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Development of a new calcium phosphate/PCL graded scaffolds in view of biomedical applications

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HAL Authorization

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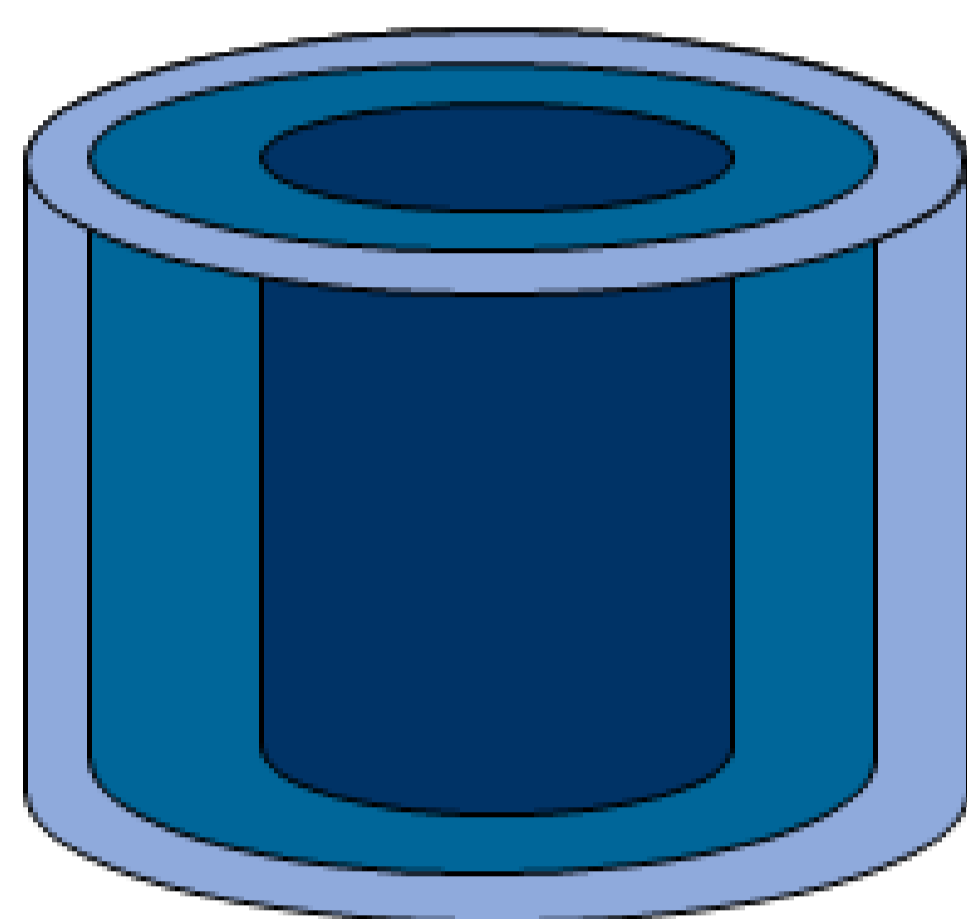
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Design of the scaffold

Context: CaP (e.g. hydroxyapatite HA and β -TCP) and biodegradable polymers/CaP composites: main materials studied for bone tissue scaffolds
 Highly porous scaffolds needed to allow bone cell colonization and vascularization of the implants
 Main challenge for this application: multi-functionality of the implants

Main idea: Designing a new type of graded structure with 2 key points

- A structure with a **gradation of composition** (HA/ β -TCP wt%) and of **volume fraction of pores** from the inner to the outer layer
- A combination of a **ceramic/polymer (polycaprolactone, PCL) outer layer to a ceramic inner core**



Inner dense HA core

Core dedicated to the mechanical strength

Intermediate porous HA/ β -TCP layer

Porous layer (targeted 60 vol%) acting as a "template" for adhesion of the external layer
 HA/ β -TCP = 80/20 wt%

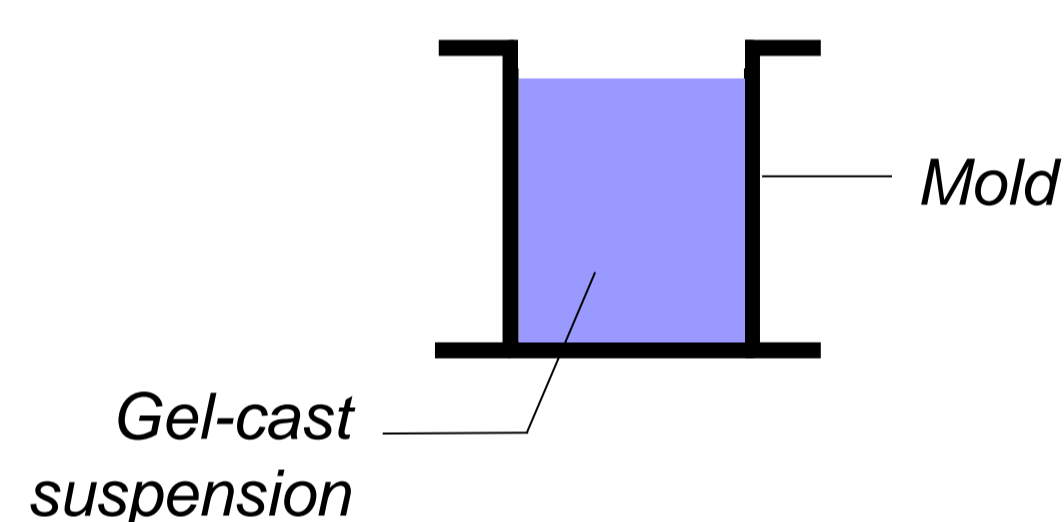
Outer porous PCL(HA/ β -TCP) layer

Porous layer (targeted 80 vol%) in contact with bone
 PCL/ceramic = 60/40 vol% and HA/ β -TCP = 30/70 wt%

Fabrication of the scaffold

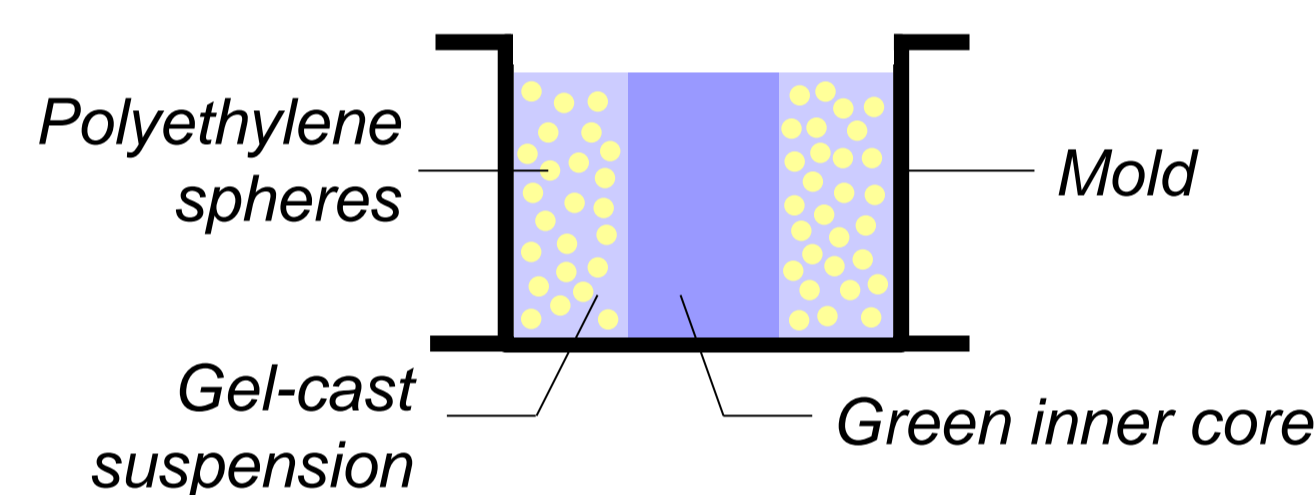
STEP 1 Inner core Gel-casting

- Casting of a suspension with HA powder and agar (gelling agent)



STEP 2 Transition layer Gel-casting / Sacrificial template

- Porogens: polyethylene spheres ($\varnothing = 224-355 \mu\text{m}$)
- Casting of the suspension around the wet and gelled inner core for a better adhesion

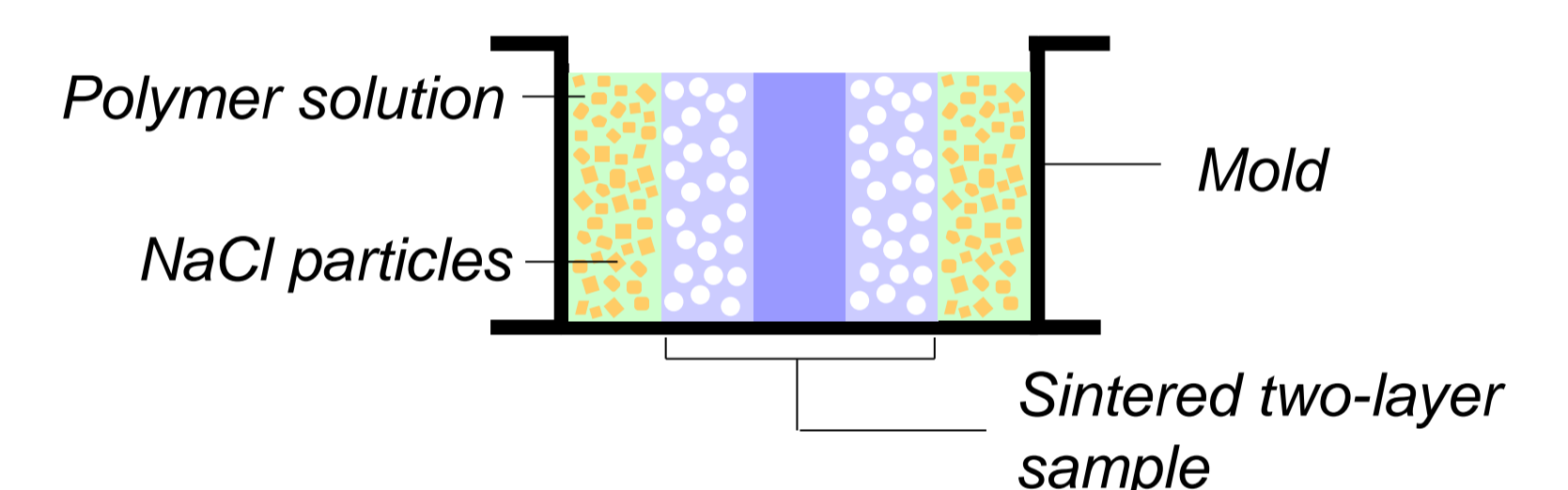


- Debinding and sintering ($1300^\circ\text{C} / 3\text{h}$)

STEP 3 Outer layer Solvent casting / Sacrificial template

Solvent casting / Sacrificial template

- Porogens: NaCl particles ($= 200-400 \mu\text{m}$)
- Casting of the solution (PCL + CaP powder + NaCl) around the sintered structure

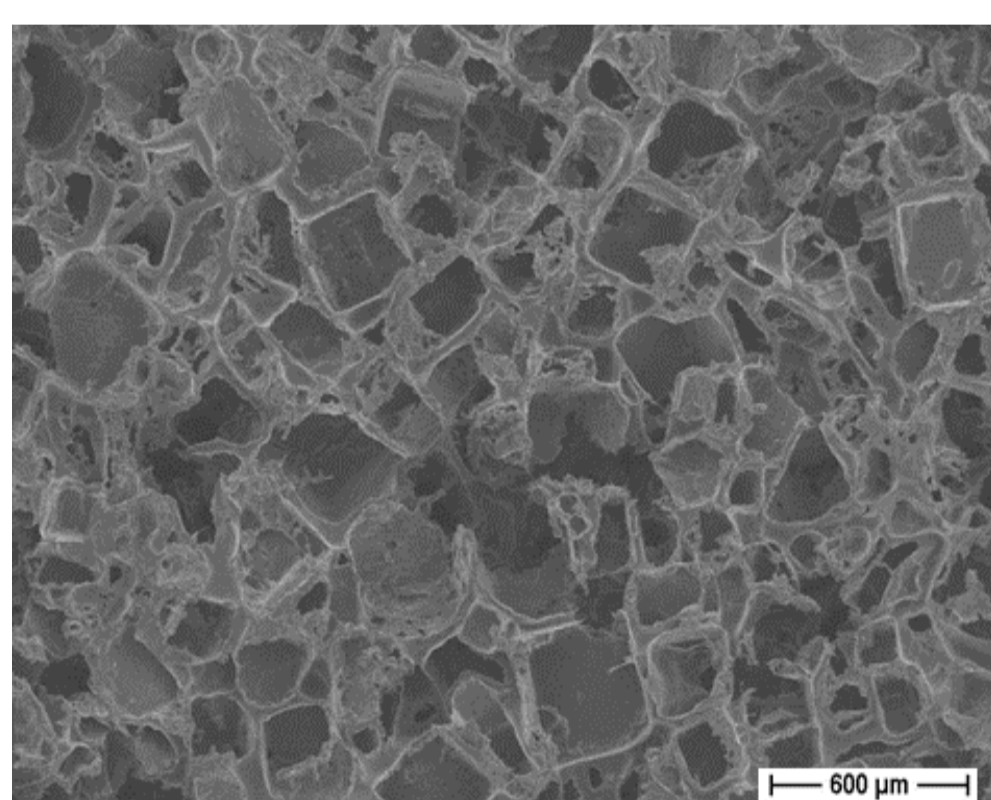


- Evaporation of the solvent

Characterization of the different layers

A three-layer structure was obtained with:

- well-bonded interfaces
- a clear gradation of porosity
- a gradation of phase composition (less marked)



Outer PCL(HA/ β -TCP) layer
 Relative density = $(23.8 \pm 1.8) \%$
 HA/ β -TCP = 32/68 wt%

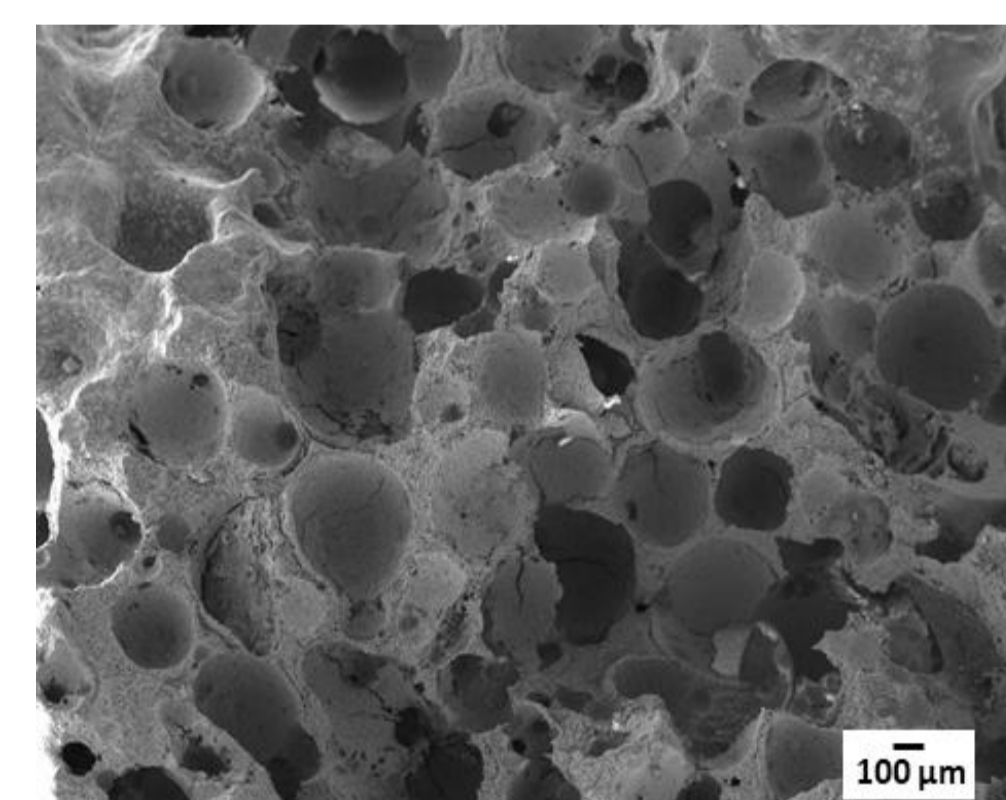
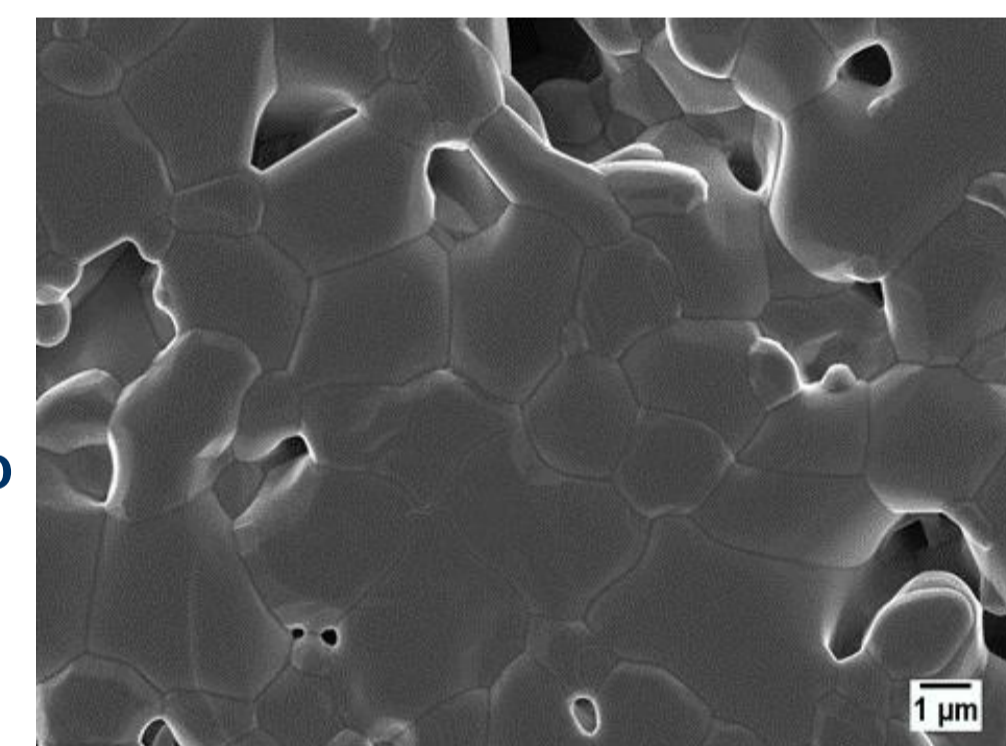


Dense HA core

Relative density = $(80.1 \pm 1.0) \%$
 HA/ β -TCP = 58/42 wt%

Intermediate HA/ β -TCP layer

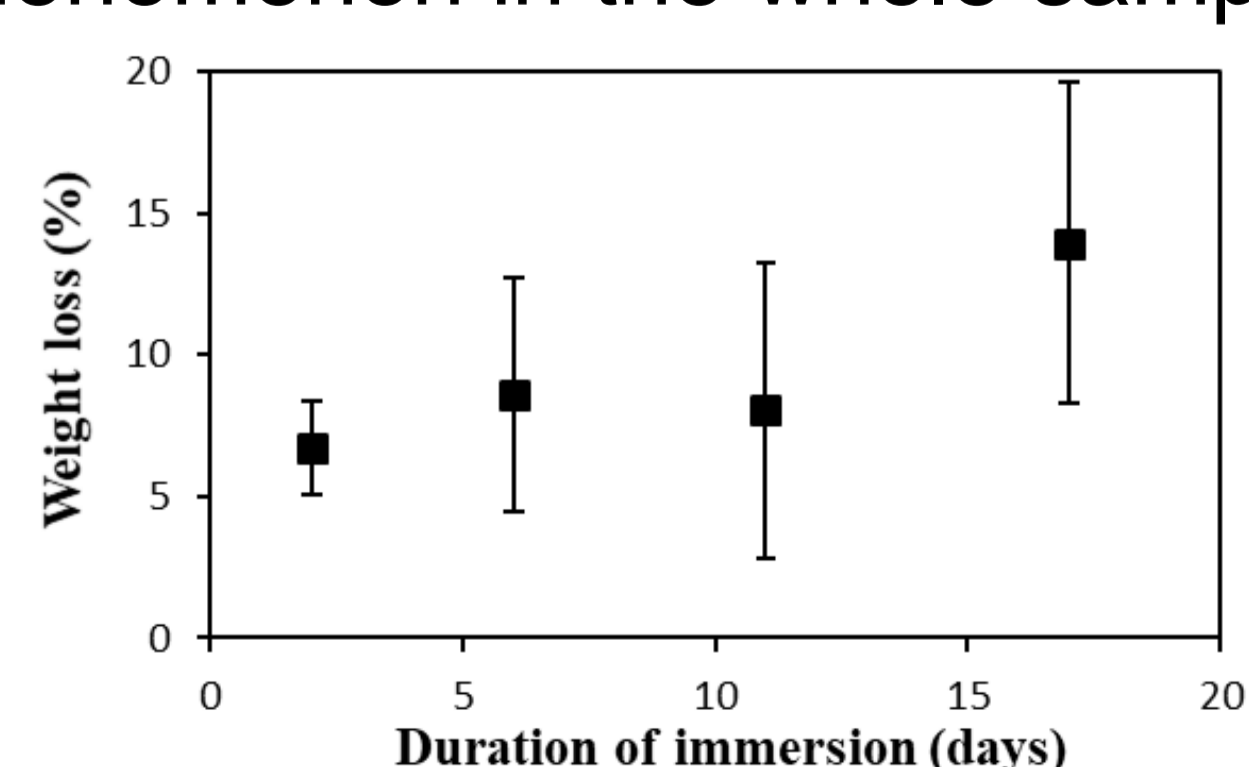
Relative density = $(34.6 \pm 1.6) \%$
 HA/ β -TCP = 47/53 wt%



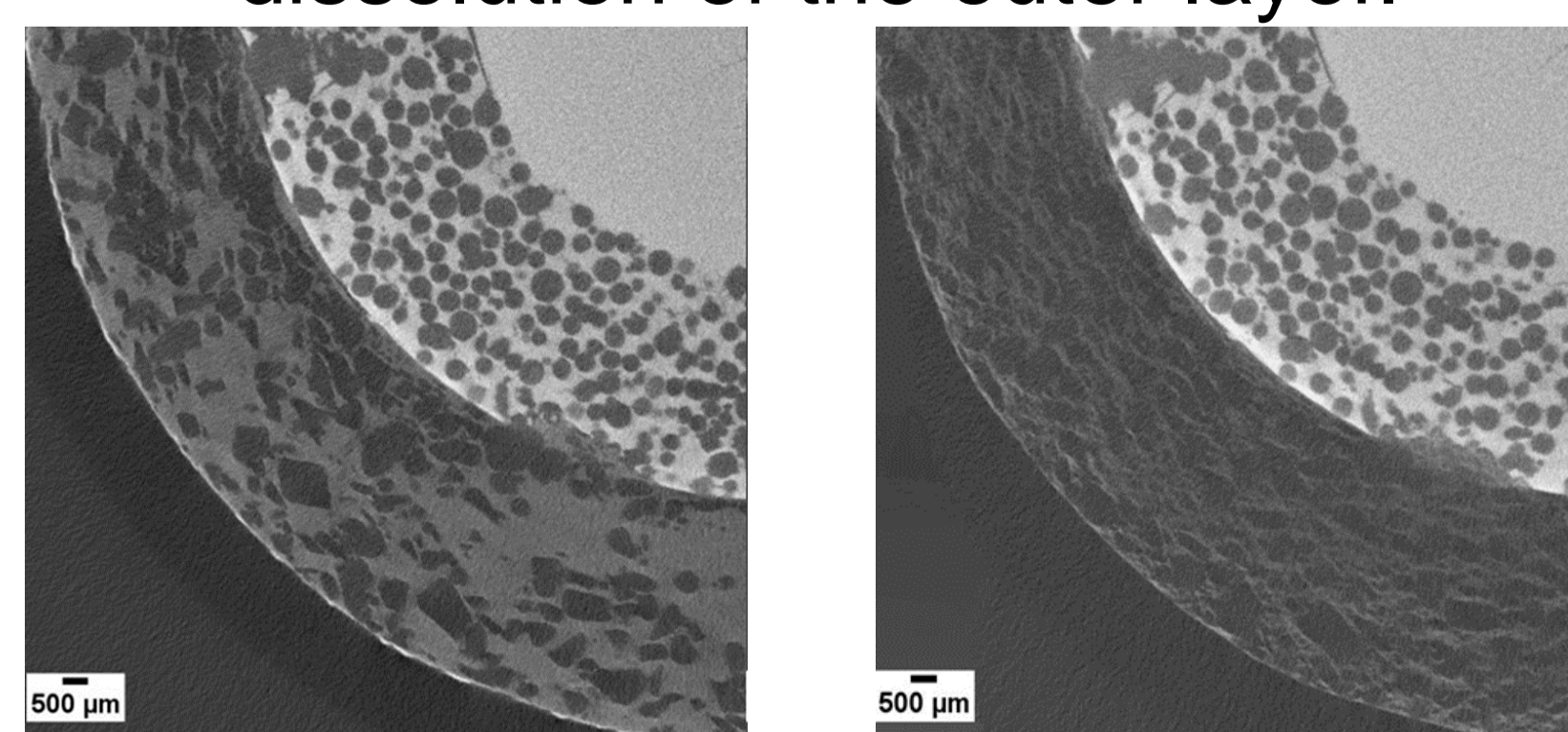
In vitro dissolution test

Experiments: immersion of the specimens into TRIS solution during 2, 6, 11 and 17 days, with a daily renewal of solution

Weight loss indicates a dissolution phenomenon in the whole sample.

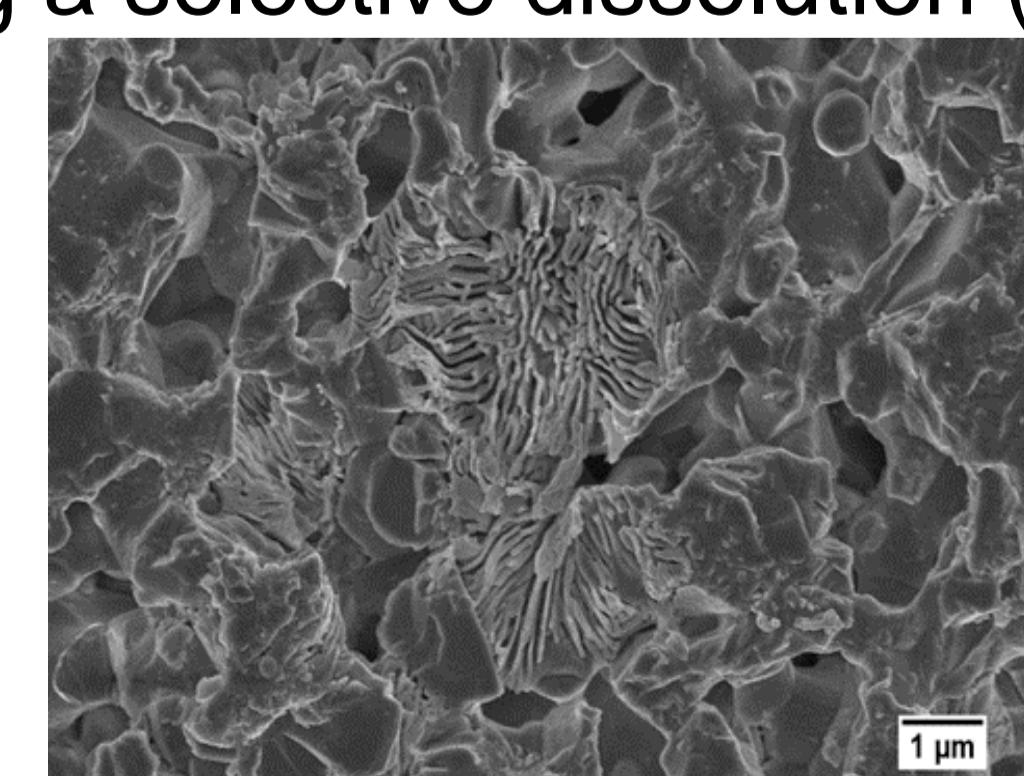


X-ray tomography shows a preferential dissolution of the outer layer.



Slices from X-ray tomography for a sample before (left) and after 17 days of immersion (right)

In the inner core, degradation "pits" are visible, evidencing a selective dissolution (β -TCP ?)



SEM micrograph of the inner core after 17 days of immersion (right)

Summary

- A three-layer structure with a gradation of porosity and composition was obtained via a combination of different processes.
- An in-vitro dissolution test evidences different signs of dissolution and possible different behaviors in the different layers, in agreement with the targeted multi-functionalities of the scaffold (i.e., a resorbable external layer combined with a core devoted to the mechanical function).