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Phase Transformations of Amorphous Calcium Phosphate in Molten Salts

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Synthetic calcium phosphates (CPs) are widely used in medicine as bone substitutes due to their excellent biocompatibility, osteoconductivity, and chemical composition similar to those of natural bone [1]. Most frequently, crystalline solids such as calcium hydroxyapatite (HAp, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), tricalcium phosphate (TCP, $\text{Ca}_3(\text{PO}_4)_2$), or octacalcium phosphate (OCP, $\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$) are used for biomedical applications [2]. On the other hand, amorphous calcium phosphates (ACPs) also attract considerable attention in the field of biomaterials science and bone regenerative medicine due to their metastability, which can result in superior biochemical reactivity. Additionally, synthetic ACPs can be used as a precursor for the synthesis of other phosphate crystalline materials. While some CPs can be prepared directly by precipitation from aqueous solution, some phases can only be obtained by employing thermal treatment or conversion of less stable phases.

CPs can be characterized by different Ca-to-P ratios, which is usually fixed for crystalline materials with an exception for calcium-deficient hydroxyapatite (CDHA, $\text{Ca}_{10-x}(\text{PO}_4)_{6-x}(\text{HPO}_4)_x(\text{OH})_{2-x}$), where this ratio can vary. Unlike crystalline solids, synthetic ACPs can be prepared as a substance with different Ca-to-P ratios ranging from 1.2 to 2.2. This ratio strongly depends on the formation conditions such as synthesis media, pH, and presence of foreign ions. In this study, we investigate the crystallization behavior of substituted ACP with a total metal ion-to-P ratio of 1.5:1. Such an elemental ratio is most commonly found in amorphous precipitates obtained in alkaline media. The crystalline analogue of an ACP with that chemical composition is TCP.

The main goal of the present study was to investigate the phase transformations of ACP in different molten salts. The influence of the flux nature, annealing temperature and time, precursors-to-flux ratio were investigated in detail. The crystallinity, crystal structure and structural changes were evaluated by powder X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy. Scanning electron microscopy (SEM) was used for the characterization of morphological features of products.

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