# Physico-chemical and Histological Comparison of Injectable Calcium Phosphate Cements: α-BSM® and $CaP_3$

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**Statement of Purpose:** As our understanding of orthopedic biomaterials continues to evolve, our ability to enhance their mechanical stability, efficacy and efficiency improves. $CaP_3$ is a calcium phosphate cement designed to improve upon the current features of α-BSM. The objective of this study was to evaluate the chemical and physical properties as well as the mechanism of resorption for $CaP_3$ with respect to α-BSM®.

**Methods:** $CaP_3$ and α-BSM® (ETEX Corporation, Cambridge MA) were prepared by mixing each with saline using a liquid-to-powder ratio of 0.45 and 0.7 respectively (histology l/p 0.4 $CaP_3$). All samples were incubated in 100% humidity at 37ºC for their respective time points. Initial setting times were assessed using a modified ASTM C266-04[1], consisting of a 1mm steel needle moving along a vertical axis under a constant load of 400 gf and a well of 5 mm depth to hold the putty. A universal testing machine (LRX-5K, Lloyd Instruments) was used to measure the compressive strength of cylindrical pellets (6mm dia and 12 mm height), incubated for 2 to 24 hours and tested wet along their height at a cross head speed of 1 mm/min. Phase analysis of the set cements was carried out using XRD and FTIR. At each time point, samples were frozen in liquid nitrogen and lyophilized for 24 hours before being powdered. Ca/P ratios were determined by microprobe energy dispersive analysis (EDAX). Porosity was determined by measuring the density of the cement mass compared to the theoretical density of the phase using mercury pycnometry. Reaction enthalpy was measured using a differential scanning coulometer (DSC) in isothermal mode. Solubility was determined by measuring $Ca^{2+}$ concentrations by ICP over a 28 day period.

Injectability was tested following the methods presented by Baroud[2] using a customized rheometer designed to hold a standard 5 mL luer lock syringe. A 50-mm 11-gage cannula was attached to the syringe prior to testing on a universal testing machine at a cross head speed of 2 mm/s. A plot of injection force versus displacement was obtained.

Cellular resorption was analyzed by histological cross sections of 17 skeletally mature rabbit femoral defects (5 mm dia and 10 mm deep) placed on the lateral femoral condyle and filled with either $CaP_3$, α-BSM or left empty. Animals were sacrificed at 6, 12, 26, 38 and 52 weeks and the femora harvested and processed for undecalcified histology.

**Results/Discussion:** Initial setting times for α-BSM and $CaP_3$ were determined to be 17.5 ± 2.5 and 3.5 ± 0.5 minutes respectively. Compressive strengths of α-BSM and $CaP_3$ were found to be 12.8 ± 2.6 and 30.6 ± 4.8 MPa respectively at 2 hours and 5.2 ± 1.1 and 33.1 ± 4.2 MPa at 24 hours. XRD analysis demonstrated a corresponding diffraction pattern between the two cements at 48 hours which are similar to diffraction pattern found in natural bone (Fig. 1).

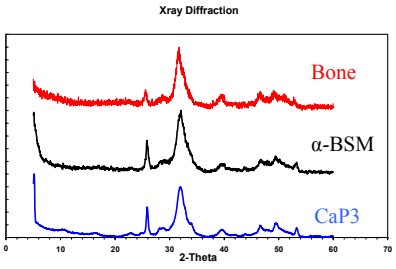


Figure 1. XRD pattern for cements (48 hours) and bone.

The Ca/P ratios for the cements were 1.43 ± 0.01 for α-BSM and 1.46 ± 0.05 for $CaP_3$, which falls within the typical range of endogenous human bone[3]. Observations of the enthalpy by DSC showed a mildly endothermic reaction for PCHA formation with the initial conversion rates for $CaP_3$ demonstrating a 10 fold increase over α-BSM. Total porosity of α-BSM and $CaP_3$ as determined by mercury pycnometry was found to be 66% and 57% respectively. The solubility of α-BSM was determined to be 2.2 times greater than that of $CaP_3$. The average injection forces were 6.16 ± 0.61 kgf for α-BSM and 3.11 ± 0.34 kgf for $CaP_3$.

Gross evaluation of the histological cross sections showed similar resorption characteristics with good bone-implant interface (Fig. 2). Both materials exhibited scalloping of the outer edges associated with osteoclastic resorption. New bone appeared to penetrate the material along fracture lines, with osteoclasts widening the fissures and osteoid filling in behind.

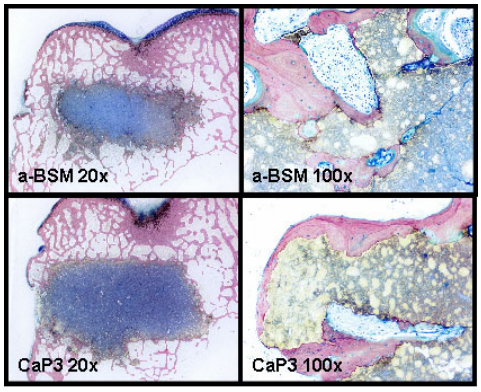


Figure 2. Fifty-two week histology slices.

**Conclusions:** $CaP_3$ shares the same chemical and crystalline identity as α-BSM with similar mechanisms responsible for cell mediated remodeling. However, $CaP_3$ demonstrates improvements in setting time, strength and injectability when compared to α-BSM®.

**References:** 1) Annual Book of ASTM Standards, 2) Bohner M. Biomaterials. 2005;26:1553-1563. 3) Komath M. Biomed. Mater. Res. 1999;43: 399-409