Fabrication of Amorphous Magnesium-Calcium Phosphate Powder Via Ethanol induced formation for Tissue Engineering Applications

Elham Babaie, Huan Zhou, Sarit B Bhaduri.
University of Toledo, Toledo, OH 43606

Statement of Purpose: This paper reports a method of synthesis of amorphous Magnesium-Calcium (AMCP) Phosphate powders via ethanol induced formation. Apart from calcium, magnesium is an important constituent of the bone composition. Both the elements have been involved in bone mineralization and regeneration [1]. In this study, AMCP powder was precipitated using ethanol in media with calcium as stabilizer. To the best of our knowledge, this is the first attempt to synthesize AMCP via ethanol and calcium as stabilizer. The precipitates were identified by x-ray diffraction to be either contains Struvite or amorphous magnesium phosphate. Our finding revealed that with the change in the ratio between Mg and Ca, different phases formed. Predominantly AMCP formed with equal molar ratios of magnesium and calcium. Other crystalline products formed by varying the molar ratio of Mg/P, Mg/Ca and drying method.

Methods: The AMCP powder was synthesized in alcoholic medium. The reaction solutions were prepared by mixing $Mg(NO_3)_2.6H_2O$, $Ca(NO_3)_2.4H_2O$, $MgNO_3.6H_2O$, ethanol, and NaH_4PO_4. Molar ratios of Mg/Ca and Mg-Ca/P were varied between 0.5 and 2. The solution containing both $Ca(NO_3)_2.4H_2O$ and $MgNO_3.6H_2O$ in ethanol and water added to a solution of $(NH_4)_2HPO_4$ in water and ethanol (PH=10.3) at 37° C. The precipitates were collected via centrifuge. As a final step the centrifuged precipitates were dried using an oven, a freeze dryer, and in the ambient condition, room temperature environment. The powders were evaluated by XRD and SEM [2].

Results: The XRD images of AMCP powders with equal molar ratios of Mg and Ca dried using different methods are shown in Figure 1. As shown, amorphous structure resulted for both oven and freeze dried samples, while crystalline structure occurred in samples left at the room temperature.

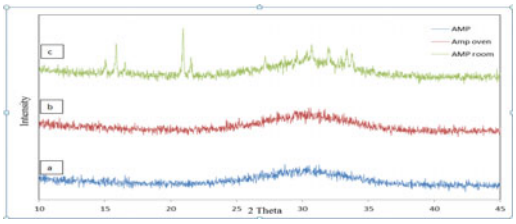

Figure 1 a) XRD patterns of AMCP, a) freeze dried, b) ovendried, c) at room temperature

Another important factor to induce amorphous and transition into crystal was molar ratio of Mg/Ca. Deviation from ratio of Mg/Ca=1 leads to crystalline structure as shown in Figure 2-3 for the freeze dried samples. Increasing phosphate concentration also causes to have poorly crystalline structure (Figure 2). AMCP is synthesized as agglomerated microspheres or irregular clusters with plate-like structure. However, the crystalline powders possess needle like shapes for Mg/Ca =2. The amorphous powders when soaked in SBF stayed amorphous after 24 hours (data not shown).

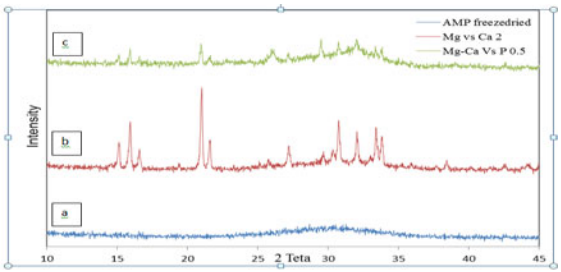

Figure 2. a) SEM patterns of a) AMCP powder b) Mg/Ca=2 c) (Mg-Ca)/P=2 for freezedried samples

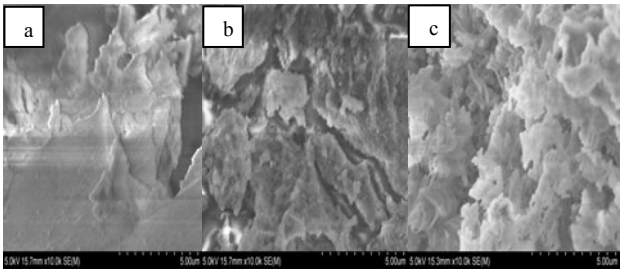

Figure 3. a) XRD patterns of AMCP powder of freeze dried b) oven dried c) Mg/Ca =2 freeze dried

Conclusions: Amorphous magnesium-calcium phosphate powders were successfully fabricated via the ethanol mediated synthesis. This paper reports the significant role calcium plays as a stabilizer to keep amorphous structure Future studies will be focused on effect of AMCP on the bone tissue regeneration and its related mechanism.

References:

[1] S. R. Kim, J. H. Lee, Y. T. Kim, D. H. Riu, S. J. Jung, Y. J. Lee, S. C. Chung, and Y. H. Kim, "Synthesis of Si, Mg substituted hydroxyapatites and their sintering behaviors," *Biomaterials*, vol. 24, no. 8, pp. 1389–1398, 2003.

[2] A. Rodrigues, A. Lebugle, "Influence of ethanol in the precipitation medium on the composition, structure and reactivity of tricalcium phosphate," *Colloids and Surface, A: Physicochemical and Engineering Aspects*,vol. 145, pp. 191-204, 1998.