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Evaluation of assembled organics liquids incorporation in geopolymer matrix: case of viscous oils and greases

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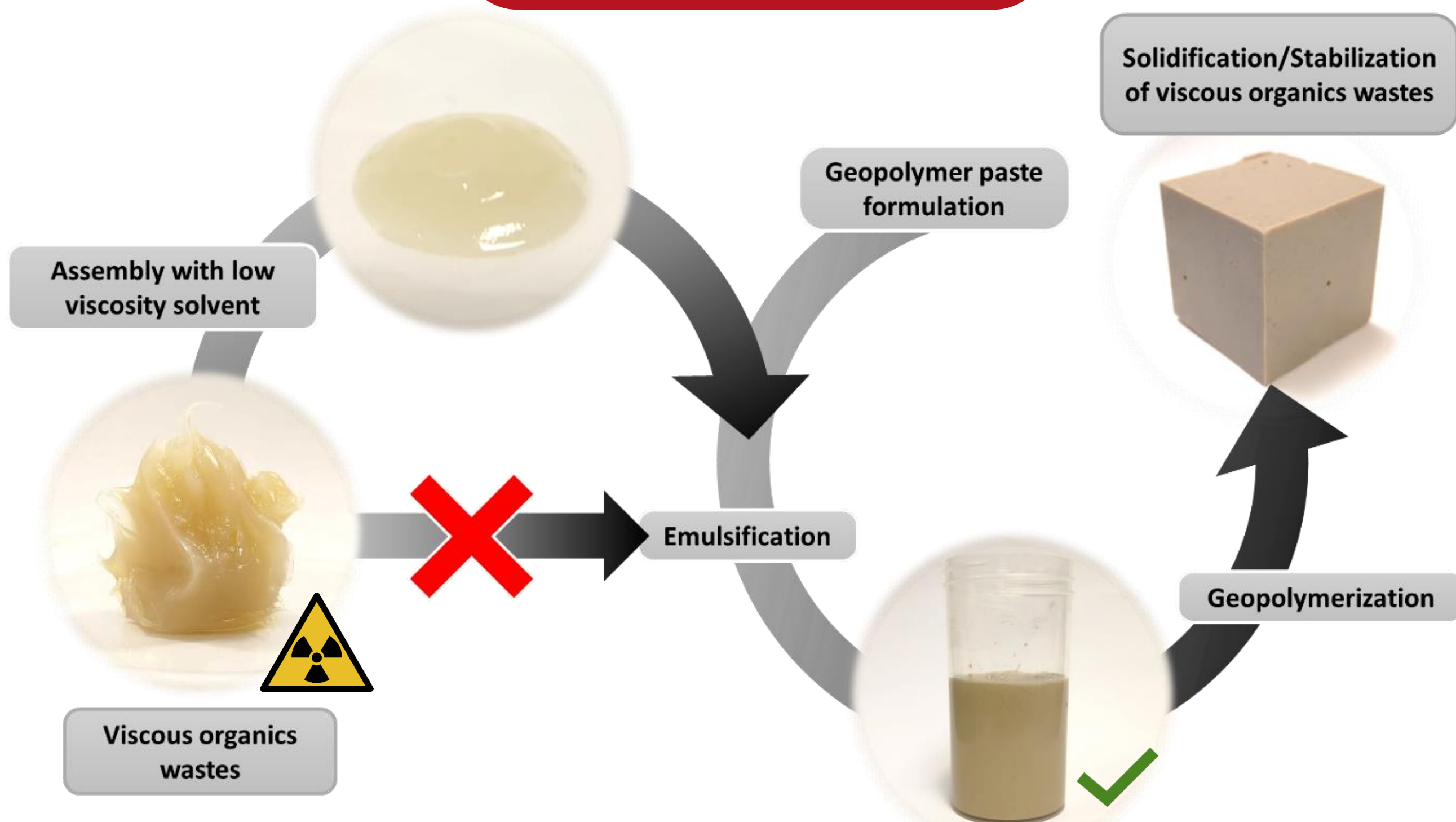
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Introduction



Issue: Processing of historical nuclear waste with a high viscosity → Direct Solidification/Stabilization route impossible

Solution: Assembling with low viscosity solvent as dodecane (C12) or mix of TBP/C12 (30/70% Vol. respectively) for a double elimination

Previous works: Possible incorporation of oil in literature^{[1]-[6]}

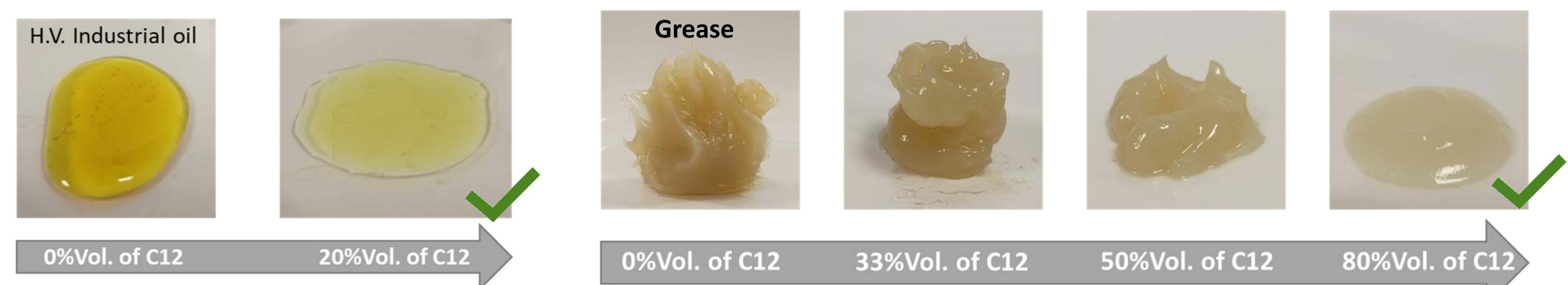
Methodology

- **Aim:** incorporation of 20%Vol. of assembled oil and grease into geopolymer
- **Rheological characterization** for assembled oil and grease with low viscosity solvent (choice of assembling rate), comparison between C12 and the mix of TBP/C12
- **Micro-calorimetry analysis** of geopolymer with assembled oil and grease
- **Microstructure analysis** of droplets network (MEB and micro-tomography)
- **Compressive strength characterization** at 28 days (aim more than 8 MPa)
- **Leaching behavior** with organic carbon measurements in lime water (under study)

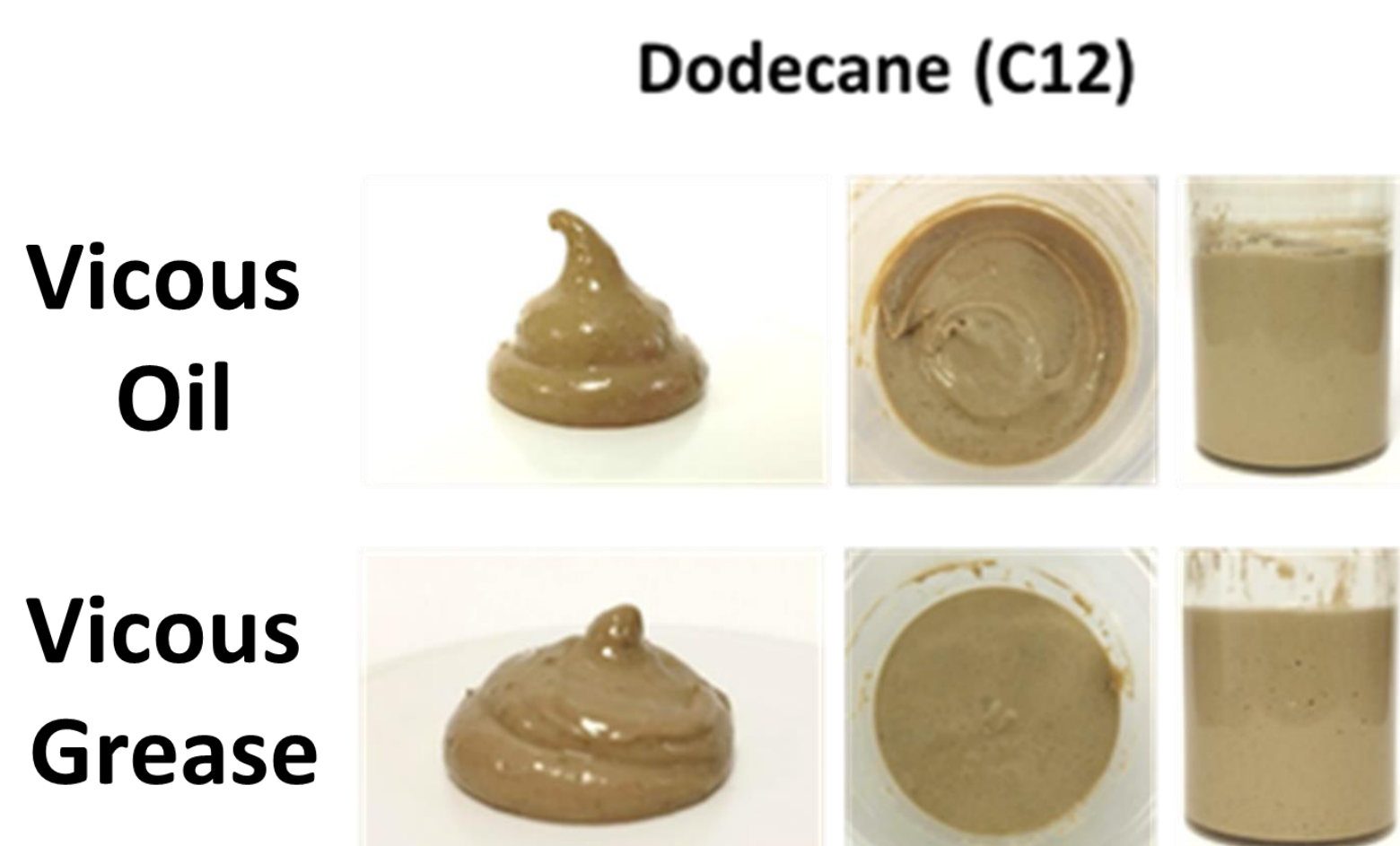
Assembly of oil and grease

Differents rheological behavior: Oil → Newtonian Grease → Non-Newtonian
Assembling rate choice: Low rate for Oil (20-30%vol.) & High rate for Grease (70-80%vol.)

Viscosity (C12) = 1,32 mPa.s & Viscosity (TBP^{30%}/C12^{70%}) = 1,90 mPa.s at 25°C



Geopolymer with assembled oil and grease



✓ Possibility of incorporating assembled oils and greases in geopolymer

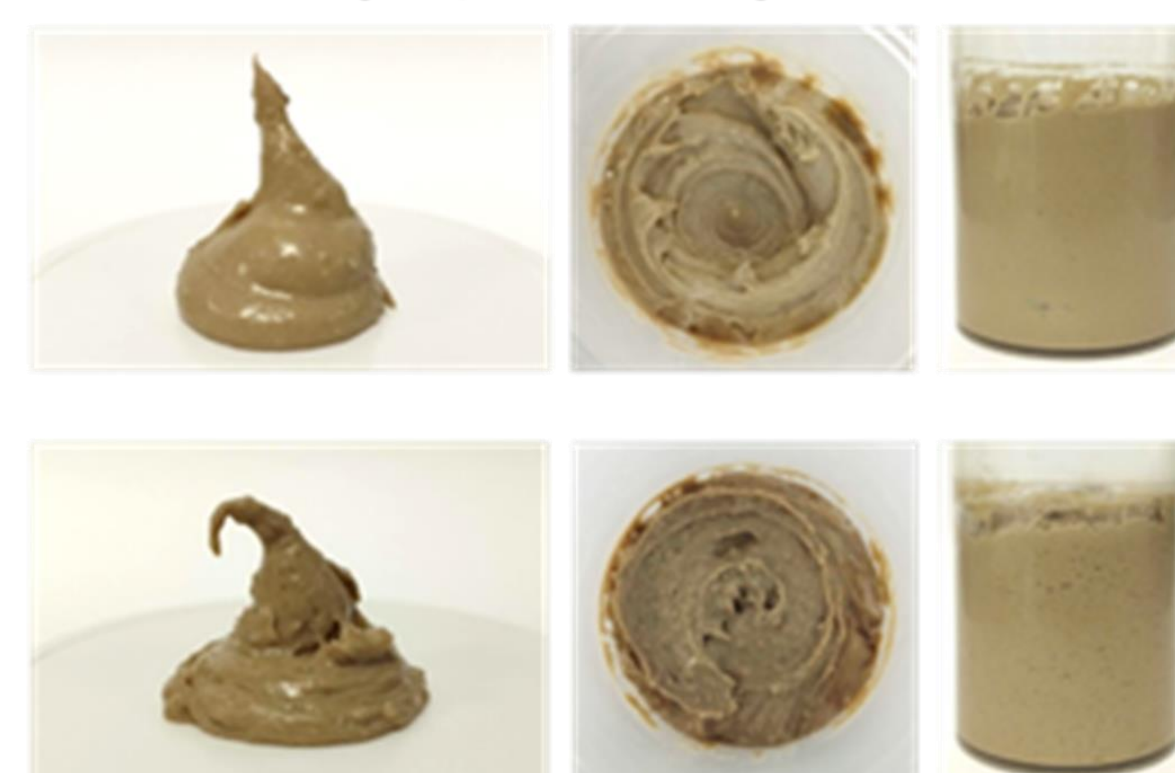
✓ No oil/grease coalescence on the samples surface

Increase of the viscosity in the case of:

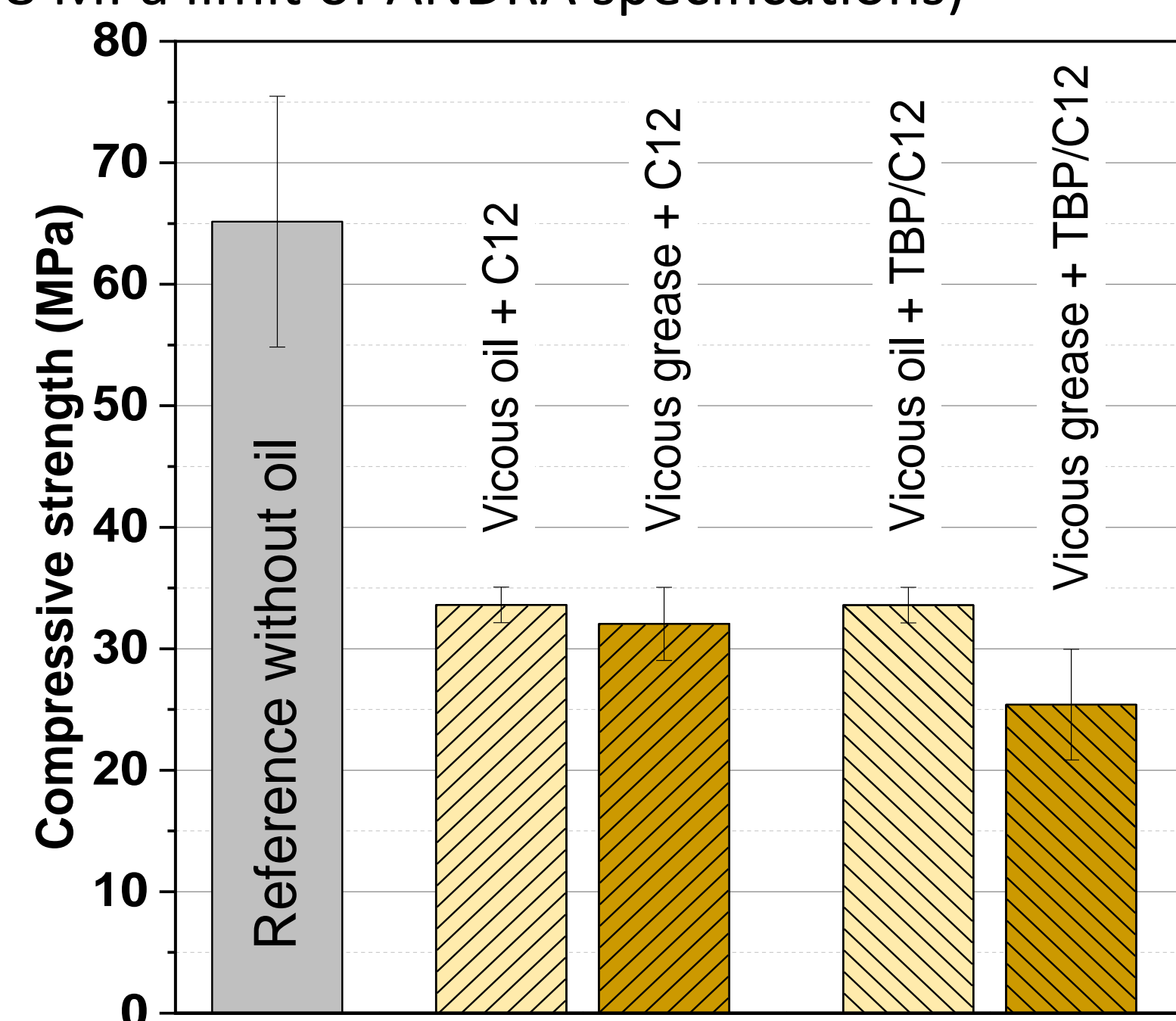
- Presence of additives in oil (comparison between industrial and model oil);
- Use of mix TBP/C12 as assembling solvent;
- No impact of TBP/C12 in case of industrial oil (i.e. with additives).

✓ **Micro-calorimetry analysis:** No impact of organics liquids on the poly-condensation of the geopolymer

Mix of TBP/C12 (30/70%Vol)

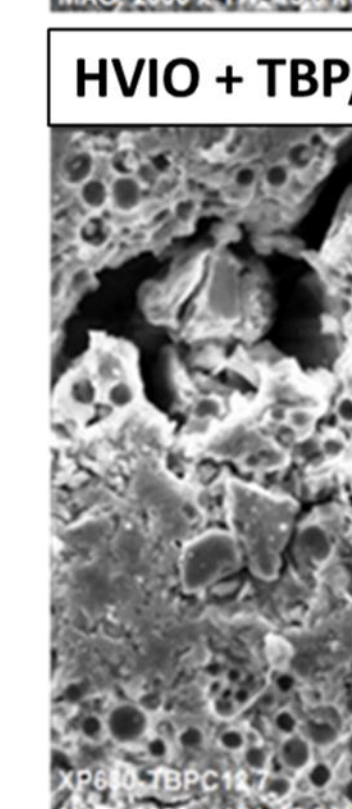
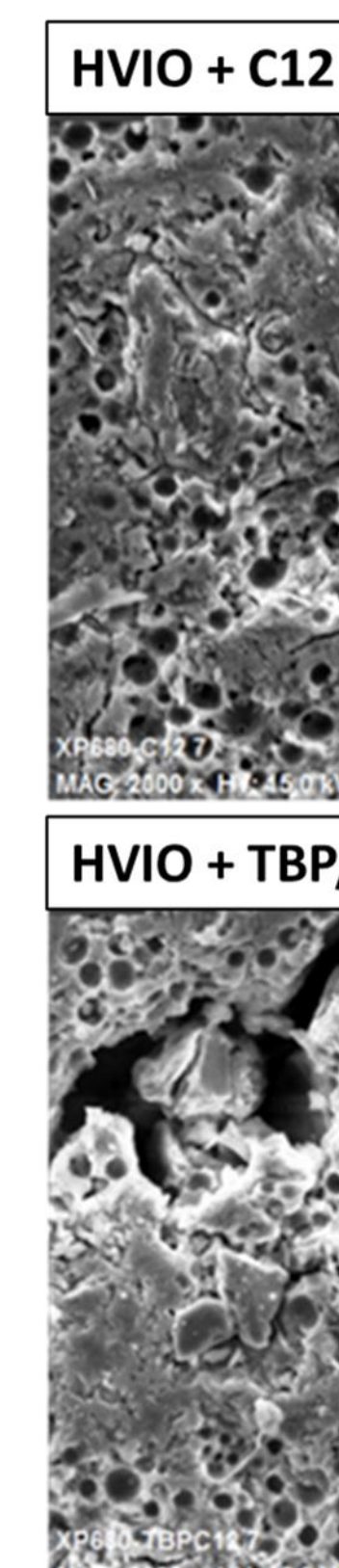


✓ **Mechanical performance** (compressive strength) about 30 MPa at 28 days (superior to the 8 MPa limit of ANDRA specifications)

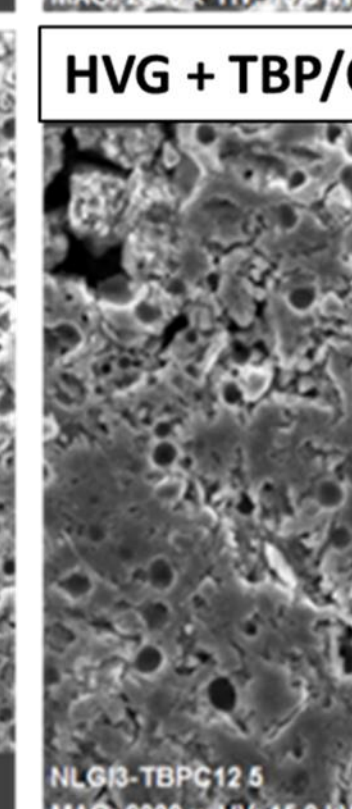
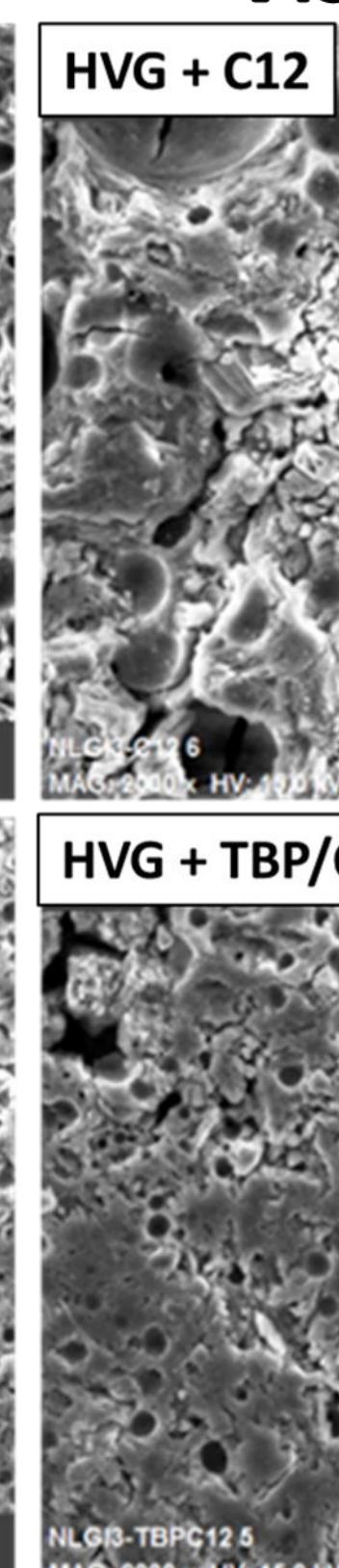


Microstructure analysis

Viscous Oil



Viscous Grease



- For assembled oil less, homogeneity of droplets with a size less than 10 μm whatever the assembling solvent used;
- For assembled grease, bigger droplets in case of C12 (10-30 μm) than TBP (10 μm with a few drops larger) as assembled solvent;
- Potential effect of TBP as a surfactant that could explain a better incorporation for the assembled grease.

Conclusion

- Choice of direct incorporation route by assembling process with low viscosity solvents
 - Possibility to decrease the viscosity of oils by assembly with low solvent content
 - Possibility to make greases fluid by assembling with a high quantity of solvent
- Possibility of using the TBP/Dodecane mixture (30/70) as an assembly solvent (double elimination in a single stabilization/solidification process)
- Simple method of application in radioactive environment
- Mechanical resistance of materials of the order of 30 MPa
- Regardless of the solvent used or the oil or grease incorporated → low droplets size

Prospects:

- Leaching studies of assembled oils/grease geopolymer composites (GEOIL)
- Durability tests of GEOIL with greases and viscous oils assembled with surfactant
- On-going work on the influence of different environments on the evolution of the porous network of geopolymers
- Study of the network of oil droplets according to the assembly of oils and greases (X-ray micro-tomography)

- References:**
- [1] V. Cantarel, F. Nouaille, A. Rooses, D. Lambertin, A. Poulesquen, and F. Frizon, "Solidification/stabilisation of liquid oil waste in metakaolin-based geopolymer," *J. Nucl. Mater.*, vol. 464, pp. 16–19, 2015, doi: 10.1016/j.jnucmat.2015.04.036.
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 - [3] C. A. Davy, G. Hauss, B. Planel, and D. Lambertin, "3D structure of oil droplets in hardened geopolymer emulsions," *J. Am. Ceram. Soc.*, vol. 102, no. 3, pp. 949–954, 2019, doi: 10.1111/jace.16142.
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 - [6] C. Reeb, C. Pierlot, C. Davy, and D. Lambertin, "Incorporation of organic liquids into geopolymer materials - A review of processing, properties and applications," *Ceram. Int.*, vol. 47, no. 6, pp. 7369–7385, 2021, doi: 10.1016/j.ceramint.2020.11.239.