**Rapid Formation of Porous β-CPP pellet by Microwave Sintering of DCPA Powders**

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**Statement of Purpose:** This study reports the results of microwave sintering of DCPA (Dicalcium phosphate anhydrous, Monetite) powders to fabricate monolithic beta-calcium pyrophosphate (β-CPP). Calcium phosphates being present in the bone mineral, play an important role as biomaterials in orthopedic applications. As CPP is not easily synthesized, it did not get a wider attention for biomedical uses. However CPP is still a good candidate with clinically relevant mechanical properties and degradation rates for orthopedic applications. Microwave sintering is a promising method for CaP consolidation as compared to normal furnace sintering. It is believed that: 1) the microwave sintering can produce better mechanical properties and unique microstructure [1]; and 2) the resulting CaPs are beneficial for cell proliferation [2].

**Methods:** The DCPA powders were synthesized by reacting mixtures of calcium hydroxide ($Ca(OH)_2$), setting solution, and DI water. 15 ml setting solutions were prepared using following compositions (Table 1.).

Table1. Compositions of two setting solutions.

| | |
|---|---|
| S1 | 0.0032g of citric acid monohydrate, 1.95ml of DI water, and 13.05ml of phosphoric acid (85% $H_3PO4$) |
| S2 | 0.0032g of citric acid monohydrate, 6g of NaHCO3, 1.95ml of DI water, and 13.05ml of phosphoric acid (85% $H_3PO4$) |

Three different groups of DCPA powders were synthesized by manually mixing of calcium hydroxide with setting solution and DI water in an agate mortar. The compositions of are shown in Table 2. After final dispersion, powders were pressed into the mold of 12mm diameter and 8mm height to make it a pellet for further sintering. Before sintering, the pellet samples were dried in air for 24 hours. For microwave sintering, the pellets were placed between two SiC susceptors and surrounded by alumina fiber blanket. The power of microwave furnace was manually controlled until the temperature of sample reached 1100 °C. The temperature was measured by a high temperature pyrometer. The soaking was kept at 1100 °C for 20 minutes for all samples.

Table 2. Compositions of three groups of DCPA powders.

| | |
|---|---|
| M1 | 3g of $Ca(OH)_2$, 2.774ml S1 and 1.5ml DI water |
| M2 | 2.47g of $Ca(OH)_2$, 3ml S2 and 1ml DI water |
| M3 | 2.4 g of $Ca(OH)_2$, 0.6g of $Mg(OH)_2$,3 ml S2 and 1ml DI water |

The as-synthesized powders and sintered pellets were analyzed by XRD and SEM for characterization.

**Results:** The XRD spectra of the DCPA powder (Figure 1.) showed DCPA powders were synthesized with different additions. Sinterd pellets examined by XRD (Figure 2.) showed that DCPA transformed to beta-calcium pyrophosphate (β-CPP).

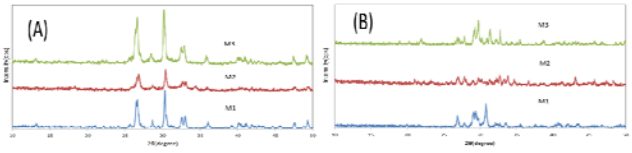


Figure 1. (A) XRD patterns of DCPA powders M1, M2 and M3, (B) XRD patterns of sintered pellets.

SEM images of sintered pellets (Figure 3.) showed that the particle sizes of M2 and M3 are much larger than the particle size of M1. From images (D) and (E), it is seen that larger pores were formed on M3 pellet than M2 pellet after microwave processing. As a result of low melting point elements Na and Mg were added into the DCPA compositions, the large pores formed on the surface and inside of M2 and M3 samples. Moreover, a small amount of $Mg(OH)_2$ facilitates to the formation of larger pores.

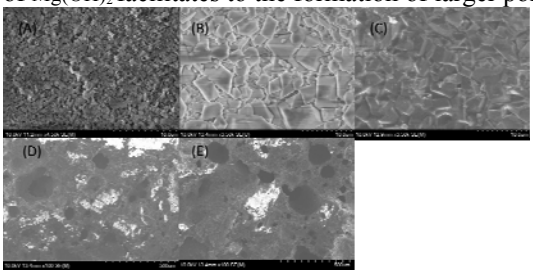


Figure 2. SEM images of (A) M1 sintered pellet, (B) sintered pellet of M2, (C) sintered pellet of M3, (D) large pores formed in M2 pellet, (E) large pores formed in M3 pellet.

**Conclusions:** Microwave sintering was found to be beneficial for fast consolidation of DCPA powders and production of porous beta-CPP from DCPA. The additions of Na and Mg are beneficial for forming large pores during the microwave heating process. As recent literature showed, large pores in the structure help supporting cell attachments and new bone growth, we can believe the β-CPP formed by microwave sintering could be attractive in bone tissue engineering application. Future study will be focused on optimizing of composition and sintering parameters for the specific use in the future.

**References**:

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[2] Darcy E. Wagner, Andrew D. Jones, Huan Zhou, Sarit B. Bhaduri,"Cytocompatibility evaluation of microwave sintered biphasic calcium phosphate scaffolds synthesized using pH control," Materials Science and Engineering: C, Vol. 33, no. 3 , pp. 1710-1719, 2013.



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