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5-29-2018

Acid geopolymers materials based on different aluminosilicate sources

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Sylvie Rossignol, Jenny Jouin, Virginie Mathivet, and Michel Parlier, "Acid geopolymers materials based on different aluminosilicate sources" in "International Conference on Alkali Activated Materials and Geopolymers: Versatile Materials Offering High Performance and Low Emissions", J. Provis, University of Sheffield C. Leonelli, Univ. of Modena and Reggio Emilia W. Kriven, Univ. of Illinois at Urbana-Champaign A. Boccaccini, Univ. of Erlangen-Nuremberg A. Van Riessen, Curtin University, Australia Eds, ECI Symposium Series, (2018). <http://dc.engconfintl.org/geopolymers/84>

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Science des Procédés Céramiques
et de Traitements de Surface

new name



Acid geopolymmer materials based on different aluminosilicate sources

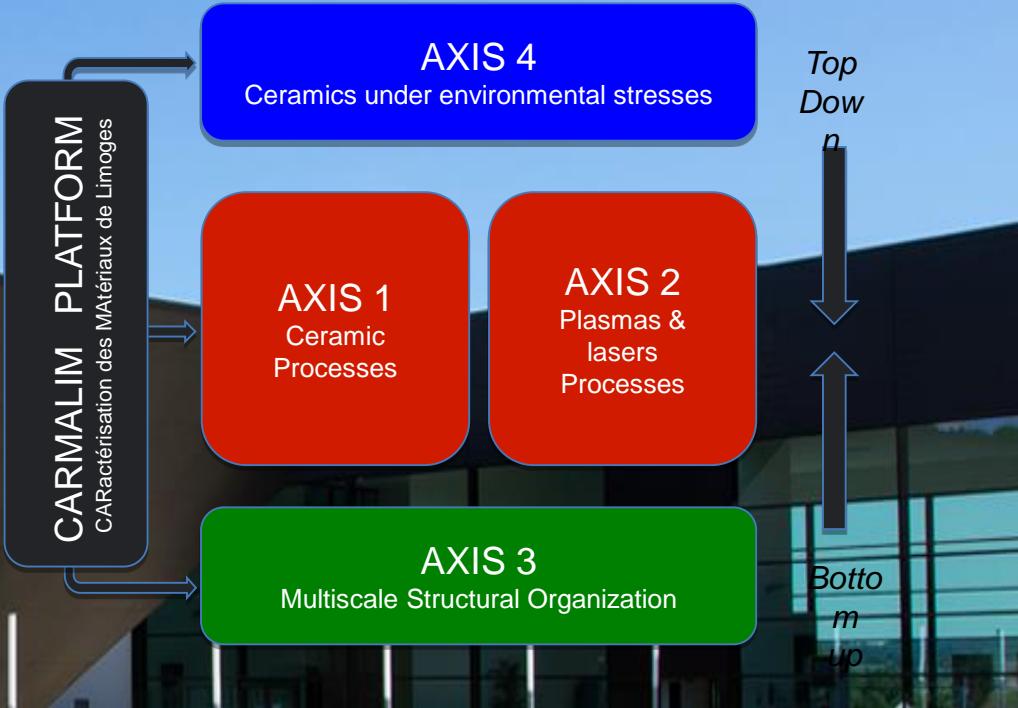
ROSSIGNOL Sylvie

with:

Hélène Célérier, Virginie Mathivet
Nicolas Tessier-Doyen, Jenny Jouin



IRCER
200 pers.
8200 m²



→ Comprehension / mastering of elaboration (ceramic processes and surface treatments) of real objects with new or improved properties

Context

Materials in drastic conditions



refractory



aerospace



⇒ Thermal behaviour, water resistant, mechanical properties?

Geopolymer binders

Synthesis:

Alkaline silicate solution



Dissolution of the mineral source



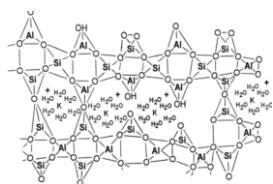
Oligomer formation (Si – Al)



Polycondensation



3D network(Na, K)

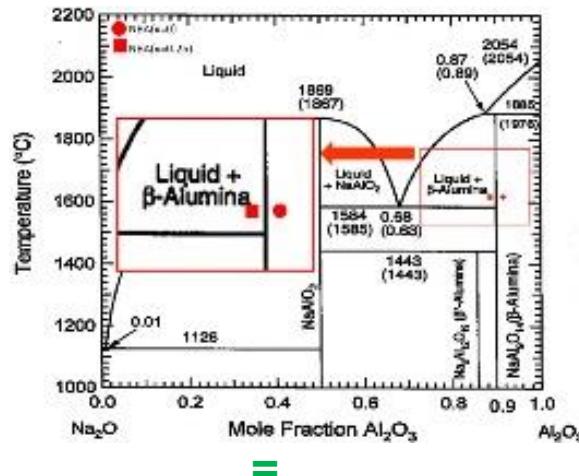


Use of alkaline prohibited

in presence of alumina as example



Degradation in temperature

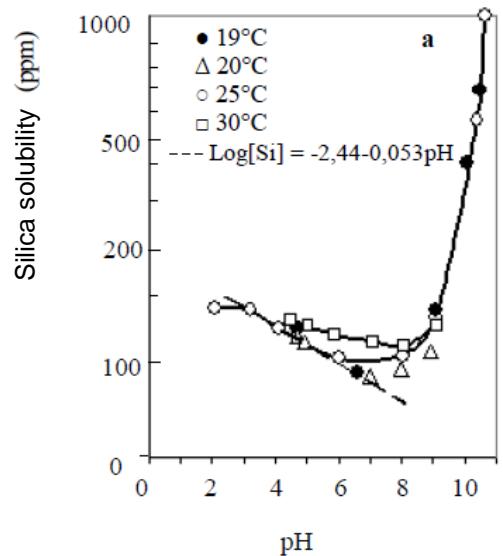


viscous flow

Necessary to change → choice of another way

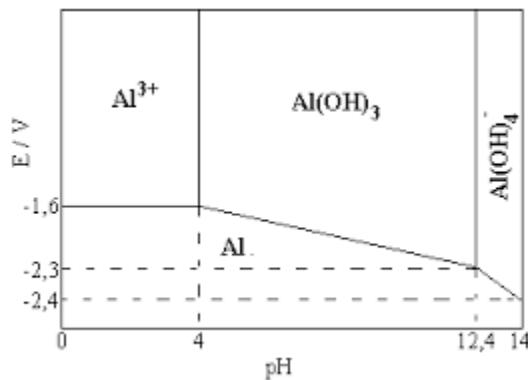
Aluminosilicate sources

Si species



Silica dissolution
only basic medium

Al species



Alumina dissolution
acid and basic medium

Difficulty in acid medium?

Previous works on acid geopolymers

Phosphoric source¹

Various concentration 4-12 M
Homogeneous samples

High compressive strength value to 10M

Disused phosphoric acid²

High compressive strength value = 67 MPa

Resistance in high temperature 1300°C

⇒ What happens in presence of several formulations and how to understand the chemical composition effect?

Aim and tools

Determination of the existence domain



Evaluation of the thermal
and water resistance

- Identification of several compositions

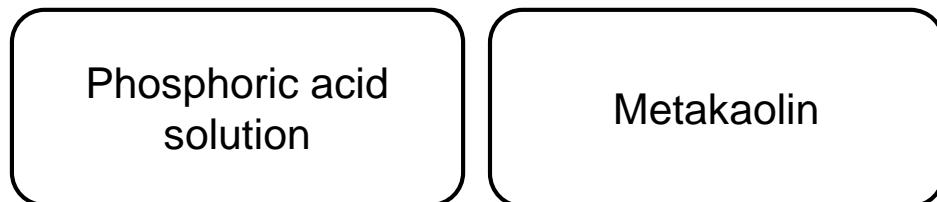


Characterization of the
samples

- Amorphous state
- Water amount
- Mechanical behaviour

Samples preparation and identification

Protocol



Mixing

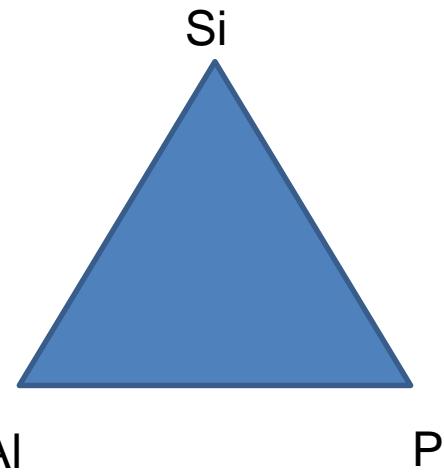
Reactive mixture

*Casting, closed mold,
different temperatures*

Consolidated material



Temperature 20 to 70°C
Various Al/P and Si/P



Different metakaolin
(M1, M3, M4)

Thermal resistant : 1000°C (1h)
Water resistant: 24h in water

Characterization methods

Evaluation after thermal and water resistance

Visual observations

- Presence and size of cracks

✓ good resistance



~ medium resistance

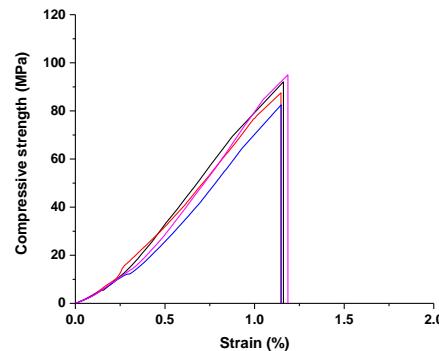
✗ lack of resistance

Compressive strength measurements

- Lloyd EZ20 apparatus



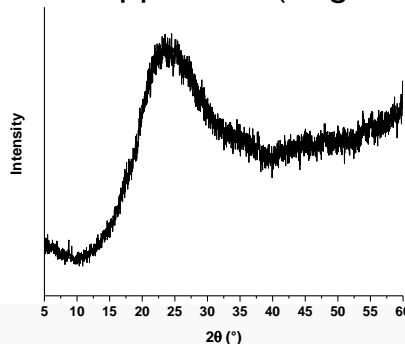
σ compressive tests



Characterization of the samples

X-ray diffraction

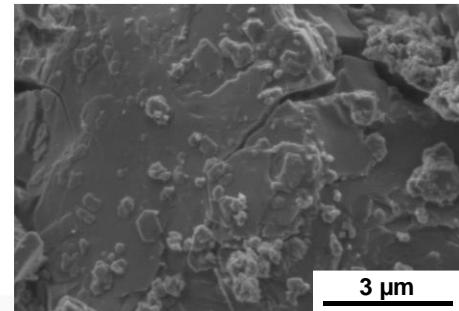
- Brucker D8 apparatus (angular range: 5 to 60 °)



X-Ray pattern of raw metakaolin M3

Scanning electronic microscopy

- JEOL IT-300 (carbon coating of the samples)

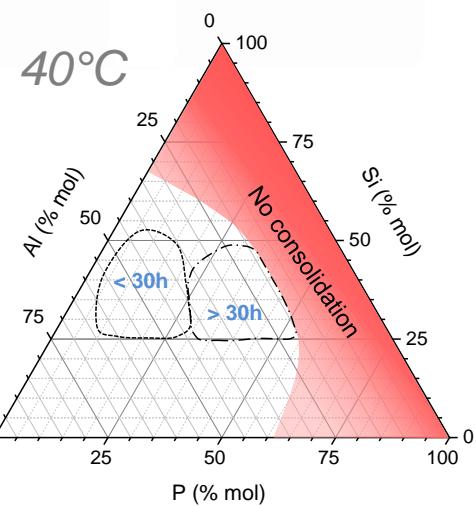
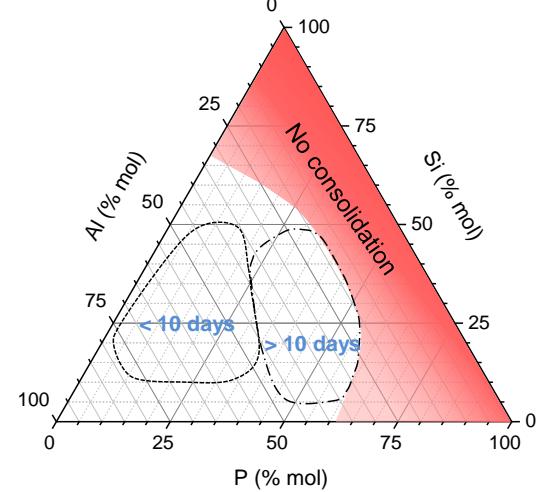


Acid based-geopolymer formed with M3

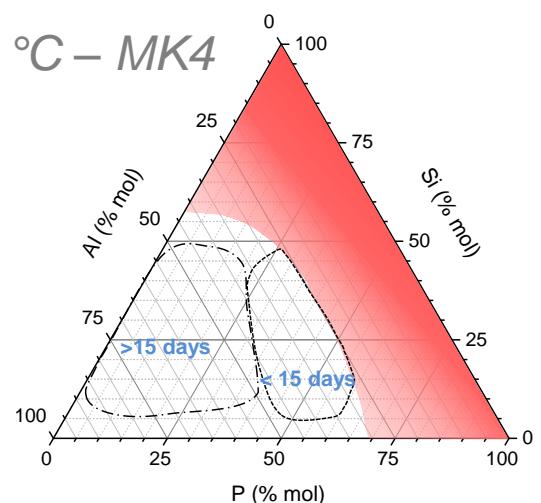
Impact of the temperature and the metakaolin

Time of consolidation

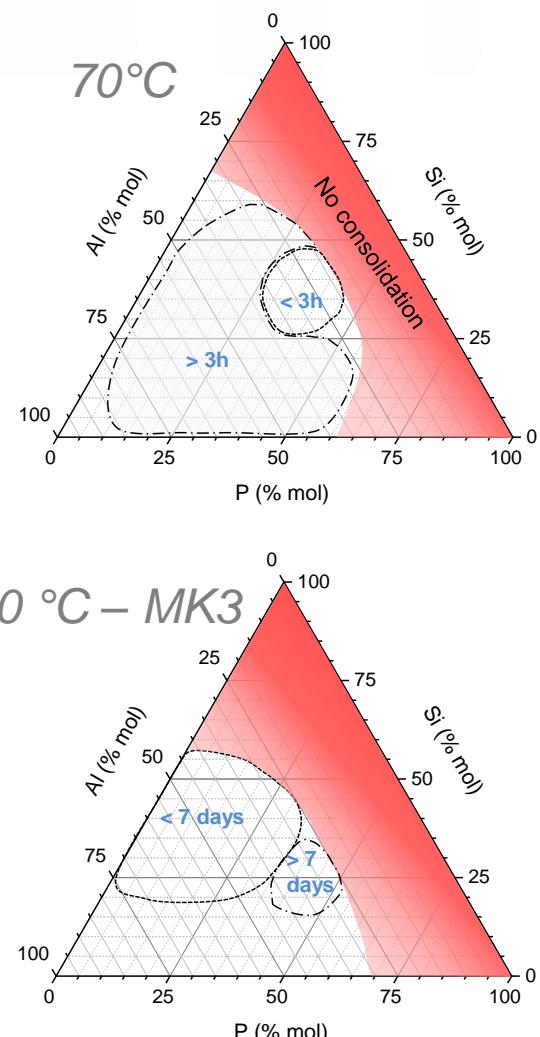
20 °C - MK1



20 °C - MK4



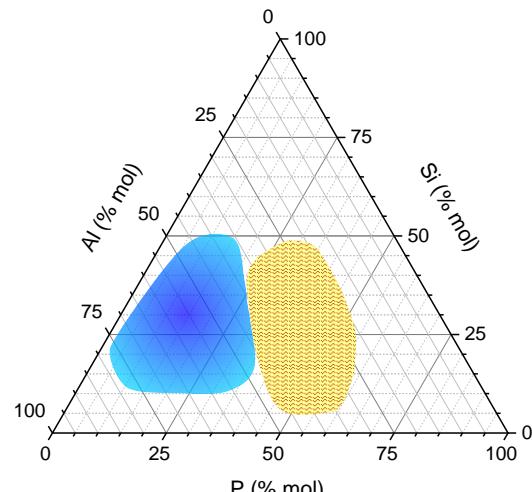
20 °C - MK3



⇒ Several zones controlled by temperature and metakaolin reactivity

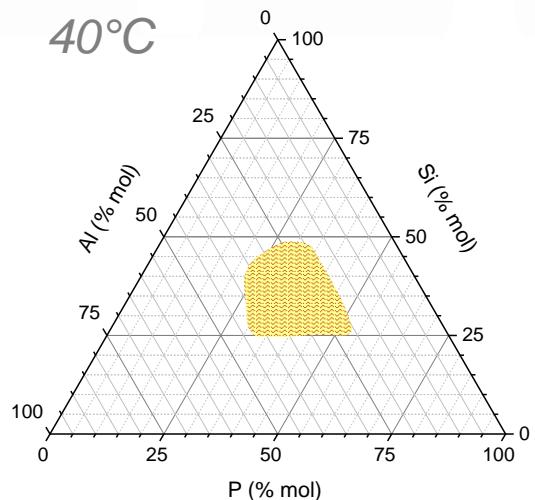
Impact of the temperature and the metakaolin

20 °C - MK1

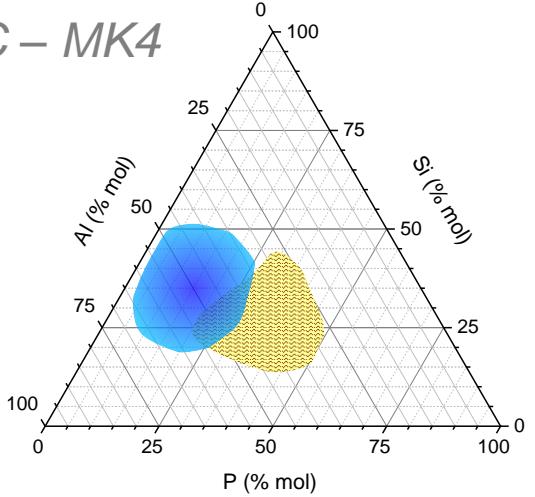


Water resistant
Fire resistant

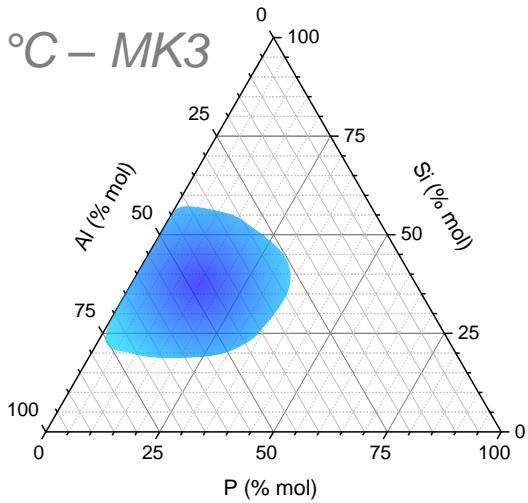
40 °C



20 °C - MK4



20 °C - MK3

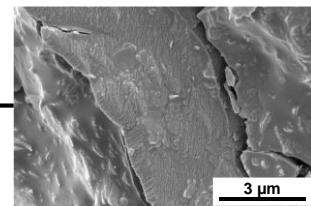
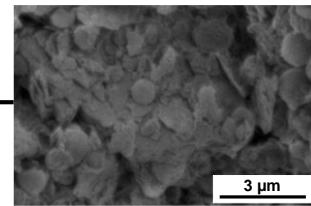
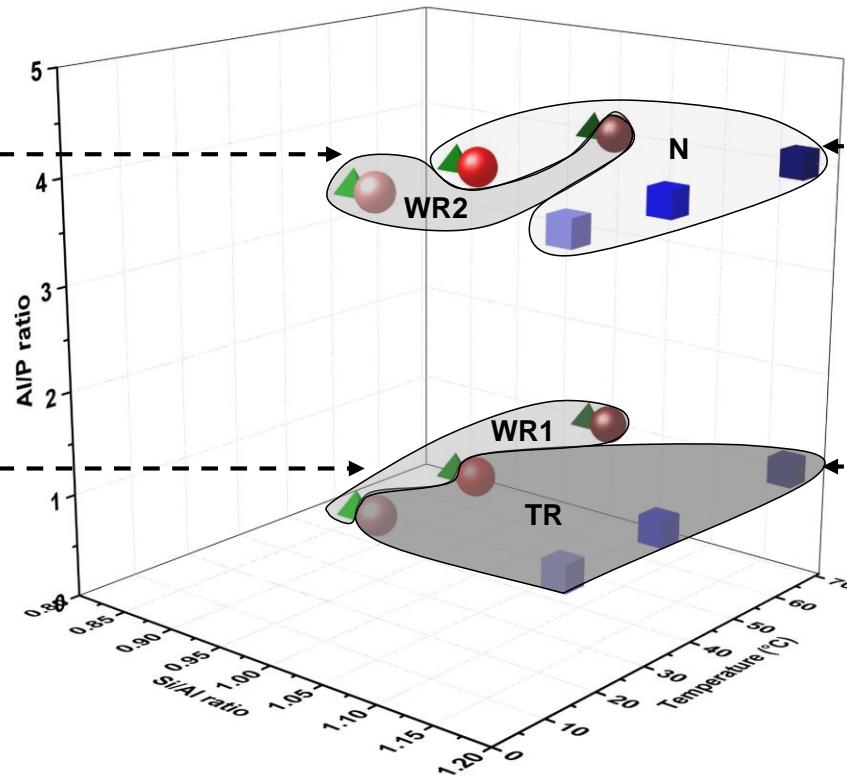
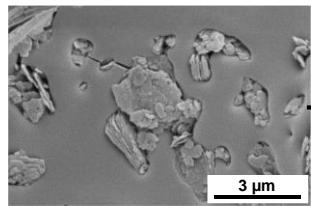
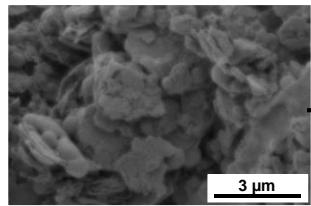


⇒ Working properties dependant on T and MK

Three family

Depending on composition

TR thermal resistant, WR water resistant, N none

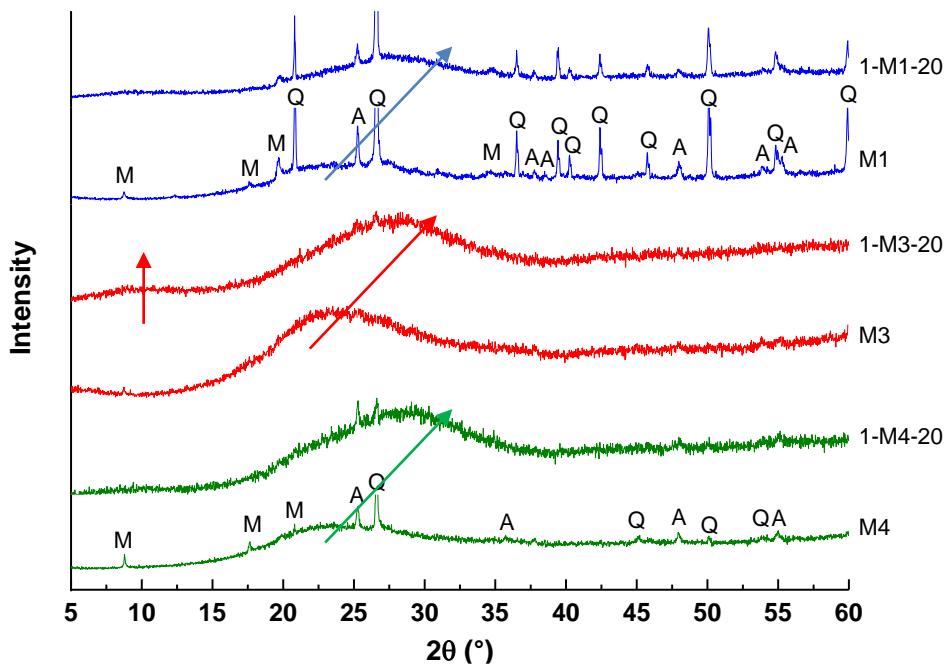


Polycondensation reaction

⇒ How to explain the final properties?

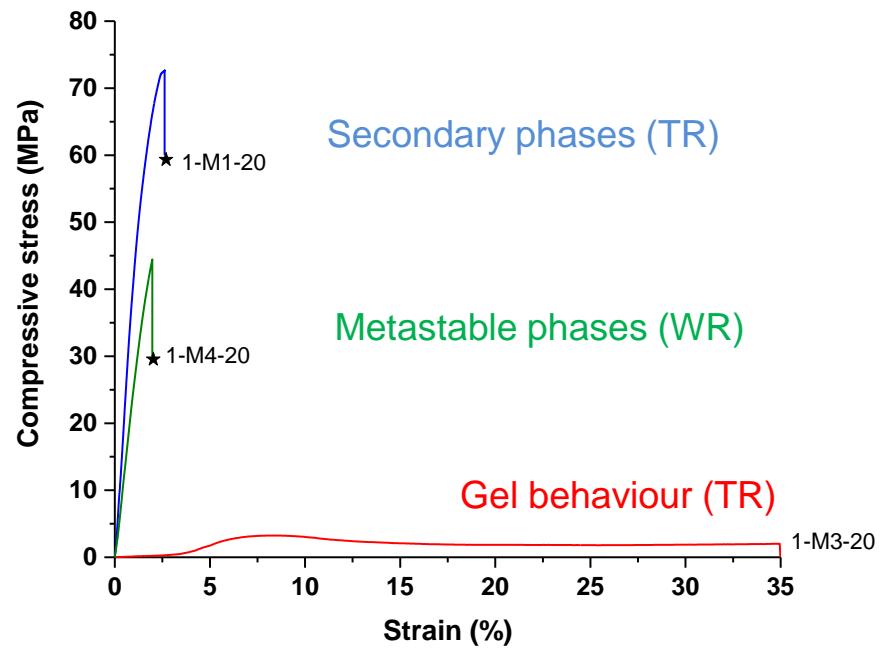
Metakaolin influence

X-ray data



- Amorphous state Al-O-P-Si?
- Another network: silicate species

Compressive strength measurements

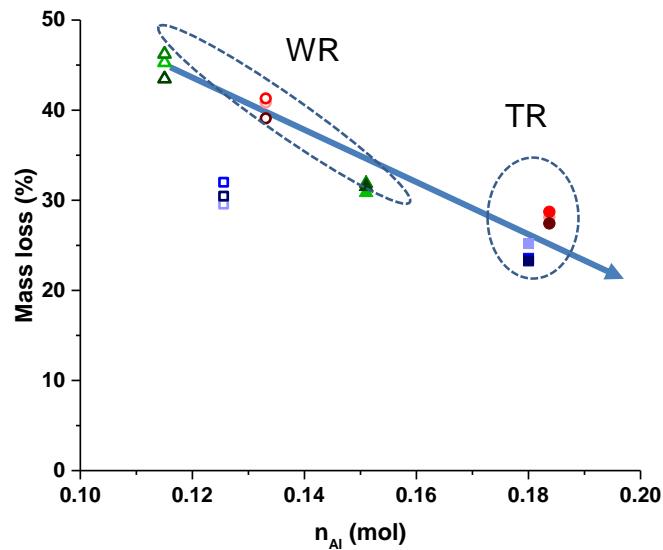


- Metakaolin reactivity

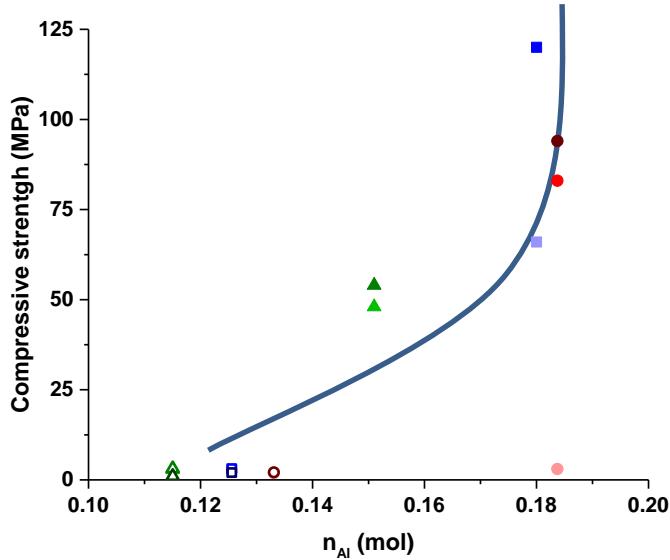
⇒ Dissolution of Al species governs the final properties

Metakaolin influence

Thermal analysis



Compressive strength



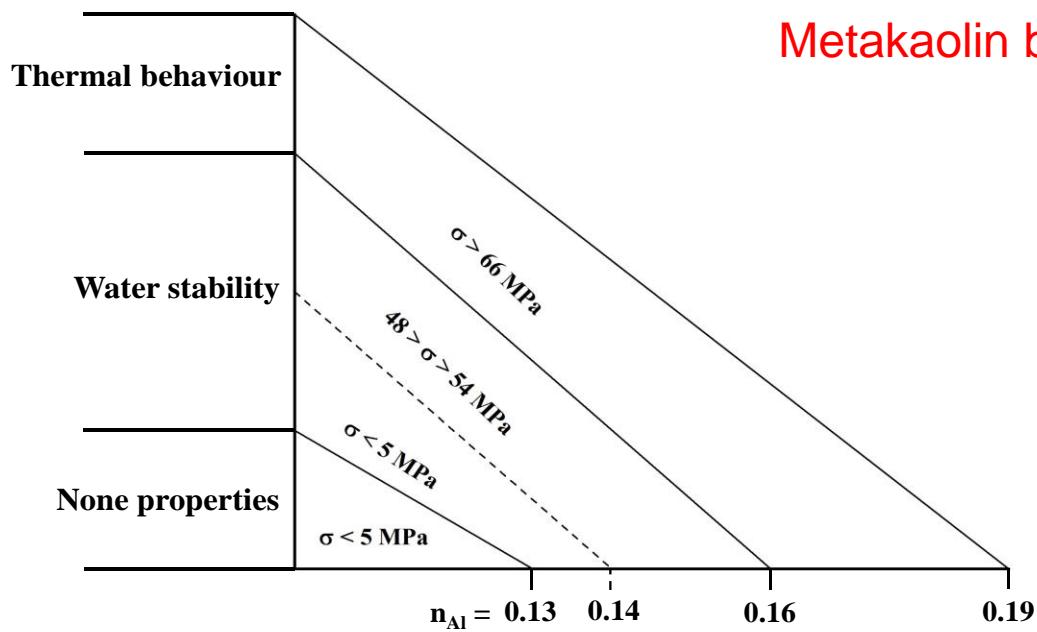
- $n_{Al} < 0.16$ WR and $n_{Al} > 0.16$ TR

⇒ Polycondensation reactions govern

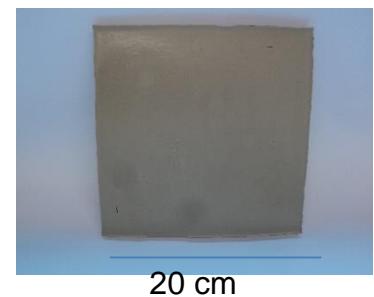
Conclusion

Summary

- Various compositions are tested.



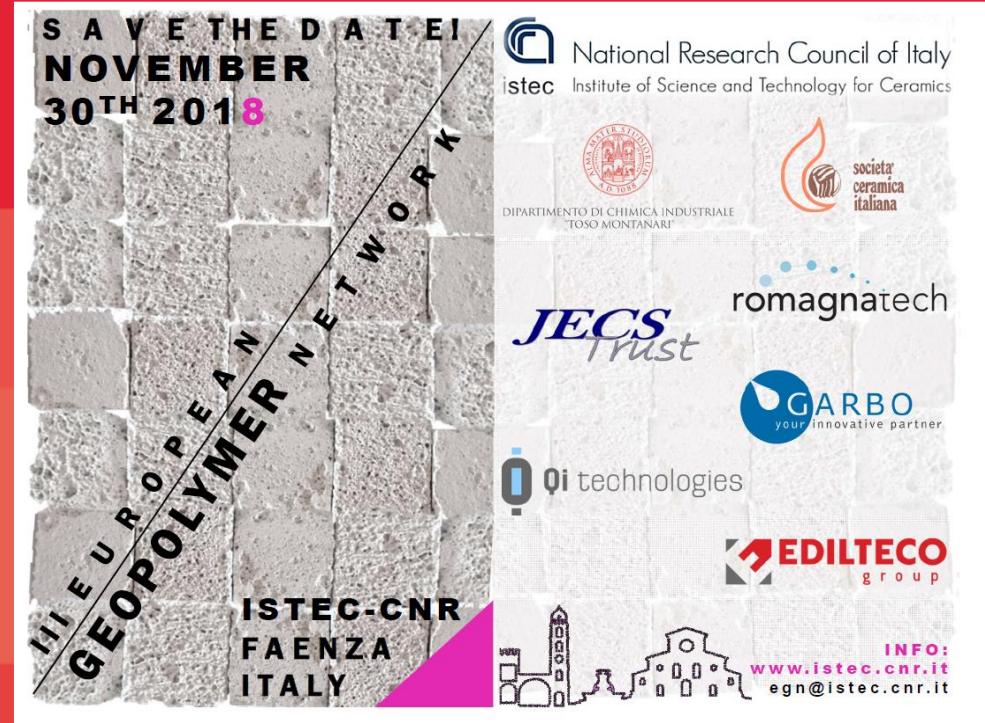
Metakaolin behaviour



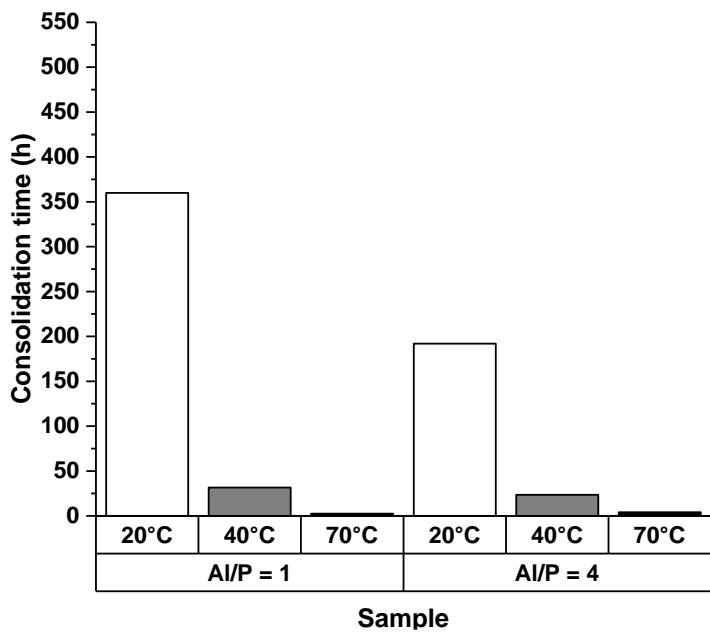
Outlook

- Understanding the formation of each compound

Thanks for
your attention.



Avec M1



Pour Al/P = 1

