Processing and Characterization of Tricalcium Phosphate with SrO and ZnO Dopants for Bone Grafts

Johanna Feuerstein, Shashwat Banerjee, Scott Rewinkel, Amit Bandyopadhyay and Susmita Bose
W. M. Keck Biomedical Materials Research Laboratory
School of Mechanical and Materials Engineering
jdfeuerstein@gmail.com

Abstract: The purpose of this study is to develop a ceramic material with the proper strength degradation kinetics to be used as bone implants. Tricalcium phosphate (TCP) ceramics have gained a lot of attention over the past several years for their similarity to bone. However, they show poor mechanical strength and uncontrolled strength degradation. For this reason, various dopants and their affect on the strength and strength degradation of TCP *in-vitro*- are being studied.In this study, three different TCP-based compounds were studied: (i) 0.25 wt% ZnO, (ii) 1 wt% SrO and (iii) binary dopants with 0.25wt% ZnO and 1 wt% SrO. The compounds were sintered at 1250° C. It was found that the binary dopants had the highest weight increase at both 14 and 28 days, indicating the most hydroxycarbonate apatite (HCA) formation on the surface of the disks and the least amount of dissolution. The SrO is proving to have the most stable degradation kinetics. The binary dopant also seems to be the most bioactive as it has the most HCA formation.

Objective: To develop a ceramic material with adjustable degradation kinetics to be used as implants for bones in the body.

Introduction

- Tricalcium Phosphate (TCP) materials have recently gained attention and are being studied as biodegradable materials and tissue engineering scaffolds.
- Controlled degradation kinetics are necessary for these biomaterials to have applications as implants for bones in the body as different parts of the body require different biodegradation times. [2]
- Despite extensive research, the biodegradation of TCP was not easy to be effectively varied or modulated.
- Addition of certain metals can help to regulate the degradation kinetics.

Maxillofacial surgery:
3-6 months to repair.

Spinal fusion:
9-12 months to repair.

Why SrO and ZnO?

ZnO
- Found in Natural Bone
- Shown to have effect on bone regeneration
- Studying 0.25 wt% ZnO

SrO
- Found in Natural Bone
- Aids in osteoblast formation.
- Prevents osteoclast formation
- Studying 1 wt% SrO

Binary - 1 wt% SrO + 0.25 wt% ZnO

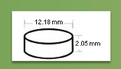
Synthesis and Formation of Ceramics

Wet Mixing on Ball Mill → Uniaxially Pressed → Cold Isostatic Press 60 ksi for 5 minutes → Sinter at 1250 C → Place in Simulated Body Fluid (SBF)

Machine used for uniaxial press.ing

- 2 shapes of ceramics were formed: cylinders and disks as shown:

750 mg powder pressed at 58 MPa for 1 minute

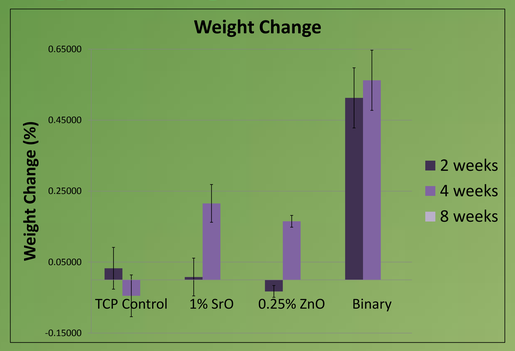


500 mg powder pressed at 145 MPa for 1 minute

.

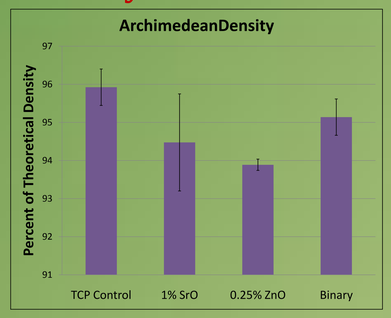
Results

Weight Change

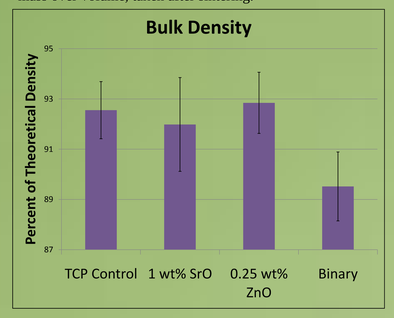


- Weight change is a competition between Hydroxycarbonate apatite (HCA) formation and TCP dissolution.
- Binary exhibited the highest percent weight increase from its original weight, indicating the most HCA formation and least amount of dissolution.
- TCP control indicates it is dissolving at about the same rate as the HCA formation.

Density



Relative density of synthesized TCP as compared to the theoretical density of TCP at 3.07 g/cm^3 Calculated as total mass over volume, taken after sintering.

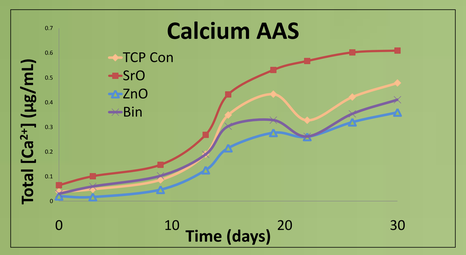


Relative density of synthesized TCP as compared to the theoretical density of TCP at 3.07 g/cm^3 . Metal addition to TCP was not calculated into the theoretical density due to the relatively small concentrations.

- TCP control achieved the highest Archimedean density, while ZnO was the lowest.
- Binary has higher Archimedean density that either of the single dopants, but a lower bulk density.

In vitro Study:

Atomic Absorption Spectroscopy

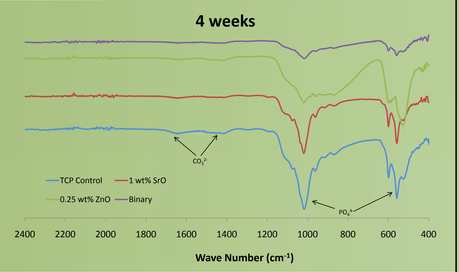


- Calcium release is an indicator of TCP dissolution.
- Calcium uptake occurs when HCA is forming.
- SrO released the highest total amount of Calcium.
- Binary released one of the lowest Ca concentrations.

To determine the amount of TCP dissolution, used SBF solution was collected from incubating samples every 3-4 days. Amounts were calculated as rates, and then added to get total concentration. Volume change was assumed to be negligible.

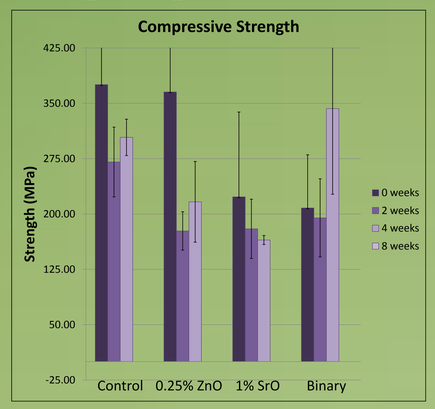
FTIR

- CO_3^{2-} was quantitatively analyzed in the apatite layer on the surface of the samples.
- TCP control exhibited the most intense phosphate peaks.

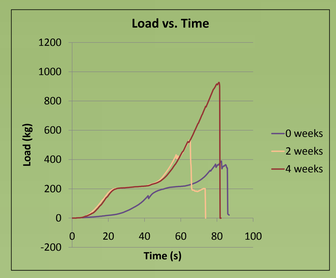


In vitro Study:

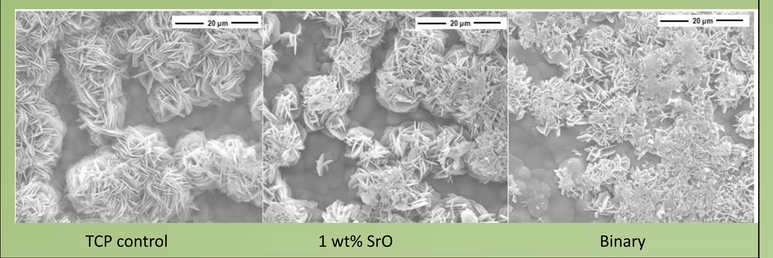
Strength Degradation



- TCP control achieved the highest strength, but is exhibiting uncontrollable degradation kinetics.
- SrO is degrading at a slow and constant rate.



Scanning Electron Microscopy



Scanning electron microscopy of samples after 4 weeks in SBF. Images taken at 5000x resolution.

- Binary formed the most HCA, but had the finest size.

Conclusions

- Addition of SrO and ZnO dopants did not increase the density.
- SrO has the most controllable strength degradation kinetics up to this point.
- Binary dopants has the highest HCA formation, and a comparatively small amount of dissolution.

Future Plans

- Carry SBF study out to 16 weeks
- Analyze XRD data to determine amounts of α- and β-TCP.
- Cell culture studies to determine cell attachment to the surface of the sample over time
- In-vivo study (using rat model) to determine ion concentration using atomic absorption spectrophotometer

References:
1. Amit Bandyopadhyay, Sheldon Bernard, Weichang Xue and Susmita Bose, "Feature Article: Calcium Phosphate Based Resorbable Ceramics: Influence of MgO, ZnO and SiO2 Dopants," J. Acer. Cer. Soc., 89 [9], pp. 2675-88 (2006).
2. Weichang Xue, Kelly Dahlquist, Ashis Banerjee, A. Bandyopadhyay and S. Bose, "Synthesis and characterization of Tricalcium phosphate with Zn and Mg based dopants," J. Mat. Sci.-Mat. in Med., **19** [7], pp. 2669-2677 (2008).

Acknowledgements: CIB Fellowship and National Institute of Health (Grant # NIH-R01-EB-007351) for Financial Support