

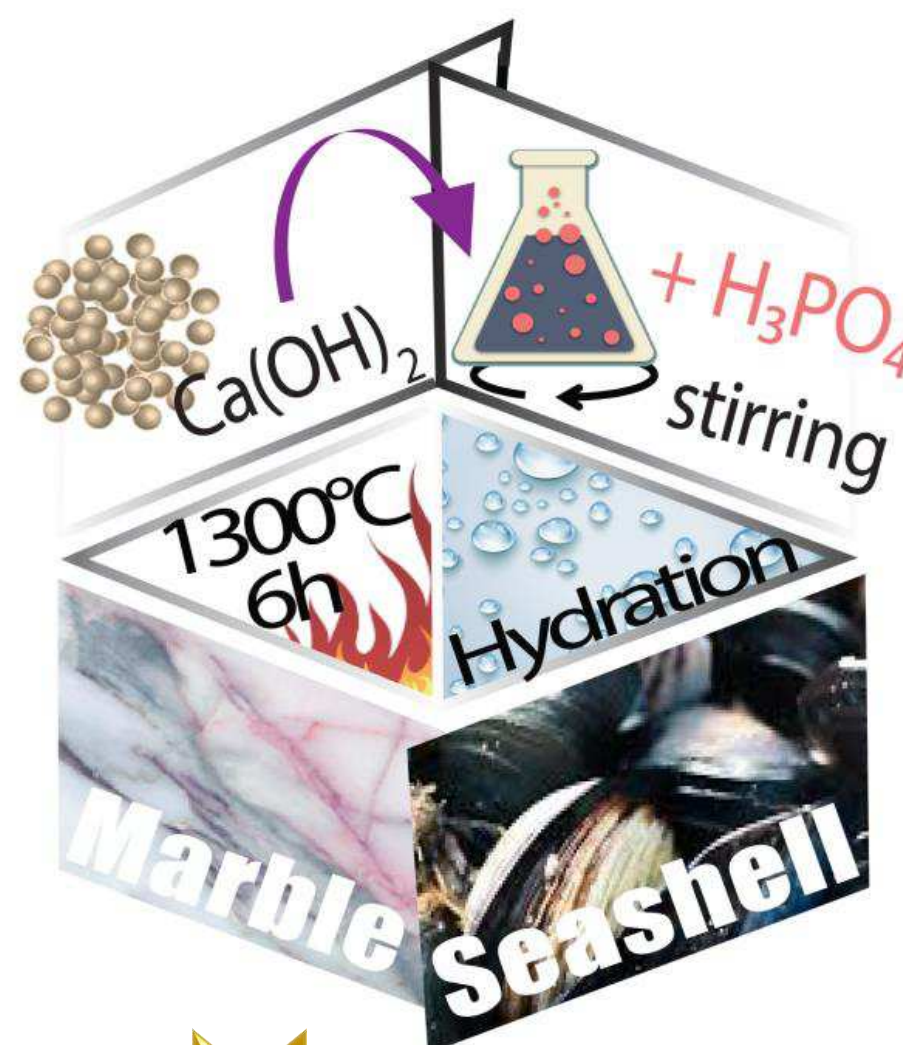
MANUFACTURING PROCESS OF A PRODUCT DESTINED FOR BONE DEFECTS RECONSTRUCTION, BASED ON HYDROXIAPATITE AND BIOGENIC BIPHASIC CALCIUM PHOSPHATE

F. Miculescu¹ - A.C. Mocanu¹ - G. Stan² - I.V. Antoniac¹ - M.C. Costoiu¹
S.I. Voicu¹ - M. Miculescu¹ - I.M. Mates¹ - A. Semenescu¹

The invention relates to the manufacturing process of a product destined for bone defects reconstruction, based on hydroxyapatite and biogenic biphasic calcium phosphate, with a controlled ratio between hydroxyapatite/tricalcium phosphate. All calcium phosphates result from the thermal dissociation of calcium carbonate in form of dolomitic marble and seashells, and treatment of calcium hydroxide solution with phosphoric acid (range: 100–130% x calculated stoichiometric amount).

1 THERMAL DISSOCIATION Biogenic calcium oxide synthesis

- Mechanical brush
- Sectioning of marble slabs
- *Thermal treatment in electric oven*
 - Temperature: **1300°C**
 - Duration: **6 hours**
 - Ambient: **air**
 - Cooling: **oven cooling**



MODULATED WET SYNTHESIS Synthesis of ceramic powders

2

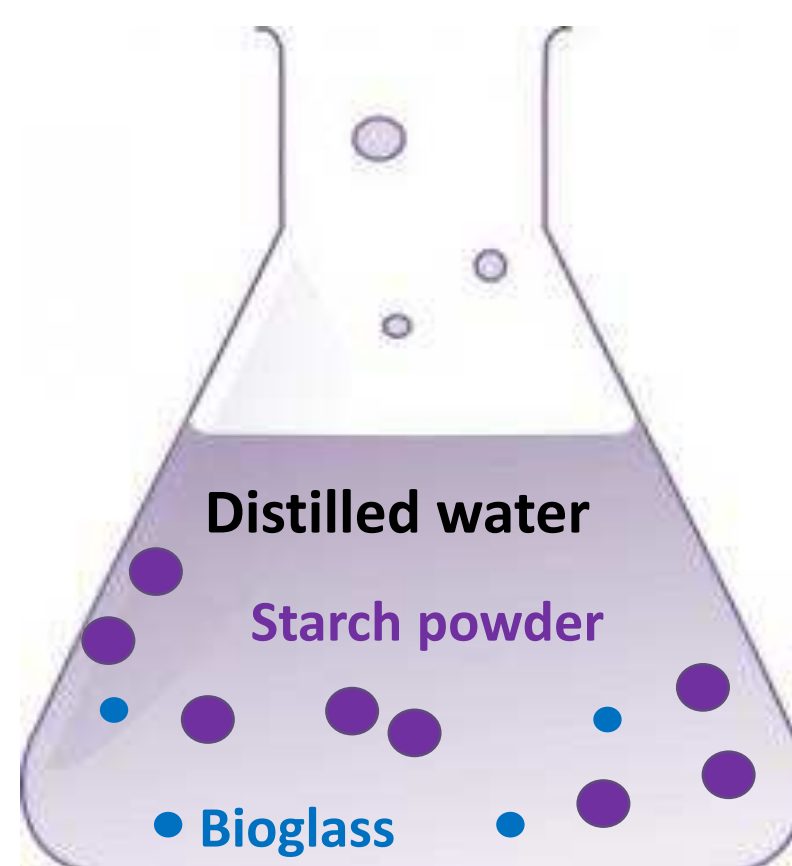
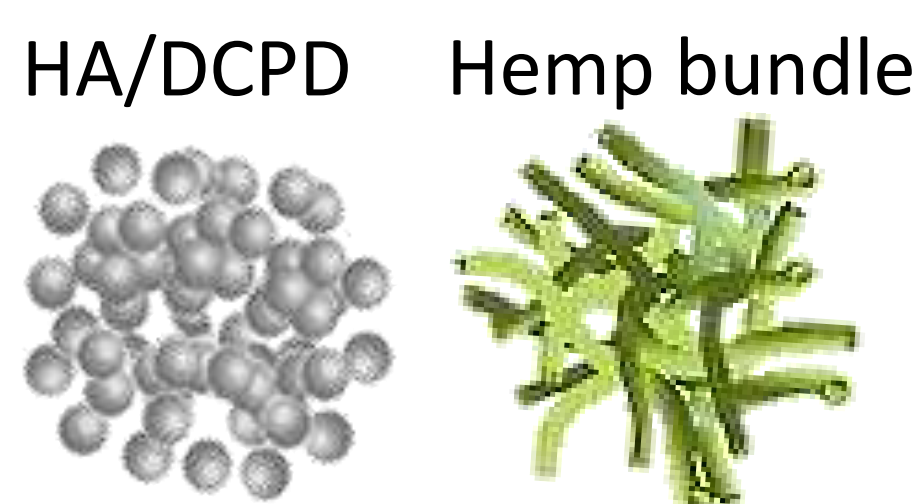
- Hydration of calcium oxide: **distilled water**
- *Synthesis solution formation*
 - Distilled water: **200 ml**
 - Calcium hydroxide: **10 g**
 - Stoichiometric H₃PO₄ (85%): **5.5 ml**
 - Acid addition rate: **1 ml/min**
- *Magnetic stirring*
 - Temperature: **25°C**
 - Time: **2 hours**
 - Rotations per minute: **700 rpm**
- *Drying thermal treatment*
 - Preliminary: **24 hours/ RT**
 - Temperature: **1300°C**
 - Duration: **6 hours**
 - Ambient: **air**

The porosity of the product, evidenced as interconnected channels and pores, results after the complete combustion of the incorporated organic materials. The modulation of the mechanical and biological features is performed function of the sintering parameters (modification of the temperature and holding time).

Thermal treatment in electric oven with continuous ventilation

- Temperature: **1200°C**
- Duration: **10 hours**
- Ambient: **air**
- Cooling: **oven cooling**

4 Internal architecture forming SINTERING



The obtained powders are mixed with hemp fibres braided/twisted as a bundle and then soaked in aqueous-solution of starch and bioglass. The mixture is compacted by cold isostatic pressing.

Aqueous starch and bioglass solution

- Bioglass: **0.1-2%** of the starch mass
- Vol. parts starch+bioglass/distilled water: **1/5**
- Environment: **thermostatic bath**
- Temperature: **75°C**

Hemp fibres soaked in aqueous starch+bioglass solution

- Sectioning dimensions: **12 mm**
- Bundle diameter by twisting: **1 mm**
- Modelling: **various shapes**
- Drying: **room temperature (RT)**

Mechanical processing

- Mixture formation – mass ratio of ceramic powder/soaked hemp fibres: **30/2**
 - Pressing: **10-25 MPa**
 - Shape: **cylindrical**
 - Cylinder dimensions:
 - h = **30 mm**; Ø = **10 – 50 mm**

Preparation technology

FABRICATION OF 3D PRODUCTS

3



Bone reconstruction SCAFFOLD

- Theoretical porosity: **29%**
- Dimension of hollow pores and channels: **500 – 1000 µm**
- Dimensional shrinkage (after sintering): **34%**
- Composition: **90% hydroxyapatite – 10% β – tricalcium phosphate**