# CRYSTALLINITY AND POROSITY OF HYDROTHERMALY SYNTHESISED HYDROXYAPATITE NANOPARTICLES IN A MIXED SOLVENT SYSTEM

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The use of synthetic materials like calcium phosphate ceramics for repair and replacement of hard tissues, such as bone and teeth, has attracted the primary attention of researchers in the biomedical field, as a result of increasing osseous and dental defects. Among the calcium phosphate ceramics, hydroxyapatite (HA) is the major inorganic component that is stable and bioactive under physiological conditions, and also it displays remarkable properties. Such affinities have made HA an ideal bone graft substituent. However, high brittleness and low fracture toughness hinders the potential application of it as a bone graft.

Porous nature of synthesised HA is very important in allowing host bone in-growth and body fluid and nutrient circulation. But the porosity of the synthesised powders should match with that of natural bone apatite. Hence, it is essential to focus on the synthesis conditions, structure, crystallinity and morphology of HA to improve the properties for it to be more applicable. Herein we describe the synthesis of HA nanoparticles via hydrothermal method, without the use of any surfactant, under different pHs and solvent compositions (Water/ethanol solvent mixture, W/E). The calcium and phosphorous precursors (Ca-sucrate and $Na_2HPO_4.2H_2O$, respectively) were mixed thoroughly, at 80 °C, maintaining the Ca/P ratio at 1.67.

By studying the powder X-Ray Diffraction (XRD) patterns, HA synthesized at pH 11.0 was taken as to have the highest crystallinity and the particles were spherical in morphology. The degree of crystallinity has increased with increasing pH, whereas the mean crystallite size has decreased with increasing pH. The product was divided into equal parts and sintered, at 200 °C, for 12 h under varying W/E volume ratios. Powder XRD patterns and Fourier Transform Infra-Red (FTIR) spectra confirmed the formation of pure HA in all the samples. Only that with the increase of E/W ratio had the degree of crystallinity declined together with the presence of traces of $CO_3^{2-}$ impurities. The $D_{(002)}/D_{(300)}$ ratio, which is a measure of uniformity of crystallites, was determined for each sample. It shows that increase volumes of ethanol favour the 3D growth of particles over 1D growth. Hence, not only the crystallinity and the crystallite size but also the shape can be tuned by varying the W/E volume ratio.

We also describe the effect of W/E ratio on the porosity of HA samples. Porosity was calculated by the volume fraction porosity method. Although, a remarkable variation could not be observed from the porosity values, all the values for corresponding samples lie in the range 0.2 - 0.5 which is the porosity range that is employed in most applications of porous materials including HA. Sample with the code W2E3 has the lowest crystallinity (64.3 %) and a small crystallite size (23.32 nm) while W1E0 has the highest crystallinity value (74.6 %). But both have relatively similar porosity values.