

**PROCESS DEVELOPMENT OF THE
APATITIC CALCIUM PHOSPHATE BONE
CEMENTS FROM NATURAL RESOURCES**

PhD (DISSERTATION)

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ABSTRACT

Study has been made on the preparation, characterization of apatitic calcium phosphate cement from available local raw material. Its feasibility as an orthopedic and in dental application has also been conducted. The present work also describes its characteristic properties including results of biocompatibility studies. Apatitic calcium phosphate cements (apatitic triphasic calcium phosphate, apatitic biphasic calcium phosphate and hydroxy apatite) have been prepared by precipitation and hydrothermal methods using local raw material (limestone) which is an easily available resource in Myanmar. The apatitic triphasic calcium phosphate cement (ATcp) block consisting of hydroxy apatite (HA), octacalcium phosphate (Ocp) and β -tricalcium phosphate (β -tcp) has been prepared by hydrothermal method. The nature of phase purity of cement block was analyzed by X-ray diffraction (XRD) and Fourier Transform infrared spectroscopy (FT-IR). The microstructure analysis using scanning electron microscopy (SEM) showed the nature of a monolithic piece. The structure has an apparent porosity of $\sim 42\%$. The apatitic biphasic calcium phosphate (ABcp) consisting of hydroxy apatite and β -tricalcium phosphate has been prepared by precipitation method. The X-ray diffraction analysis of the product I (dried sample, hydroxy apatite) revealed that ABcp was partially crystalline state. However, on heating at 800°C for 8 hrs, XRD pattern indicated a perfectly crystalline form of ABcp. This observation was supported by FT-IR measurement. The change in morphology regarding in the functional nature was inferred by the shift in the FT-IR frequency. The hydroxy apatite has been prepared by precipitation method. The nature of the precipitated cement was analyzed through X-ray diffraction and Fourier Transform infrared spectrometry. The results showed the phase to be hydroxyapatite. The microstructure analysis using scanning electron microscopy showed hydroxy apatite nano crystallite structure with particle size of a few micrometers. MALDI-TOF-MS

measurements on the prepared composites were done using dithranol (1, 8 - dihydroxy - 9 - anthrone) as matrix. The strong evidence for the formations of the carbonate apatite composites were achieved from MALDI-TOF-MS spectra of the composites. The potential yields of apatitic calcium phosphates, based on slaked lime (18.5 g) were estimated to be about; β -tcp - 79.39%; ABcp - 77.11% and HA - 76.45%. The prepared cements do not show appreciable dimensional or thermal changes during setting. The optimization of the prepared apatitic calcium phosphate cements were done by the variation of disodium hydrogen phosphate concentration, setting time, hardening time as well as compressive strength. The injectability was estimated by extruding through needle, and cohesive property was assessed by water contact method. The prepared apatitic calcium phosphate cements passed the toxicological screening which was done by haemolysis test. Hence, physico-chemical properties of prepared cements showed clinically relevant values, it may be used in the orthopedic and dental applications.

Keywords: apatitic calcium phosphates, precipitation method, hydrothermal method, orthopedic and dental applications, clinically relevant values, haemolysis test.