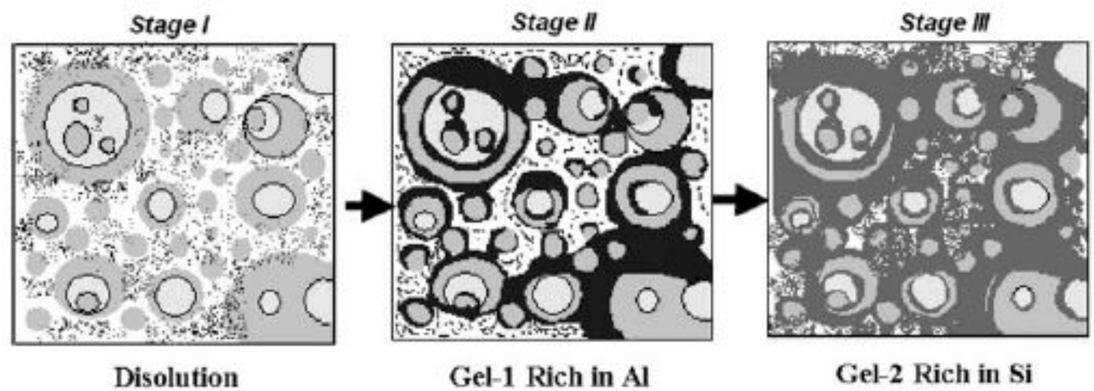


Kinetics of setting and rheology:



Duxson et al. (2006)



A. Fernandez-Jimenez et al. / Microporous and Mesoporous Materials (2006)

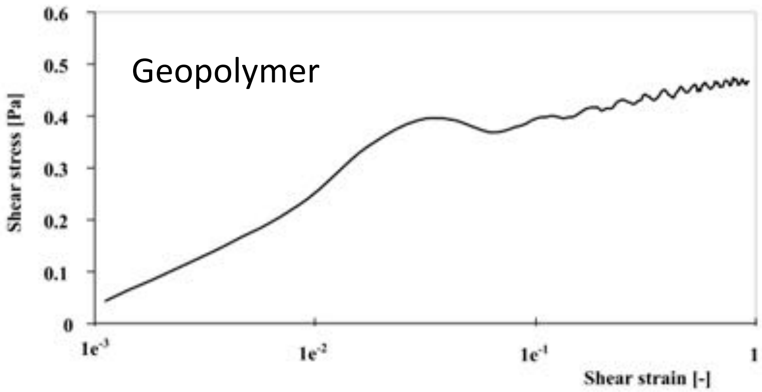
Kinetics of setting and rheology:

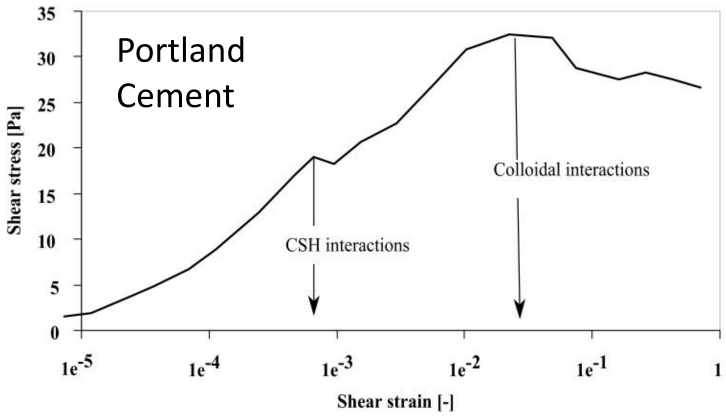
Rheological behavior of fresh geopolymer pastes :

- Geopolymer pastes can be assimilated to semi-dilute suspensions of non-colloidal particles in a Newtonian fluid of high viscosity.

(Roussel & al, 2014)

- Fresh geopolymer pastes have a **very low yield stress.**





A.Favier et al. / Soft Matter (2013)

→ Understanding of the time evolution of rheology of geopolymer pastes is a major issue for the elaboration process

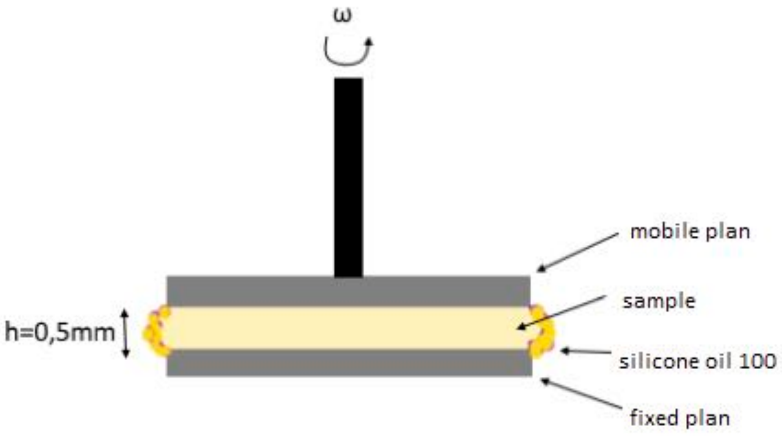
Materials & Methods:

- Metakaolin (Argical M1200s) + Sodium Silicate solution

- Oscillatory rheology (f= 1Hz)

$$\underline{\varepsilon}(t) = \varepsilon_0 e^{i\omega t}$$
$$\underline{\sigma}(t) = \sigma_0 e^{i(\omega t+\varphi)}$$
$$\rightarrow \quad \underline{G}^* = \frac{\underline{\sigma}}{\underline{\varepsilon}} = G' + iG''$$

G' : elastic modulus
G'' : viscous modulus

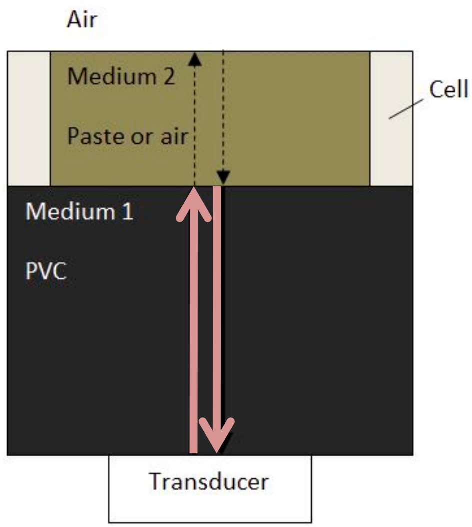


- Ultrasonic rheology (f=100KHz)

$$G' = G = \frac{Z_1^2}{\rho}\frac{(1+R)^2}{[1-R]^2}$$

(Rouyer,2013)

R: Reflection coefficient
Z_1 : acoustic impedance of the medium 1



Methods:

- Proton NMR (H^1NMR) :

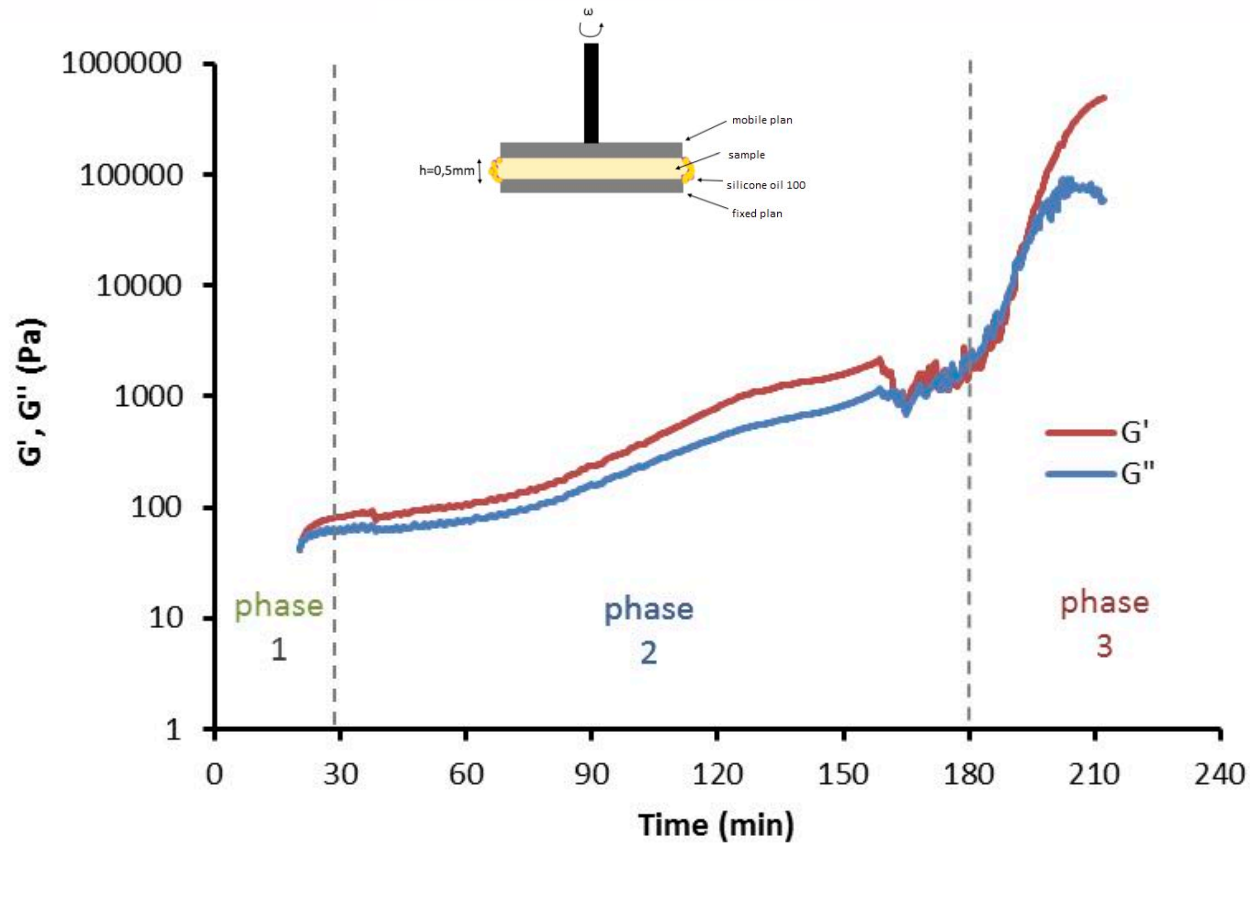
- Monitoring of relaxation times T_1 and T_2 which characterize respectively the times of return to the equilibrium of the longitudinal and transversal components of magnetic moments,

- $T_{1,2} \sim V/S$, already used to characterize the setting of Portland cement.
  (P. Faure et al. / Magnetic Resonance Imaging (2008))

→ Application of Proton NMR to geopolymer pastes.

Bruker Minispec MQ20

Monitoring of the elastic modulus using standard rheology

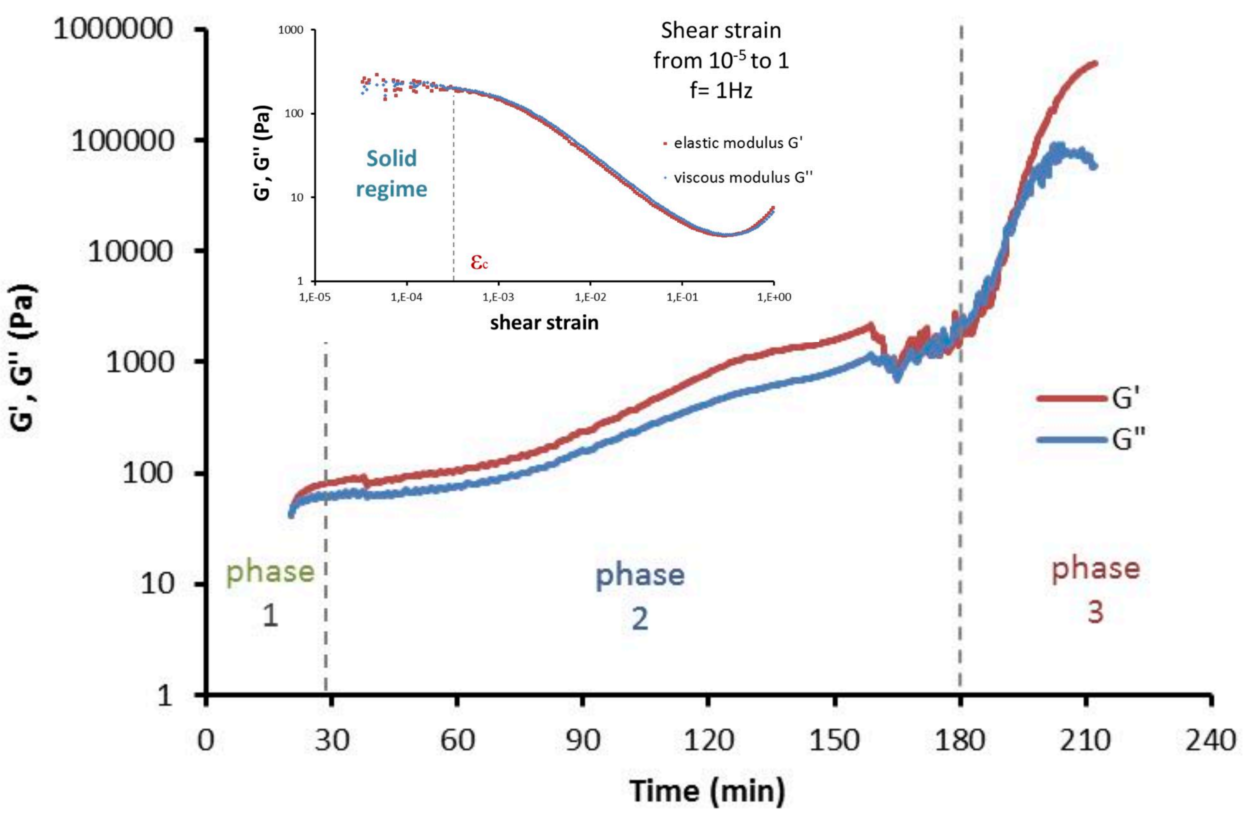


Phase 1 : formation of an Al-rich gel around Metakaolin grains (gel1)

Phase 2 : induction phase

Phase 3 : formation of a gel of a higher connectivity (gel2) → setting

Monitoring of the elastic modulus using standard rheology



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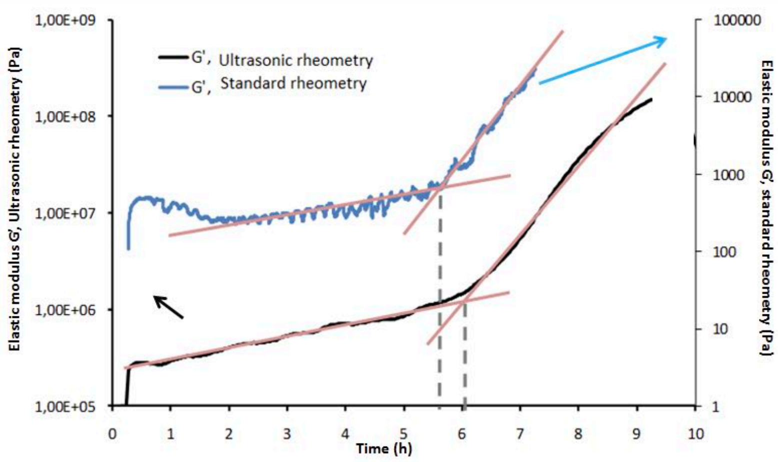
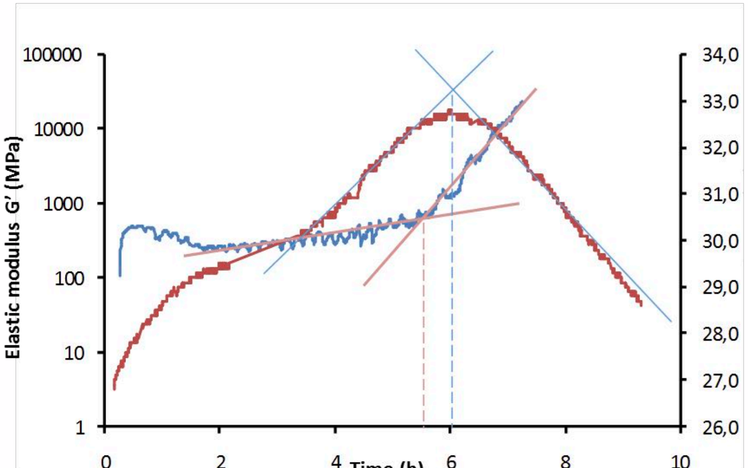
Monitoring of the elastic modulus with:

Ultrasonic rheology (f=100KHz)



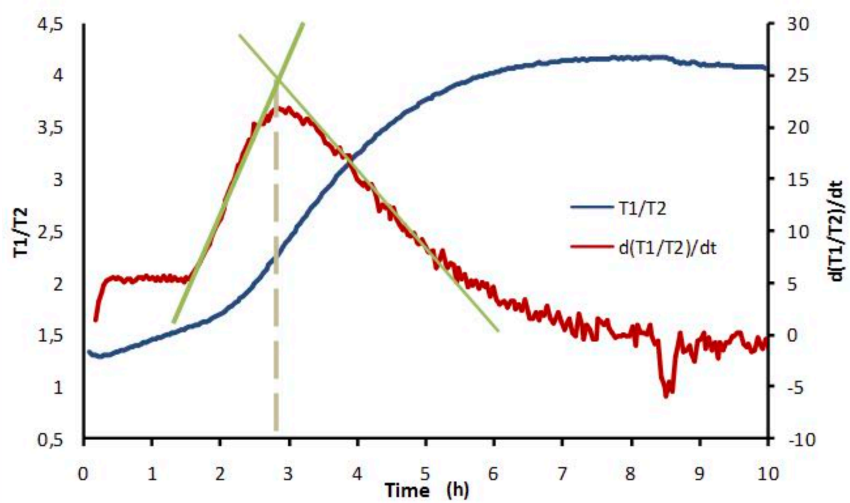
Calorimetry



Proton NMR

Solid
T_1/T_2 ~ 4

Liquid
T_1/T_2 ~ 1,5



- The curve T_1/T_2 illustrates the transition from the liquid phase to the solid phase.

- Proton NMR defines a characteristic time of geopolymer setting : $(d(T_1/T_2)/dt)_{max}$

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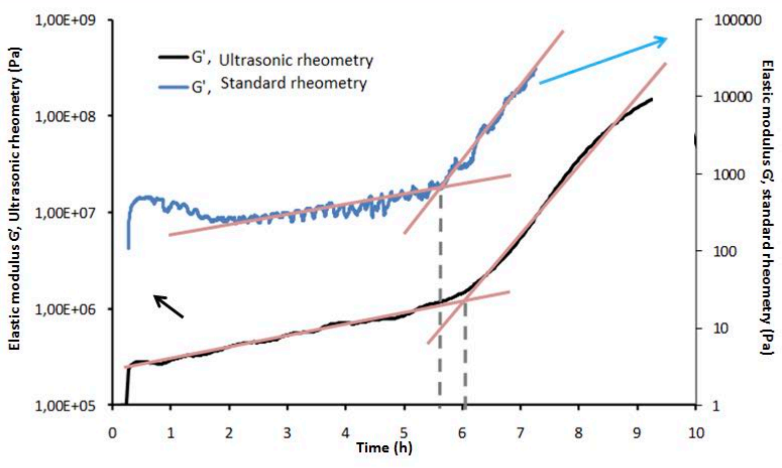

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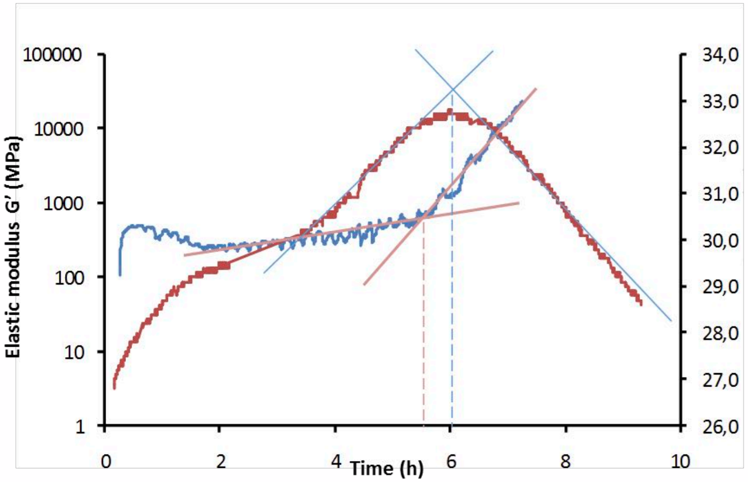
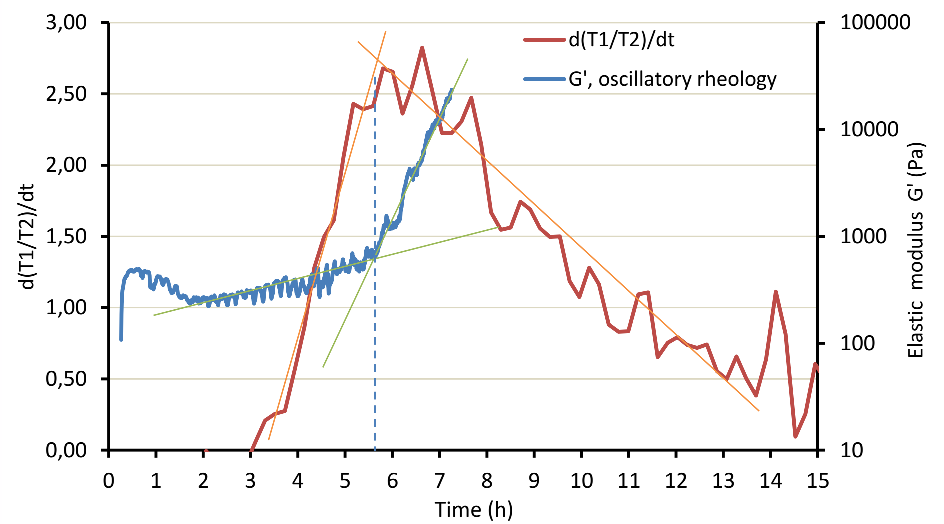


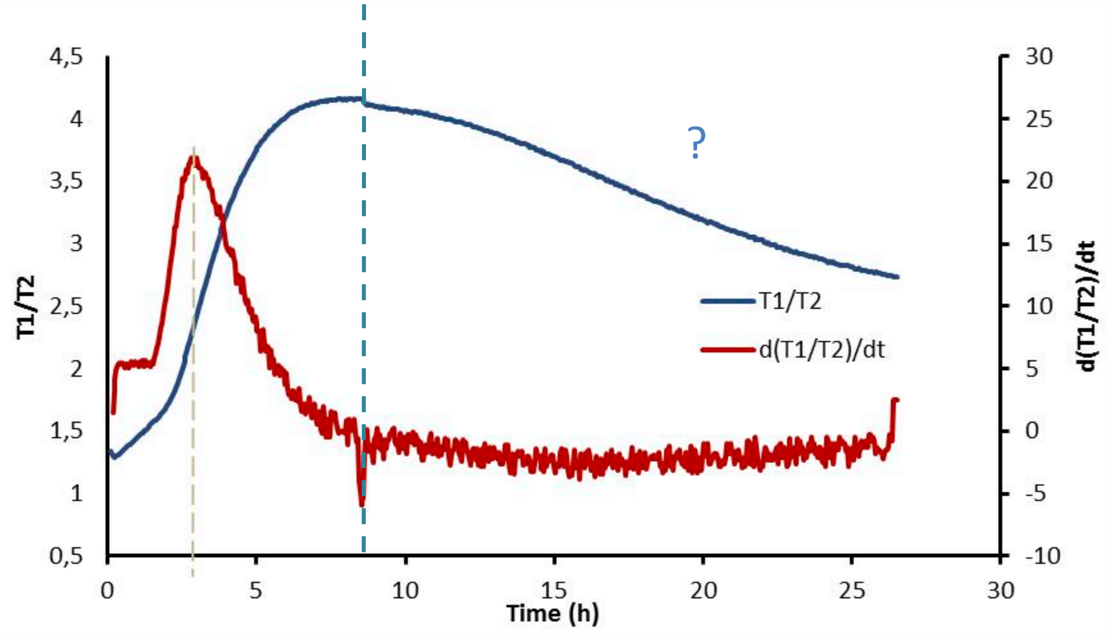
Proton NMR



→Proton NMR is a an indicator of the setting time.

Proton NMR

- Allows to monitor the setting for long periods of time



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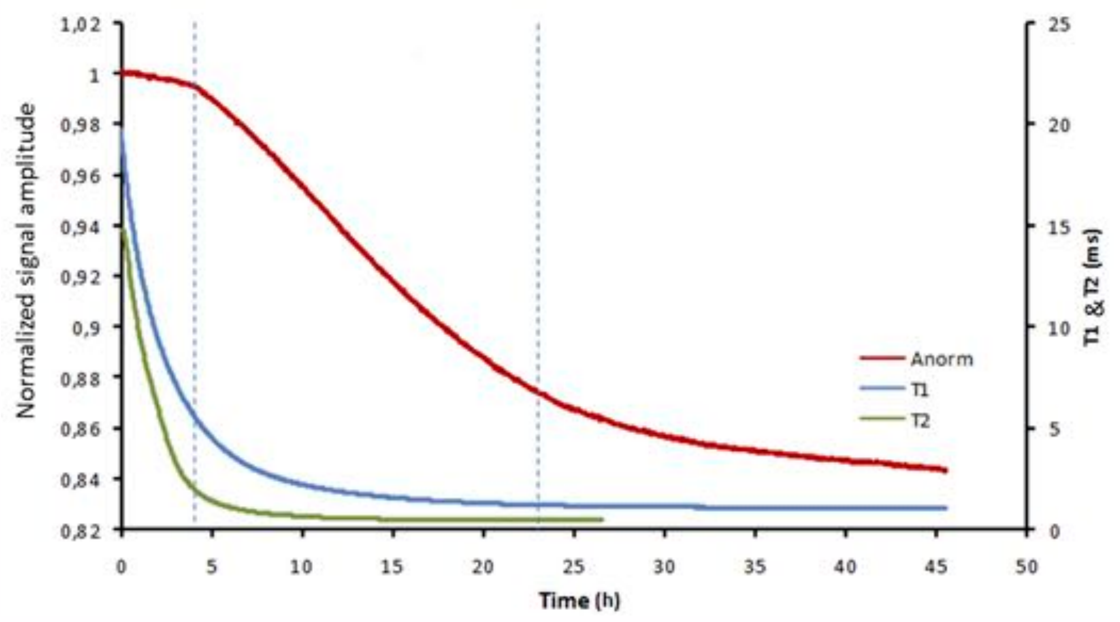
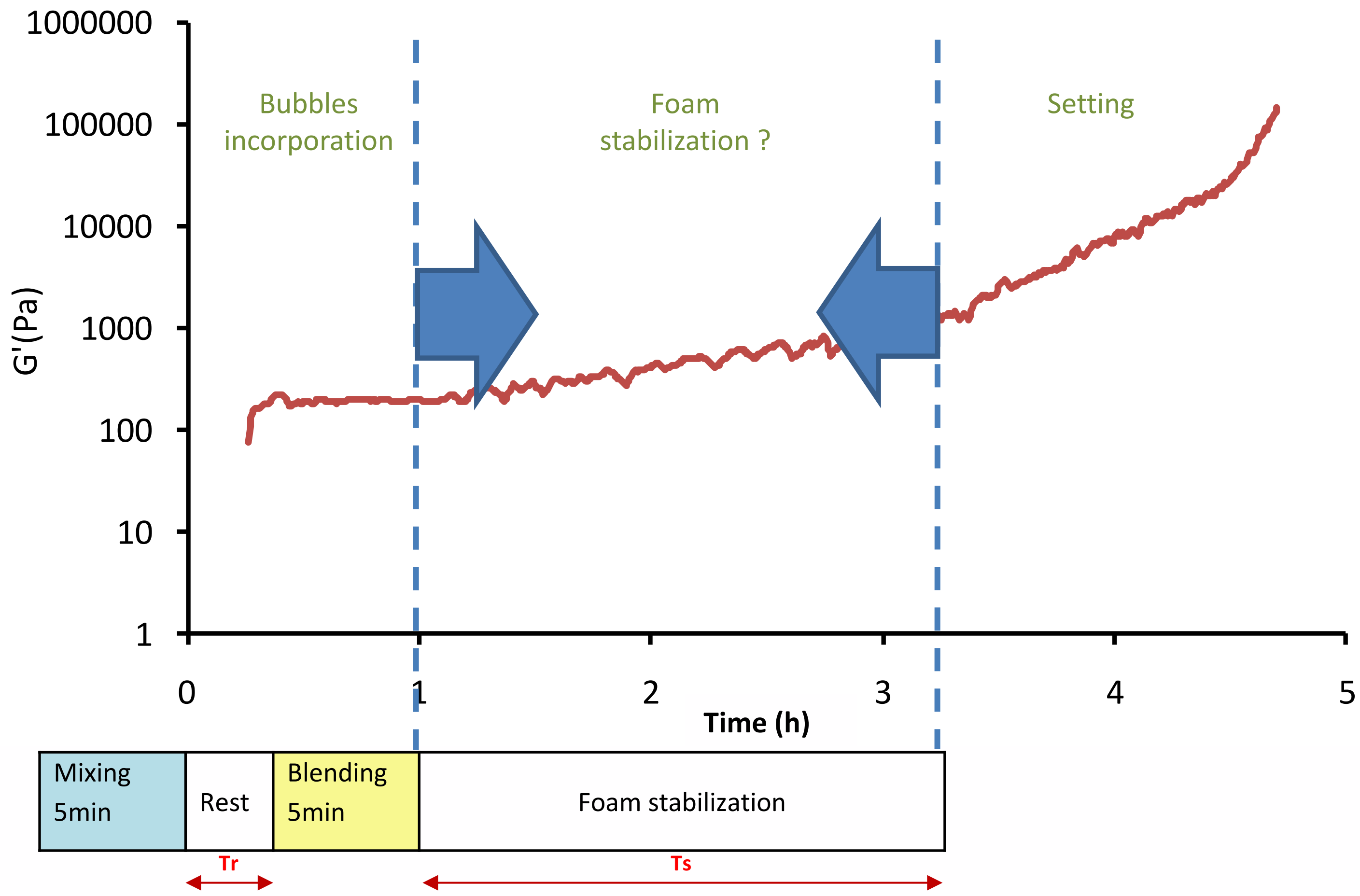
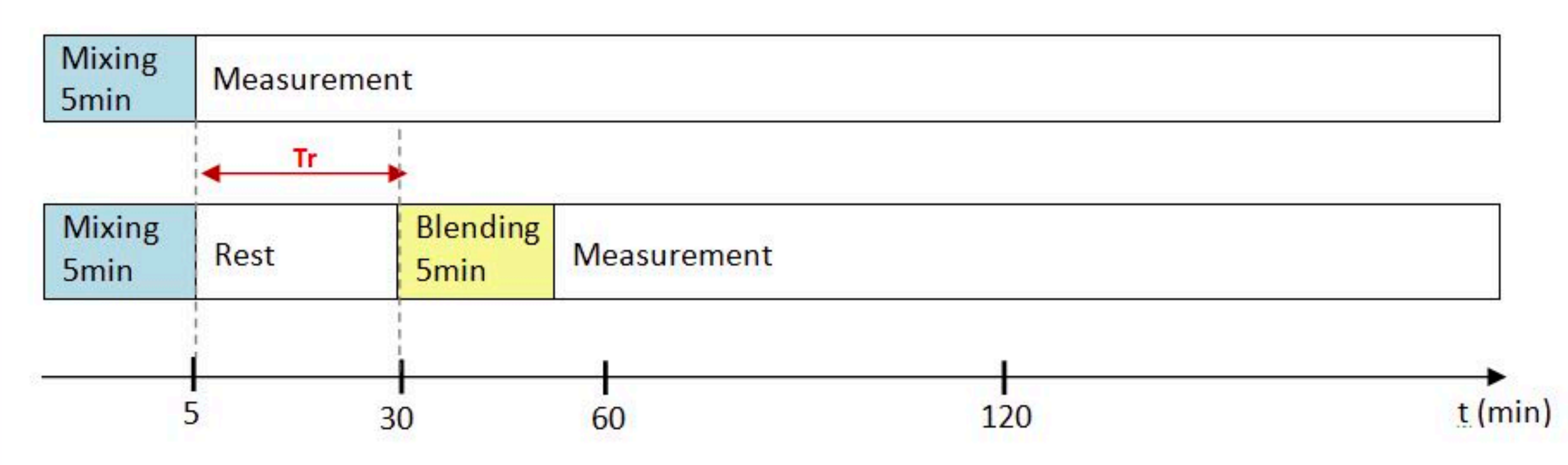


Strategy for bubbles stabilization



Influence of air incorporation on setting time

Sequence for the measurement of the effect of mixing on the time of setting
Second mixing simulates foam incorporation



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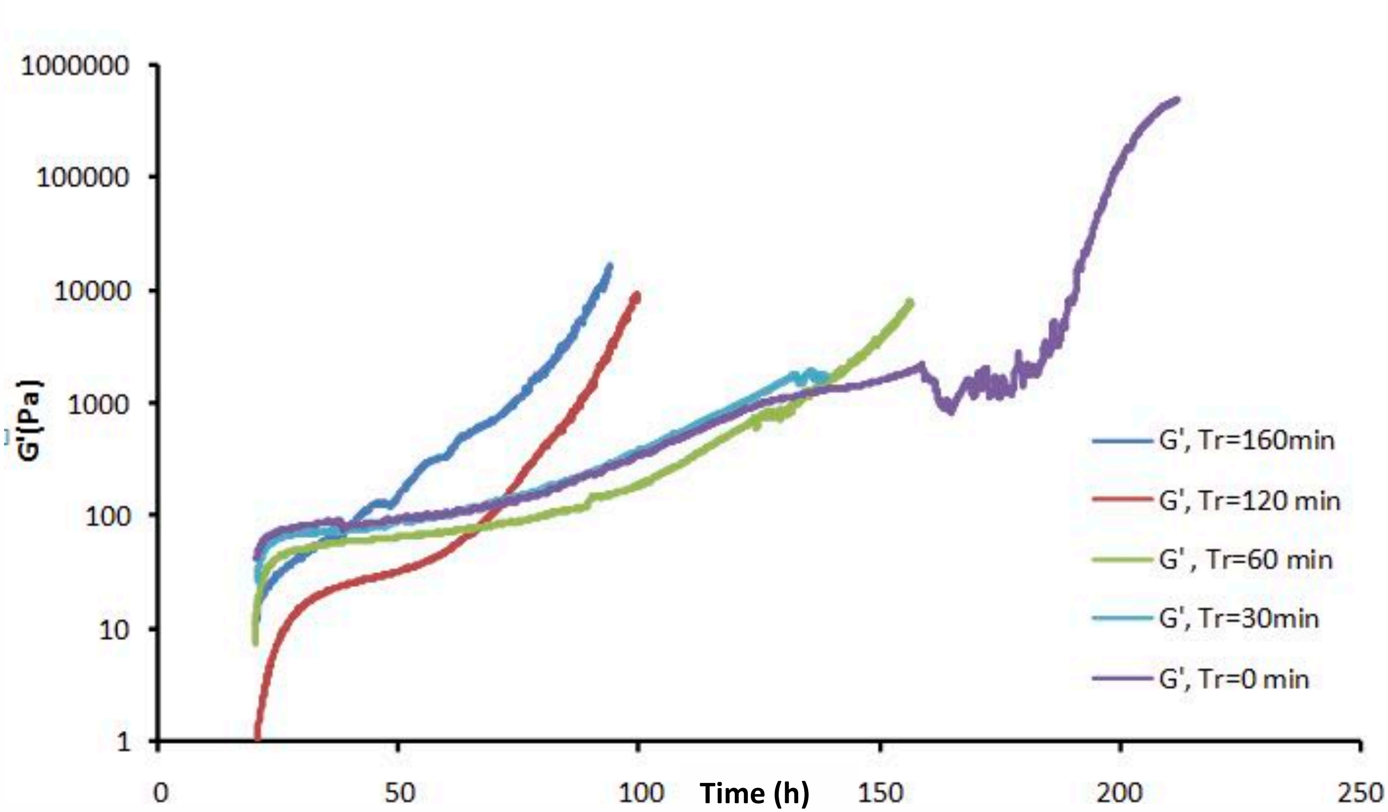
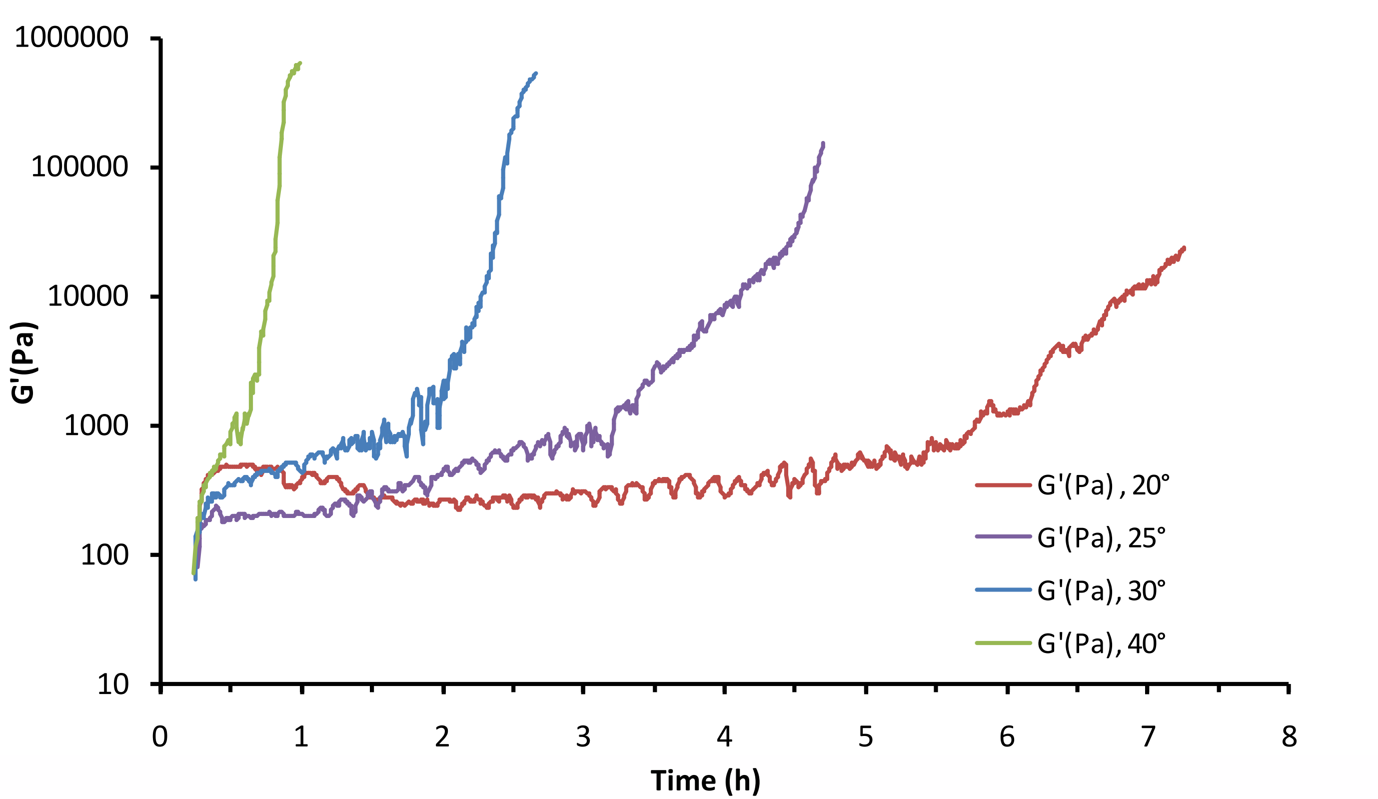


Blending 5min	Measurement of elastic modulus

- Gel 1 reforms systematically after each blending in the same way.
- Time of induction is as much reduced as the time of rest before mixing is important

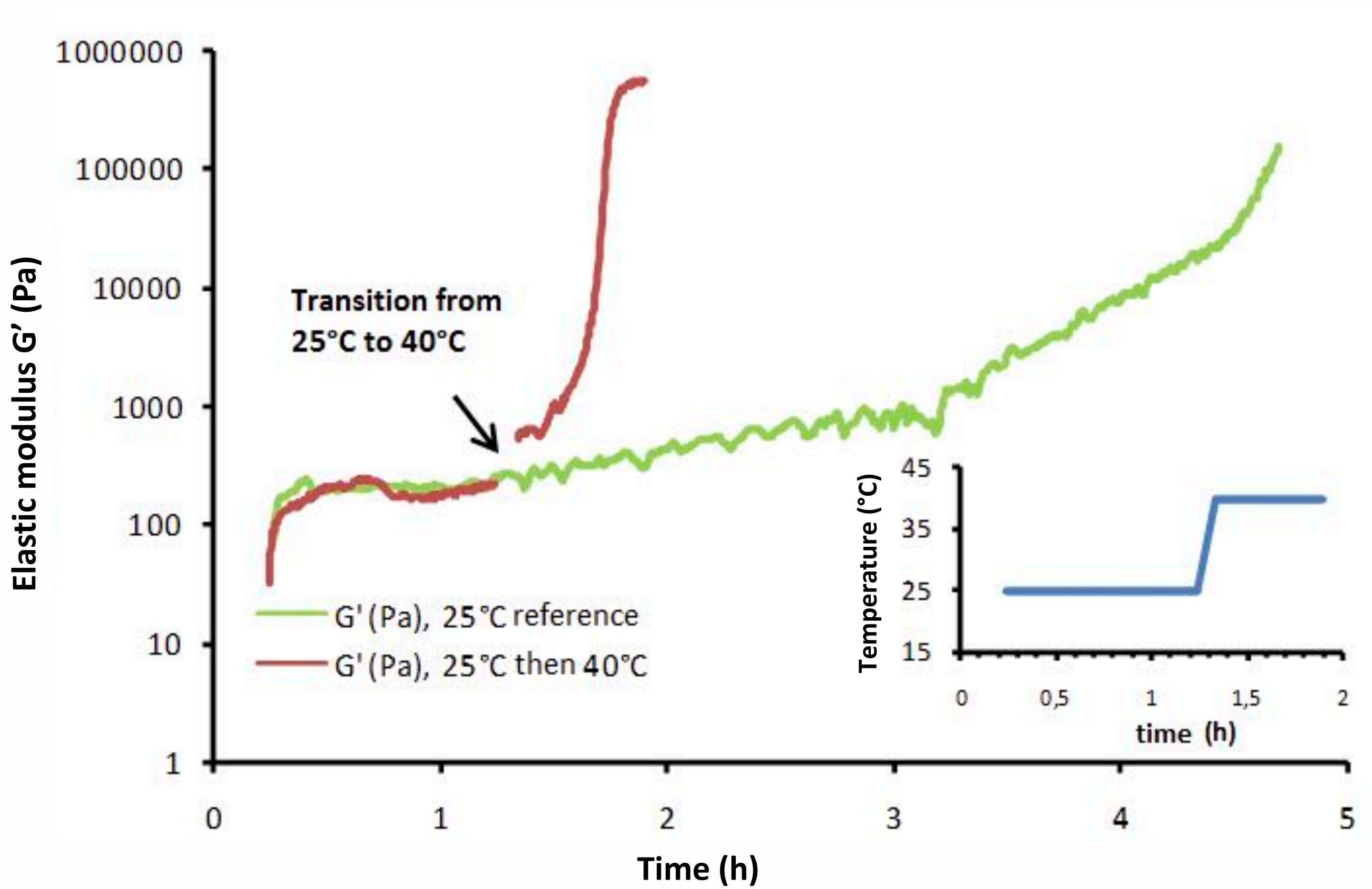
Influence of temperature on the time of setting



The difference between the times t (10000Pa) at 20°C and 25°C is 2h43min

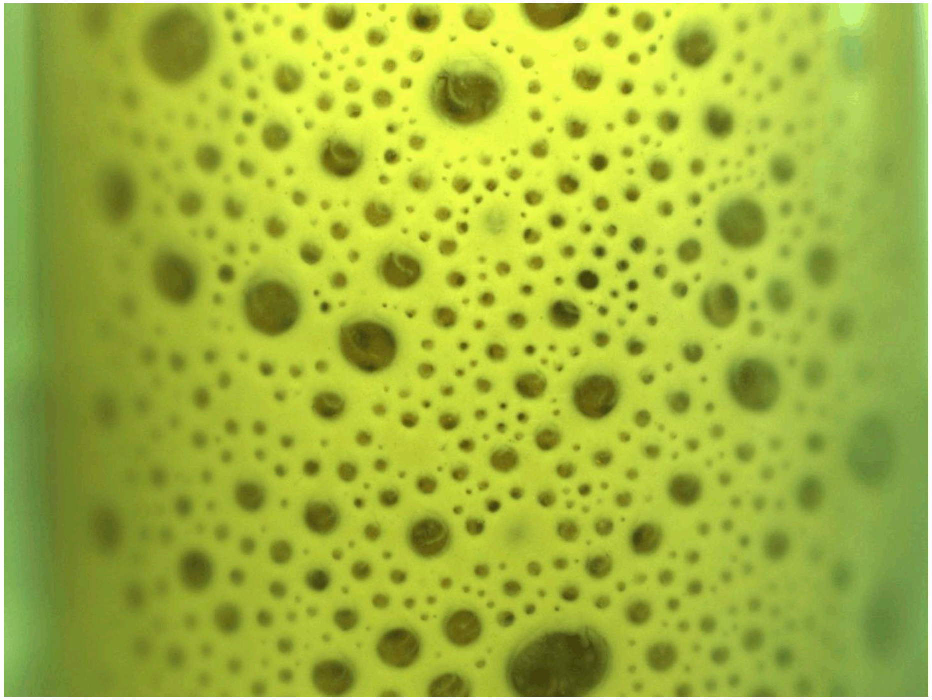
Effect of a temperature step on the time of setting

Unleash the setting with a temperature step from 25°C to 40°C
→ Gain in time of foam stabilization



Preliminary work for geopolymer foaming:

Tween 80 (Sigma-Aldrich)
(M. Strozi Cilla et al. / Ceramics International 40 (2014))



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Thank you !