Óbuda University

Doctoral School of Materials Sciences and Technologies

ELKH, Centre for Energy Research, Institute of Technical Physics and Materials Science

Development and structural characterization of bioceramics

PhD student :

Maroua Houria Kaou

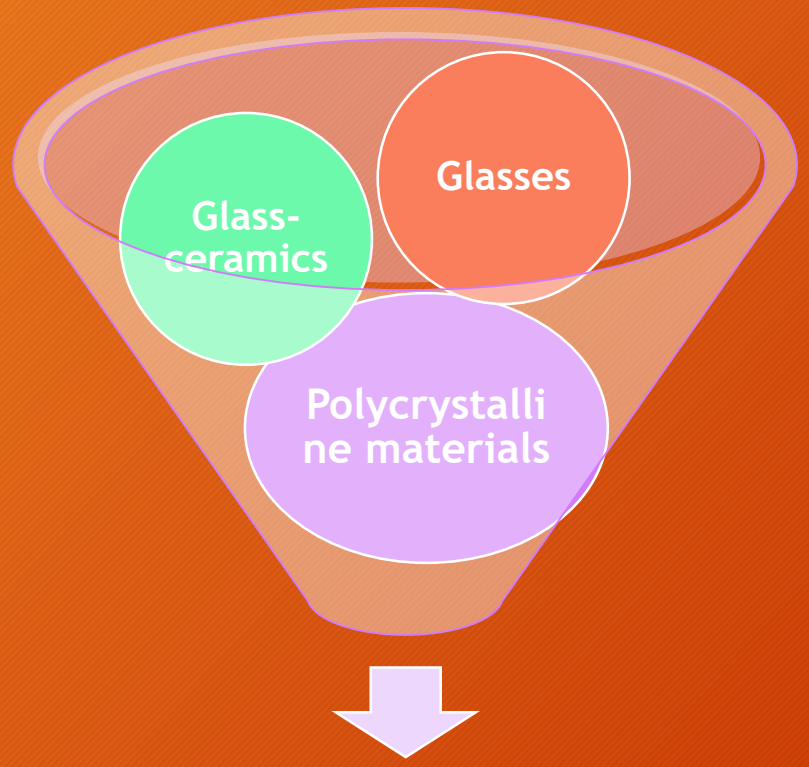
Supervisors:

Dr. Csaba Balázsi

Dr. Katalin Balázsi

2020/2021

Recall on the 1st semester



Bioceramics

Bioceramics

- Alumina
- Zirconia
- Magnesia
- Carbon
- Silica-contained compounds
- Calcium-contained compounds

  } **Calcium Silicates ($CaO\text{-}SiO_2$)**

- Limited number of other chemicals

Calcium Silicates ($CaO\text{-}SiO_2$)

Bioactive materials based on Ca-Si can be divided into four groups

- Bioactive glass
- Bioactive ceramics
- Glass-ceramics
- Cements.

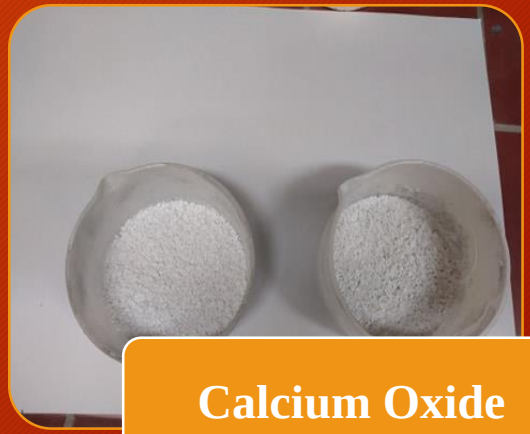
- ✓ The beneficial effects of materials based on Ca-Si on inducing bone formation were first found on glass in the system of SiO_2 -CaO-Na_2O-P_2O_5 by Hench et al. (Hench, L. L., Splinter, R. J., Allen, W. C. and Greenlee, T. K. (1971)).
- ✓ Glasses with this composition were able to bond to soft and hard tissues forming a (CHA) layer when exposed to biological fluid (SBF), (it was named bioglass).
- ✓ Since then, different types of bioactive glasses have been developed and some of them have been already used in the clinic like 45S5 Bioglass.

Starting materials preparation Calcium Oxide (CaO) extraction

The main component in eggshells is Calcium carbonate ($CaCO_3$)

Raw Eggshells

Heating Treatment in the air (7h/10h/12h, 900 °C)



Calcium Oxide (CaO)

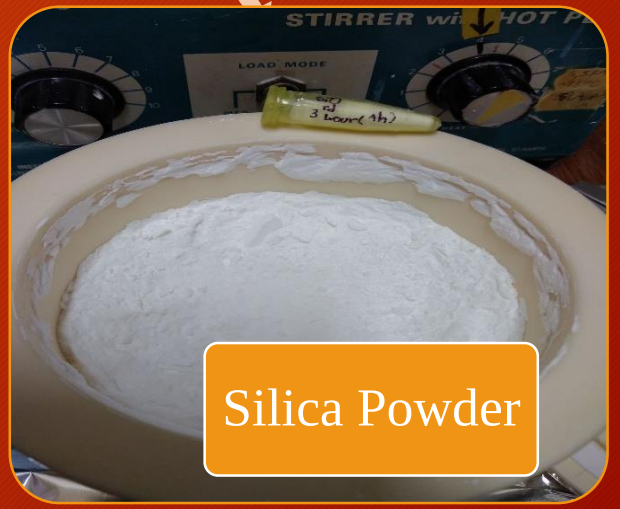
Thermal decomposition of Calcium Carbonate upon the heating treatment at 900 °C in air

$$CaCO_3 \rightarrow CaO + CO_2$$

Starting materials preparation Silica powder (SiO_2)

Silica Gel

Grinding



Silica Powder

Powder mixture preparation

Powder mixture preparation has been carried using two different milling techniques: 40g CaO+ 60g SiO2

Ball miling (3h)

Attrition milling (5h)

Ball milling for 3h
Milling with wet conditions
30,13g of ZrO2 balls, 10 balls (11,43 mm in diameter)

Attritor milling (Union process) at 5h for 2800 rpm
Milling with wet conditions
ZrO2 balls (1mm in diameter)
PEG (11,1g)

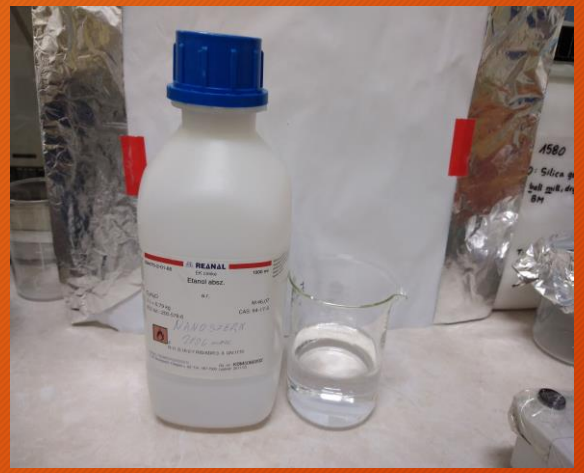
Powder mixture preparation (40% CaO (10h) + 60% SiO2) with PEG

The powder mixture of 100g (10%CaO (12h)+90%SiO2)

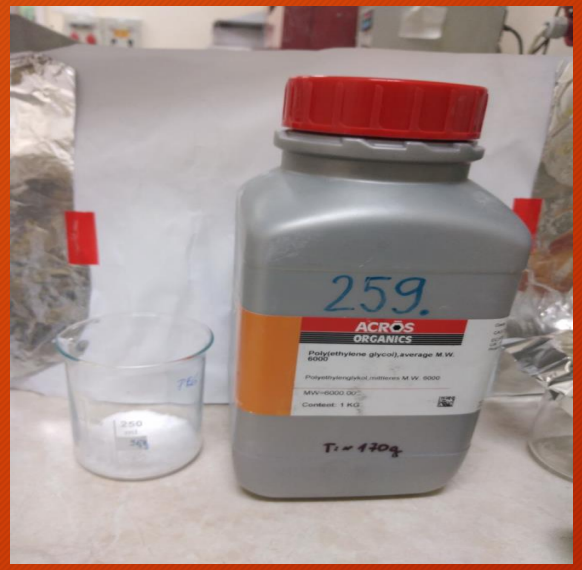
1104.7 g of Zirconia balls (“ZrO2” each one has 1mm in diameter)

Powder mixtures (10%CaO+90%SiO2+Ethanol)+ ZrO2 balls

Attritor milling (Union process type) at 3h for 2000 rpm in wet conditions
Changing the rotation speed to 500 rpm in the last half hour of milling



100 g of Ethanol



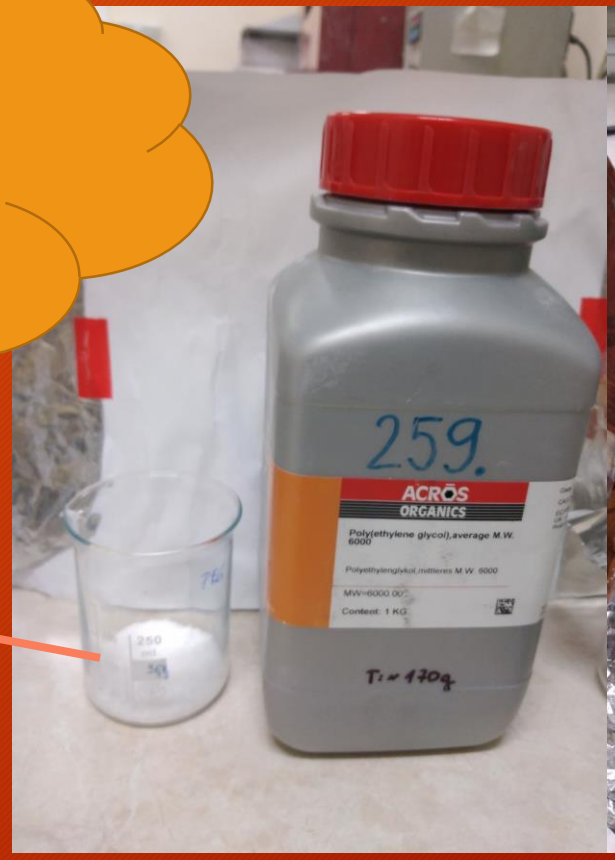
10g of PEG (Poly Ethylene Glycol)

Drying the mixture at 151 °C and sieving 100 micron mesh

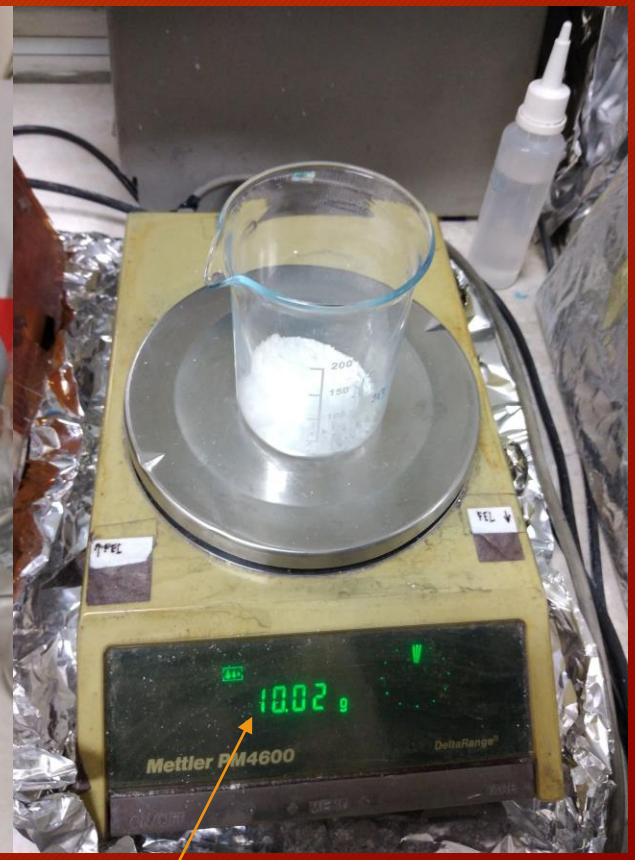
The powder mixtures were milled with 100 g ethanol and 1104.7 g of Zirconia balls (“ZrO2” each one has 1mm in diameter). The milling process has been carried out in an attritor milling (Union process type) at 2000 rpm for 3h with adding the PEG (Poly Ethylene Glycol) in the last half hour of milling and changing the rotation speed to 500 rpm; (milling in wet conditions)

Note: during the process the mixture powders must be kept in a wet medium by adding ethanol each time the mixture starts to get dry

Adding the PEG (Poly Ethylene Glycol) in the last half hour of milling and changing the rotation speed to 500 rpm



PEG (Poly Ethylene Glycol)



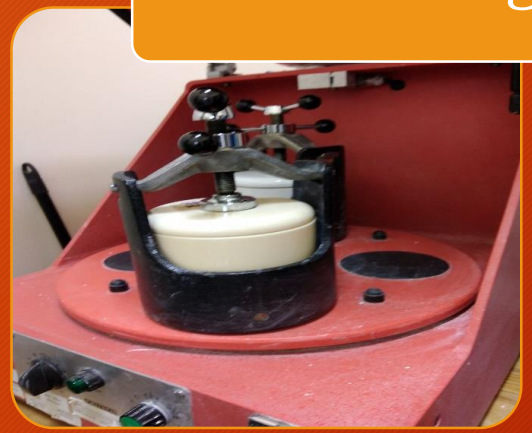
10g of PEG

Starting materials preparation

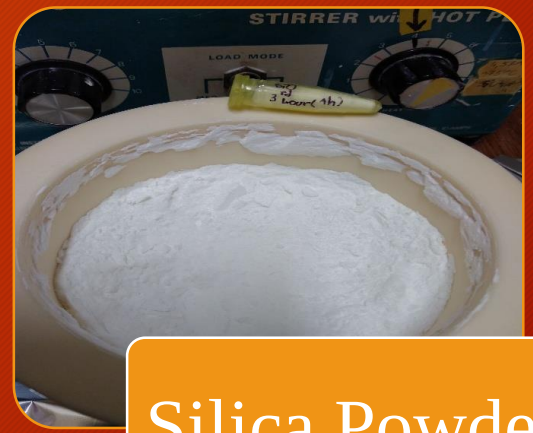
As starting materials, Silica powder (SiO_2) has been prepared by ball milling starting from silica gel by intensive ball milling for 3 hours in dry conditions using 10 balls (ZrO2 each one has 11,43 mm in diameter)

Silica Gel

Ball Milling

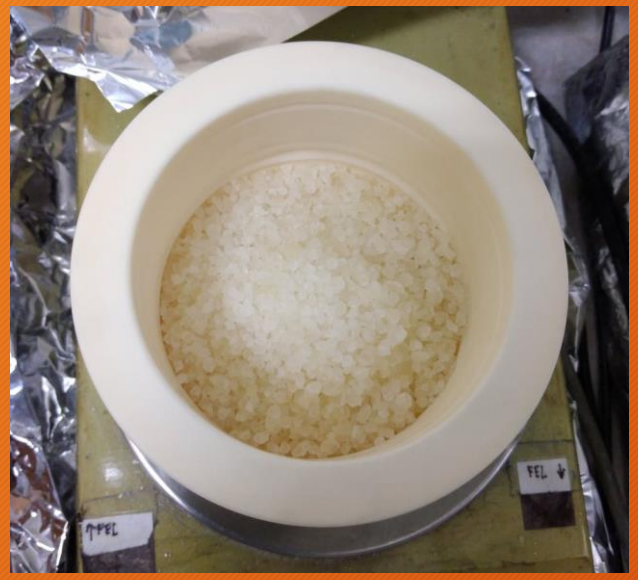


30,13g of ZrO2 (10 balls)



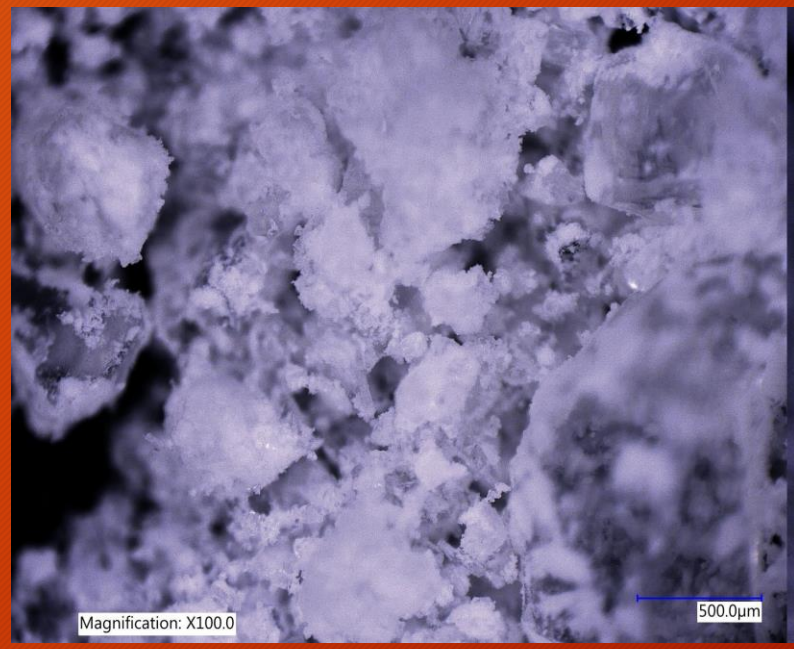
Silica Powder

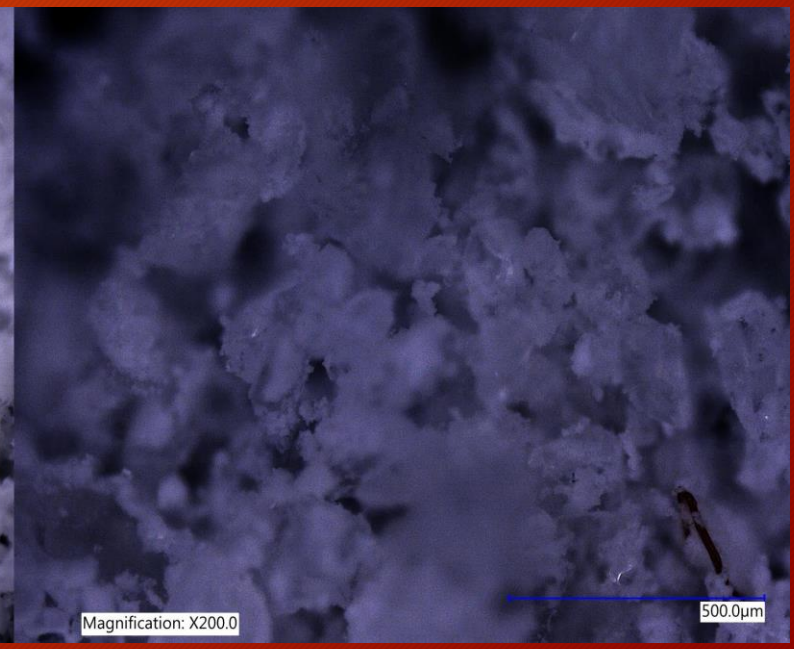
Starting materials preparation

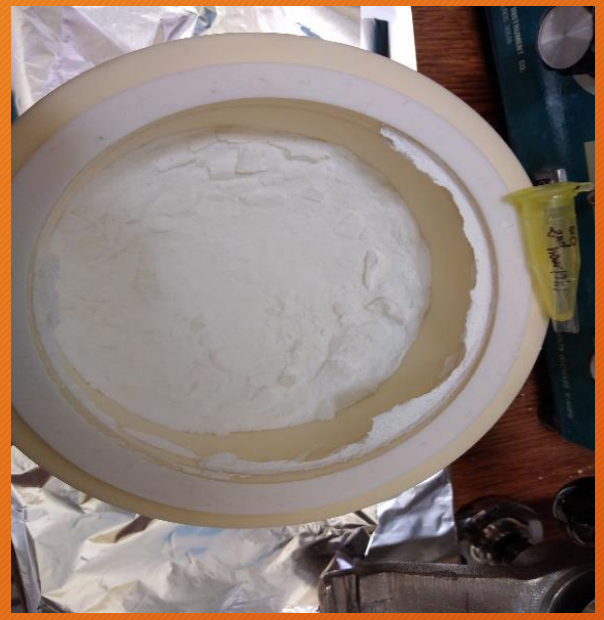


Silica gel (starting material)

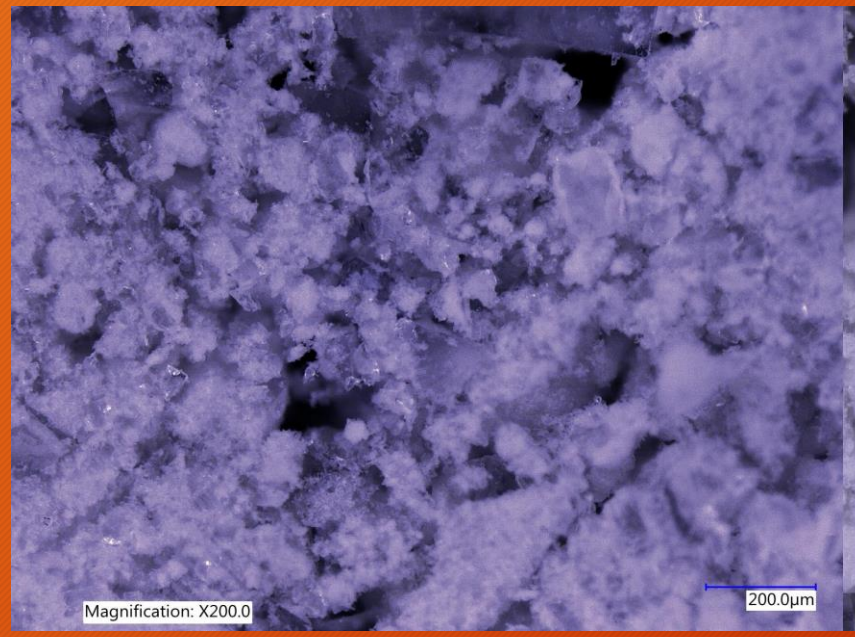
Milled Silica gel after 1 hour

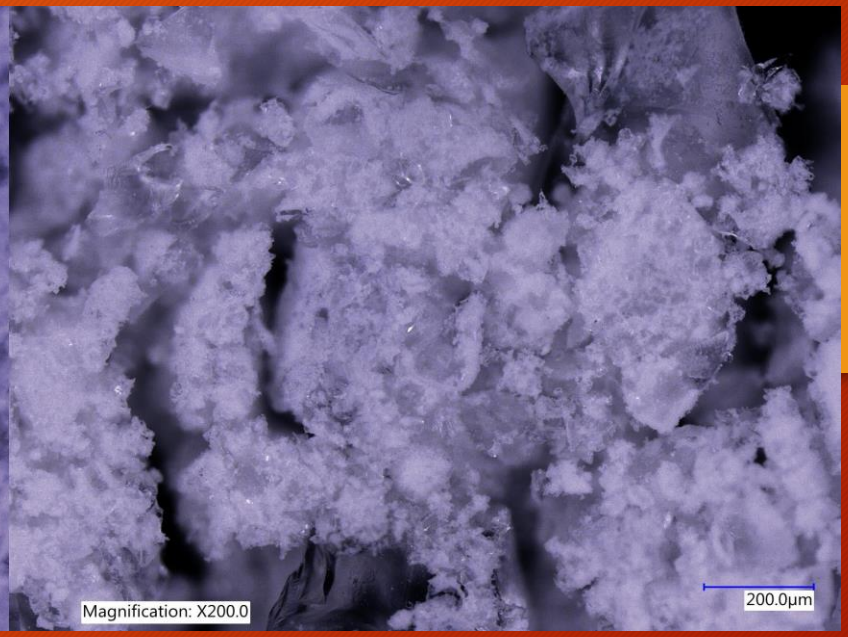


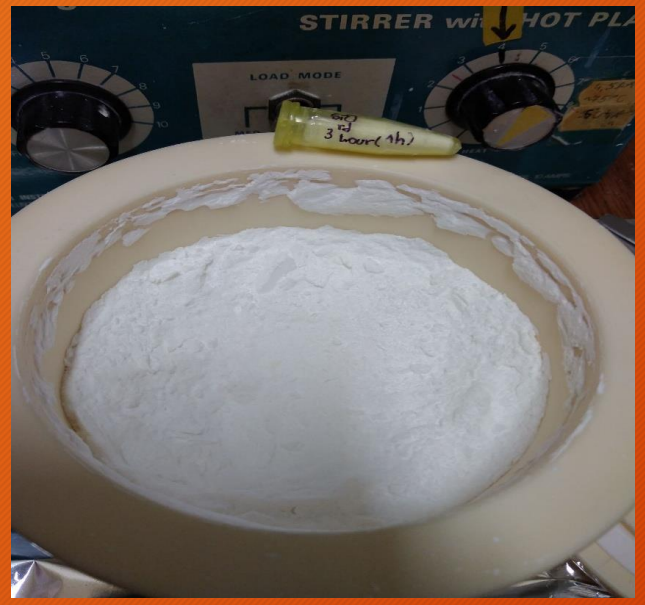


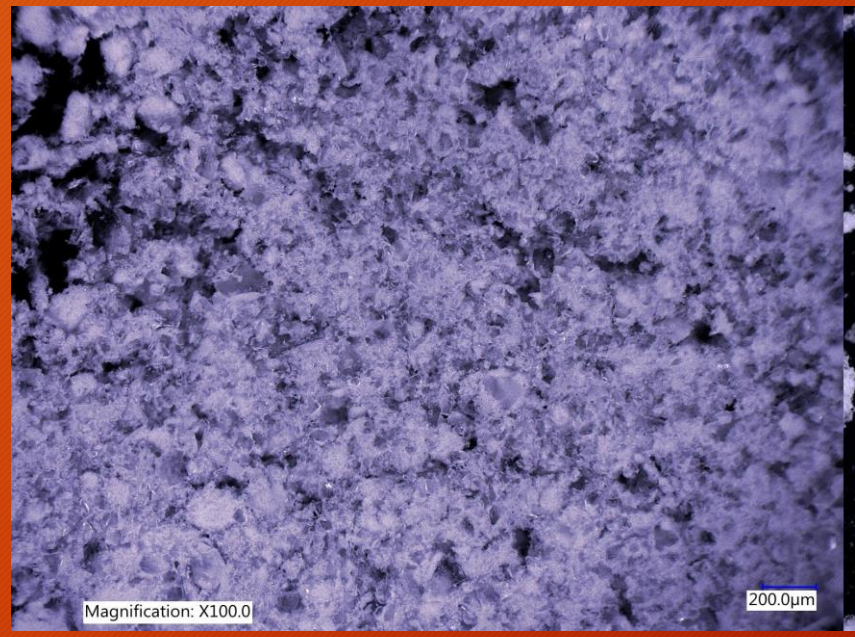


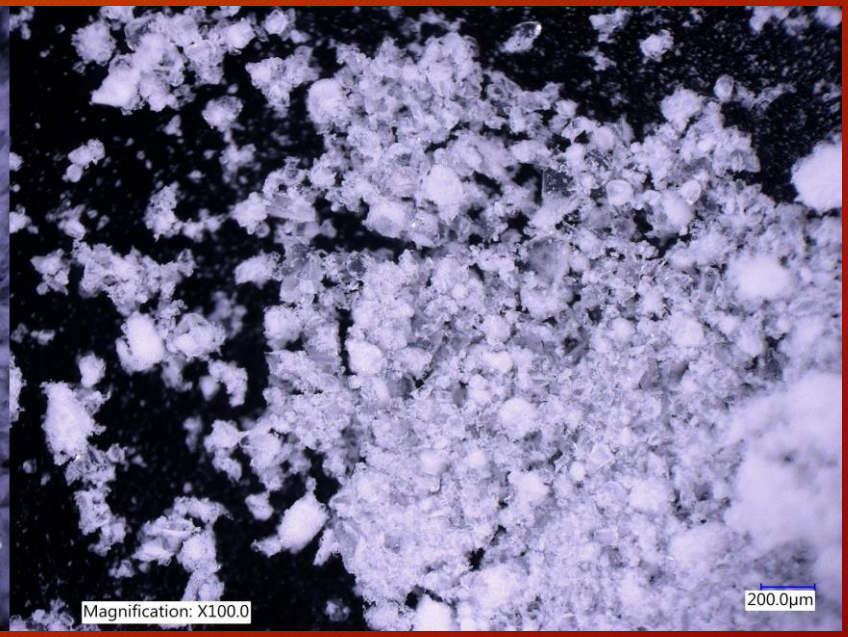
Milled Silica gel after 2 hours





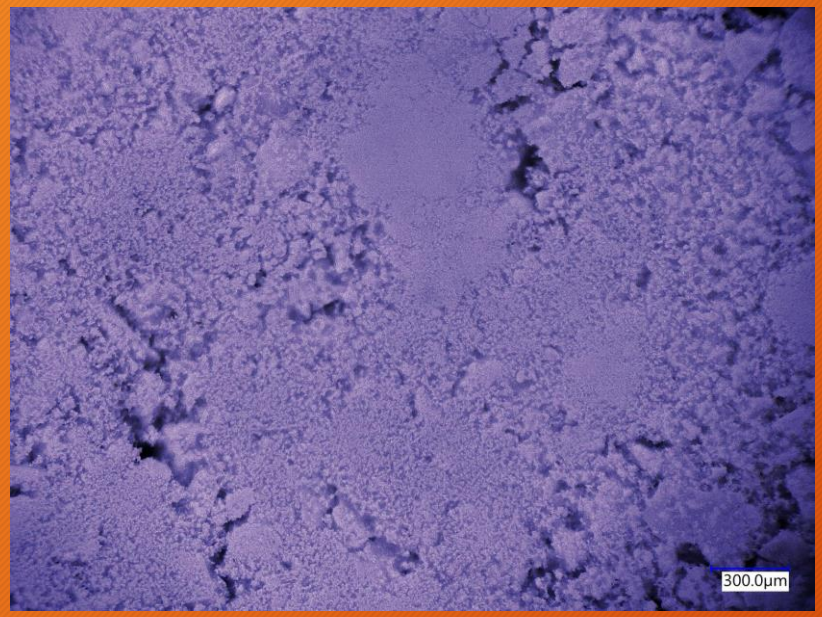


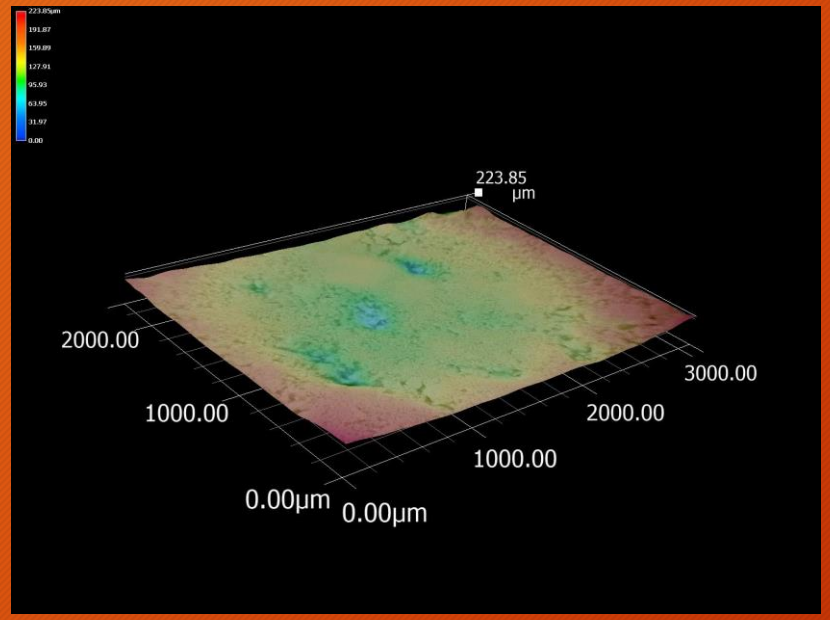


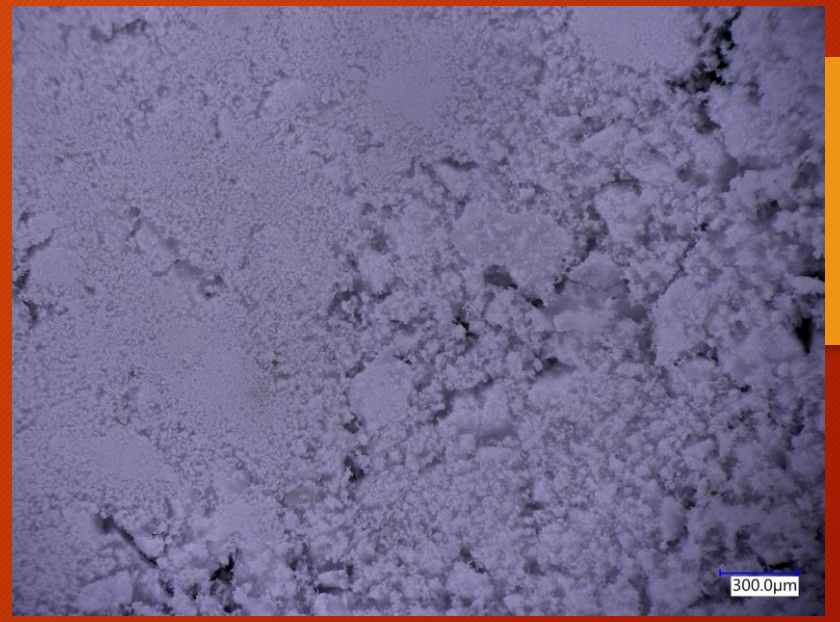


Milled Silica gel after 3 hours (Silica powder)

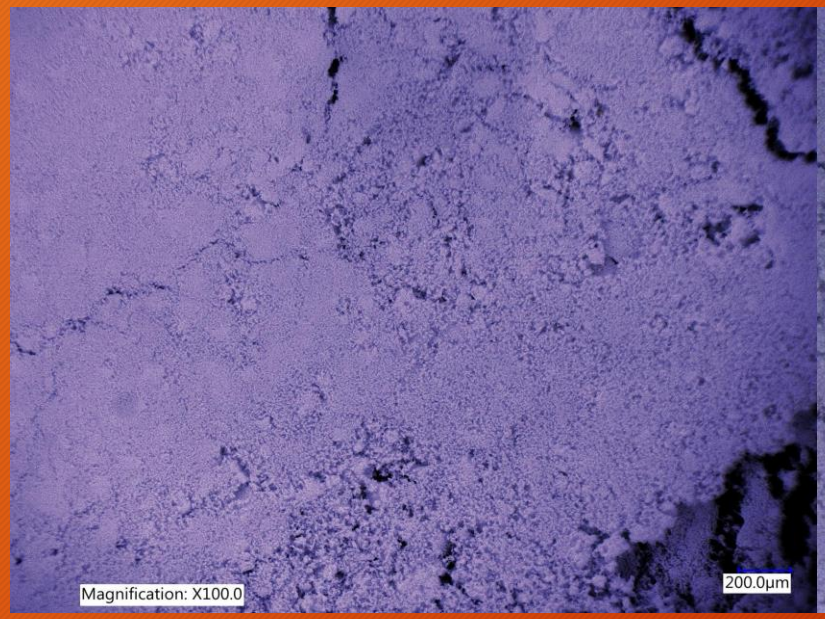
Samples analysis results using Keyence for calcinated eggshells at 900 °C for 12h

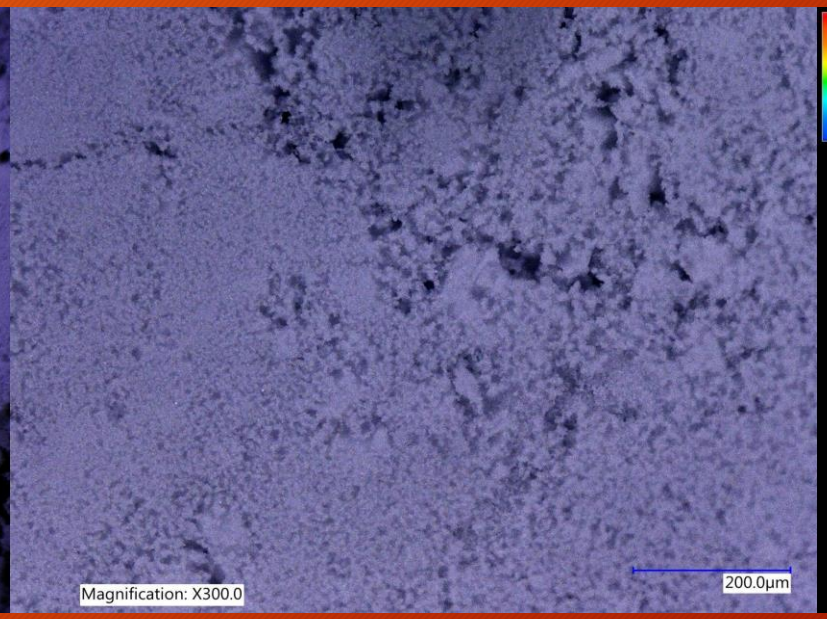


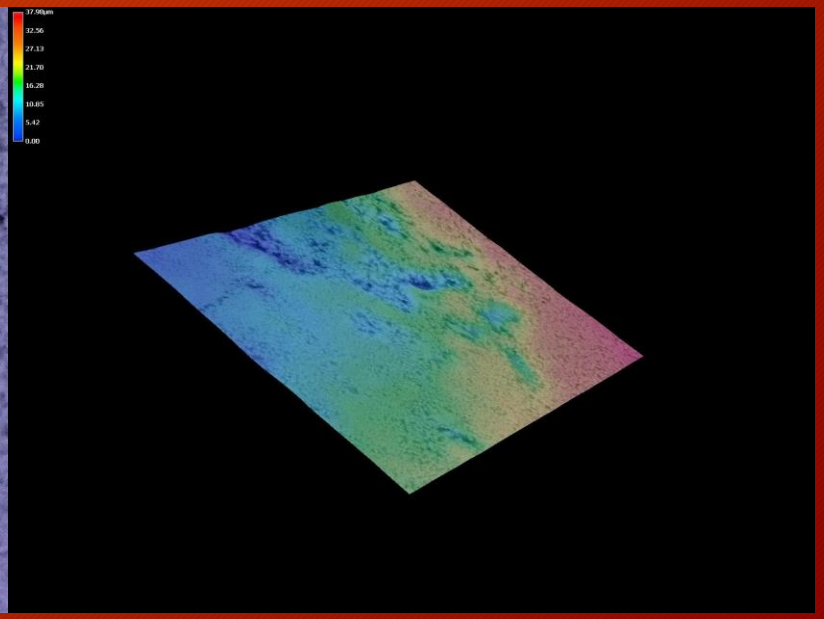




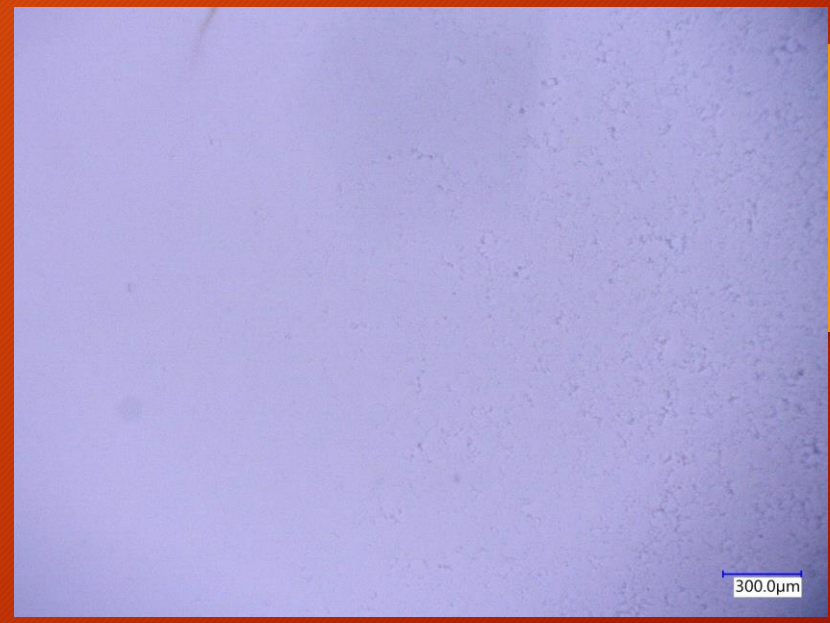
Samples analysis results using Keyence for calcinated eggshells at 900 °C for 7h



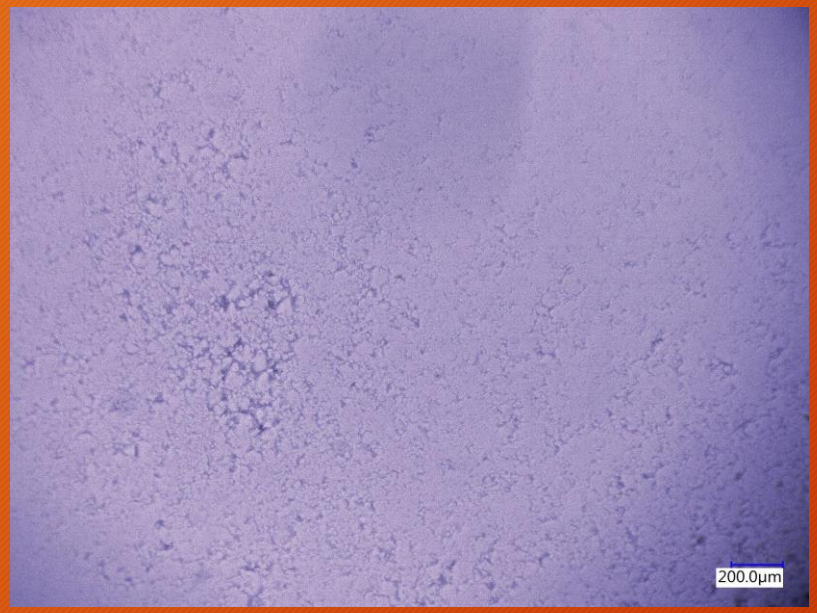


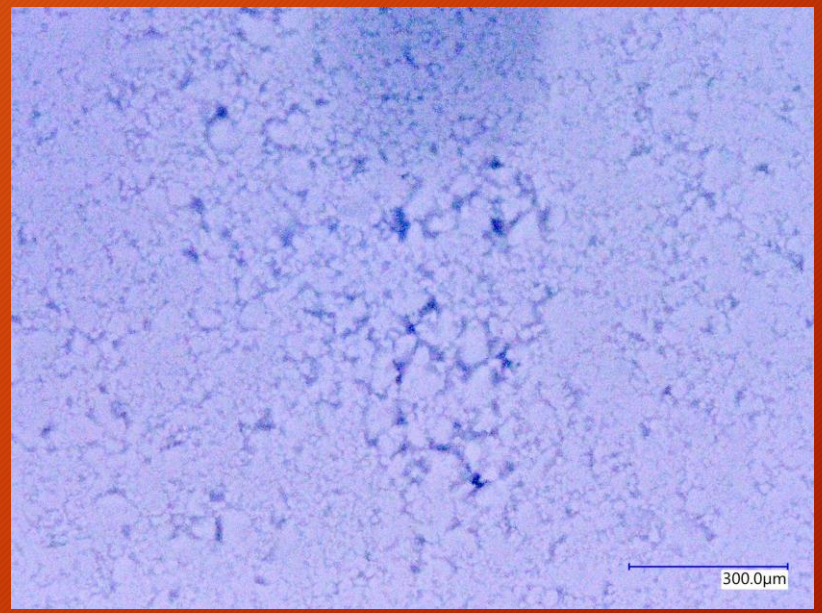


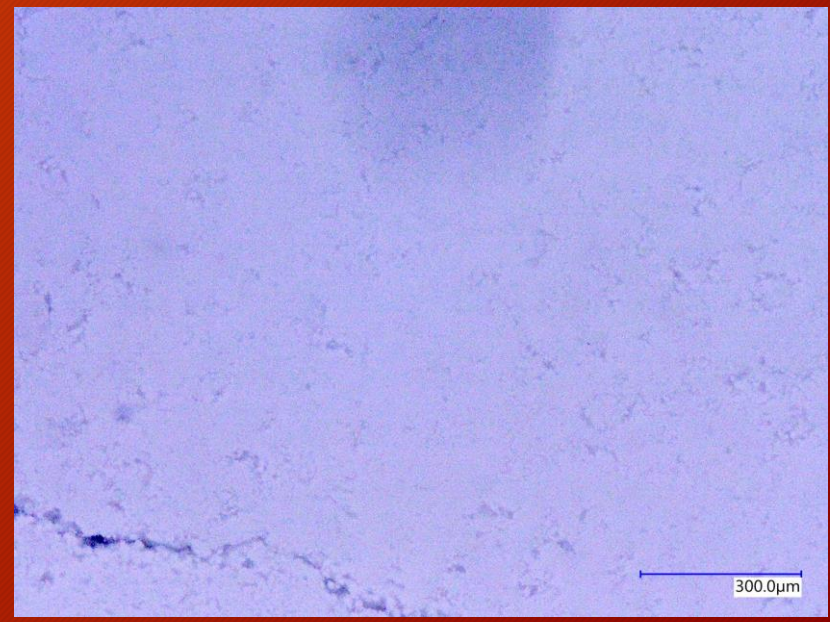
Samples analysis results using Keyence for 10% CaO (12h) + 90% SiO2 with PEG



Samples analysis results using Keyence for 40% CaO (7h) + 60% SiO2 with PEG







Research Plan

1. semester (successful):

1) Powder technology (Dr. Balázsi Csaba)
2) Biomaterials for medical applications (Dr. Balázsi Csaba)

2 semester:

1) Transmission electronmicroscopy for structural investigations of different materials (Dr. Balázsi Katalin), (not yet passed the exam)
2) Selected chapters of material testing methods I.: FTIR, HPLC/MS (Dr. Erzsébet Takács), SEM, STM, AFM (Dr. Judit Telegdi), (not yet passed the exam)
3) Hungarian I (Dr. Szloboda József Sándorné Katalin), successful

Conferences:

Submitted an abstract to participate in [Virtual] EUROPEAN CONGRESS AND EXHIBITION ON ADVANCED MATERIALS AND PROCESSES - EUROMAT 2021, September 12-16 (Poster)

Research Plan

Powder number	Powder	CaO (g)	SiO2 (g)	
1	10% CaO (12h) + 90% SiO2	10	90	100 g
2	20% CaO (12h) + 80% SiO2	20	80	100 g
3	30% CaO (12h) + 70% SiO2	30	70	100 g
4	40% CaO (12h) + 60% SiO2	40	60	100 g
5	50% CaO (12h) + 50% SiO2	50	50	100 g
6	60% CaO (12h) + 40% SiO2	60	40	100 g
7	70% CaO (12h) + 30% SiO2	70	30	100 g
8	80% CaO (12h) + 20% SiO2	80	20	100 g
9	90% CaO (12h) + 10% SiO2	90	10	100 g
10	40% CaO (7h) + 60% SiO2	40	60	100 g

Research Plan

- ✓ Preparation of bars and discs under the pressure of 200MPa in dry conditions for each composition
- ✓ Sintering them under different temperatures (800, 900, 1000°C) for 1h (for tribology, hardness, bending strength, density tests along with emerging them in SBF solution for testing the biological properties
- ✓ Analyzing all the samples (the powders along with sintered forms) by diffrent thechnical tools (Keyence, SEM-EDS), XRD and some of them with TEM/FTIR/ XPS
- ✓ Continuing the litterature overview on Ca-Si as dental closure materials

Thank you for your attention