Multifunctional Bio-Based Porous Materials for Sustainable Construction

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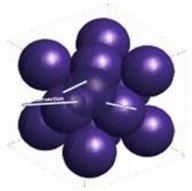
Introduction & Goals

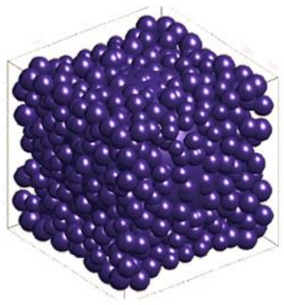
- Design of polymeric materials exhibiting **two porosity levels**, with **control** of pore size, shape and interconnectivity.
- **Double porogen templating approach** involving cubic NaCl particles or spherical polystyrene beads as imprints for larger pores, and porogenic solvents for smaller pores.
- Physical properties such as mechanical properties, thermal diffusion, and soaking/drying are **correlated with** double porosity framework.
- Application in **civil engineering**: insulation materials, soil mechanics.
- New non-invasive methods for fluid transport, *i.e.* **MRI**, 3D micro-tomography.

Double Porogen Templating Approach[1]

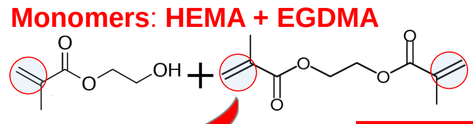
Large **macroporogen**
NaCl particles (400-250/250-200µm)
fused with SPS
or
Polystyrene beads (150µm)
linked with paraffin.



Sintering, Shaping



Monomers: **HEMA + EGDMA**

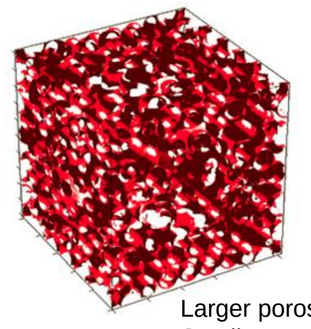


Free-radical copolymerization

Porogen Removal

+ **Porogenic solvent** (low-molar mass alcohol as EtOH) are inserted within the fused lattice

Doubly porous PHEMA-based materials with controlled morphology

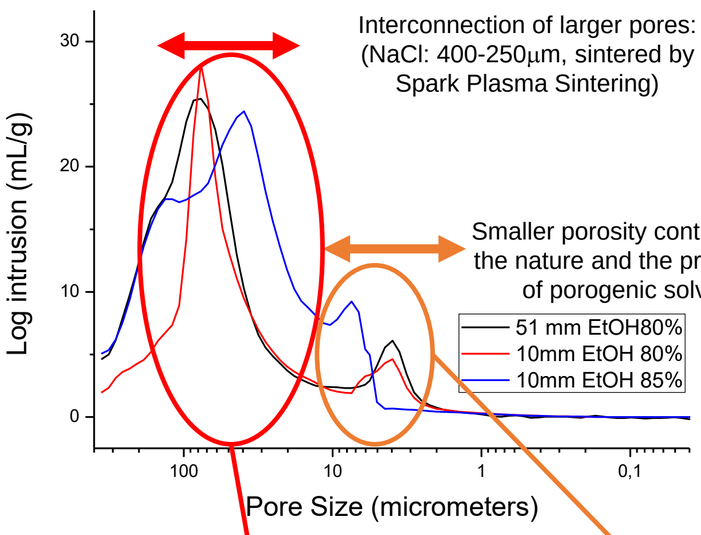


Larger porosity ~100µm
Smaller porosity ~1µm

Morphological Characterization

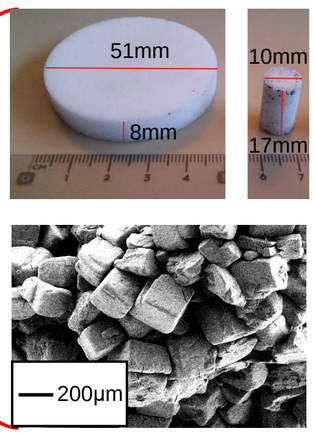
The pore morphology (size and interconnectivity) is analyzed by mercury intrusion porosimetry and SEM.

Mercury Intrusion Porosimetry

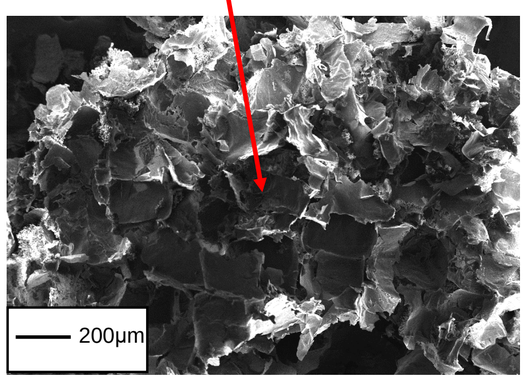


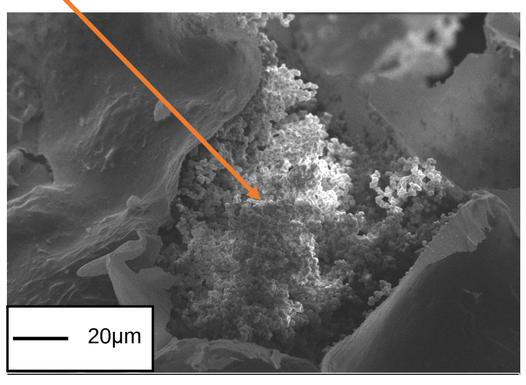
Interconnection of larger pores:
(NaCl: 400-250µm, sintered by Spark Plasma Sintering)

Smaller porosity controlled by the nature and the proportion of porogenic solvent



SEM





Two different levels of porosity visible by SEM:

- Around 200-400µm, The dissolution of NaCl particles exhibits big cubic macropores, controlled by the particle size and shape.
- Around 2-4µm, the polymerization with 80%$_v$ porogenic solvent (EtOH) creates a small micrometric porosity after extraction.

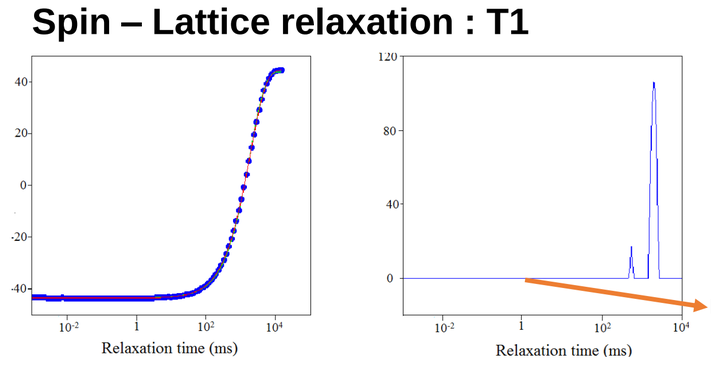
97% total porosity: [85% larger pores – 12% smaller pores]

MRI: Fluid in the Material

The relaxation mechanisms, illustrated by T1 and T2, are independent phenomena[2], and both give information on water environment in pores.

Biporous sample saturated with water

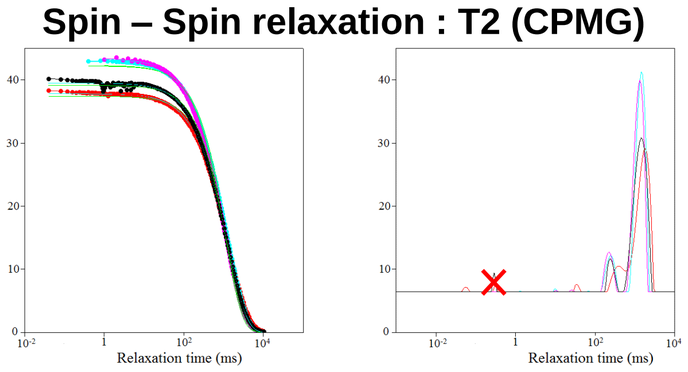
Spin – Lattice relaxation : T1



T1 : 2 relaxation times (600ms & 2000ms).
Could have 2 populations of protons for 2 pore sizes ?

No signal below 10ms, no bound water ?

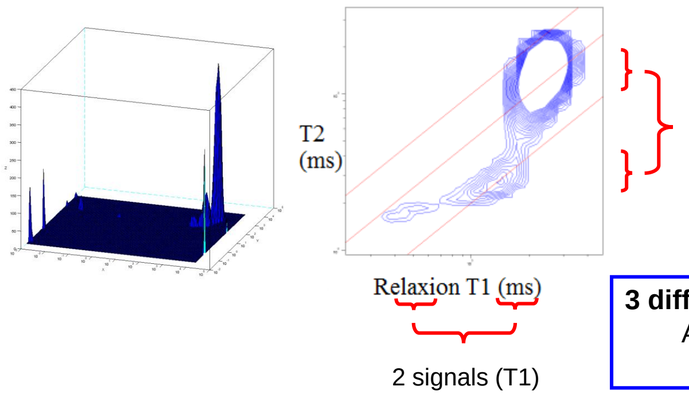
Spin – Spin relaxation : T2 (CPMG)



CPMG, T2 : 2 relaxation times too (300ms & 1400ms).
But not the same **area ratio** than for T1 relaxation !

Need a new analysis method !

T1 – T2 (2D) simultaneous scan



With the simultaneous scan, 3 different signals are highlighted. They are overlapping each other in 1D (T1 or T2 only) analysis.

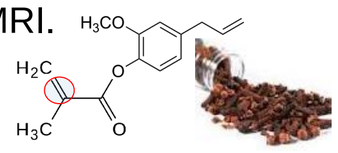
2 signals (T2)

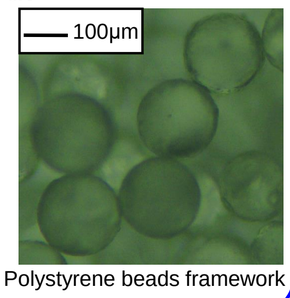
2 signals (T1)

3 different environments for water ?
Are they analysis artefacts ?
More studies are needed !

Conclusions

- Design of doubly porous materials with controlled pore size, connectivity and low density.
- Both porosity levels are independent and can be tuned separately
  ➔ Impact of various porosity levels on physical properties can be assessed.
- Shapeable materials: polystyrene beads replaces NaCl for spherical pores increasing the smaller porosity density
- Imbibition and Drying mechanisms analyzed by MRI.
- Bio-based materials with eugenol methacrylate[3].





Polystyrene beads framework

References

[1] H.B.Ly, B.Le Droumaguet, V.Monchiet , D.Grande *Polymer 78* **(2015)**, 13-21.

[2] P.Faure, U.Peter, D.Lesueur, P.Coussot, *Cem. Concr. Res.* **(2012)**.

[3] L.Rojo, B.Vazquez, J.parra, A.L.Bravo, S.Deb, J.S.Roman, *Biomacromolecules 7* **(2006),** 2751-2761.