Hydrothermal Synthesis of Zirconium Substituted Hydroxyapatite

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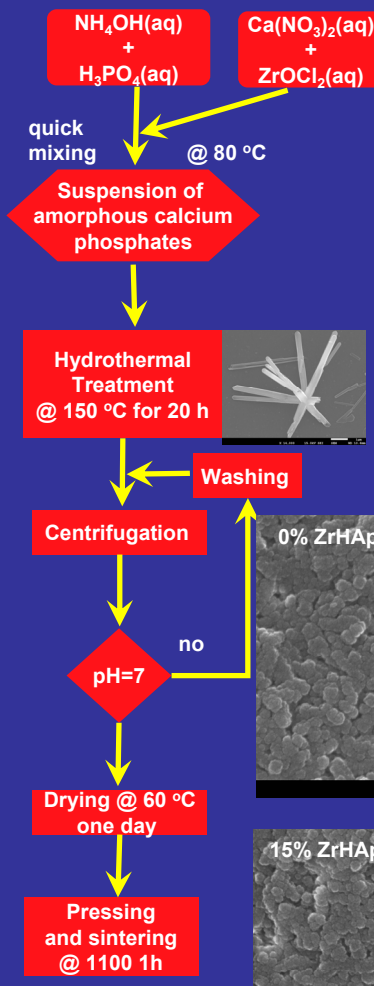
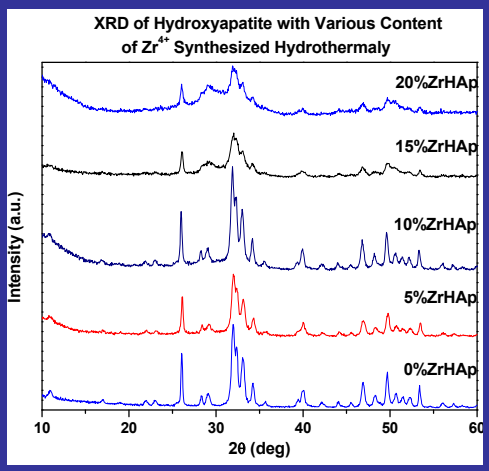
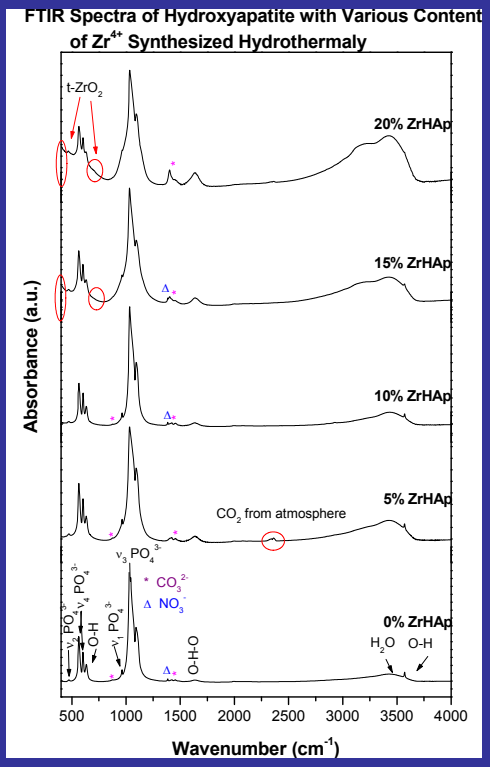
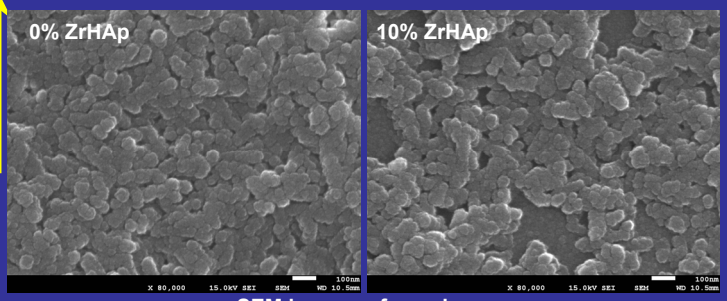
Introduction

As a main mineral constituent of bones, synthetic hydroxyapatite ($Ca_5(PO_4)_3OH$) as well as natural find usage in reconstructive treatment of bones in humans. Various forms of pastes, composite and ceramic implants and other are in medical use. For ceramic implants improvement of mechanical properties is one of main tasks. By partial substitution of Ca^{2+} ions in hydroxyapatite (HAp) with other metallic cations can be increased fracture toughness, mechanical strength and other properties in full dense sintered ceramic. Transition metal ions such as Ti^{4+} and Zr^{4+} can be substitutes in HAp (e.g. $Ca_{5-x}Zr_x(PO_4)_3OH$). Aim of this work is to investigate influence substitution possibility and limits, influence of Zr addition on properties of hydrothermal synthesized HAp powders, also their sinterabilty and phase composition in sintered ceramics.

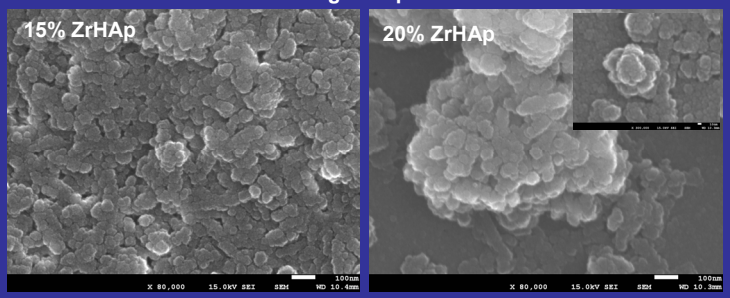


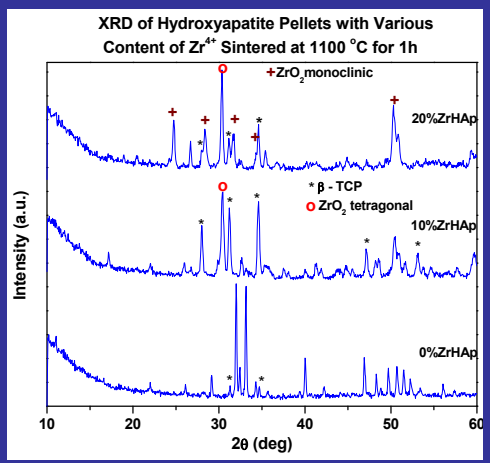






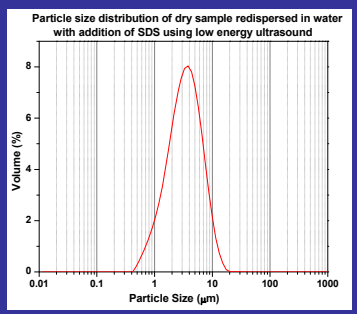
SEM images of powders





Experimental

HAp powders, with different amount of Zr^{4+} was prepared as is schematically shown. Two solution are mixed all at once @ 80 °C to form suspension. Concentration of $Ca^{2+}+Zr^{4+}$ in solution was 0.2 M. Concentration of PO_4^{3-} in solution was 0.12 M which is required for stoichimetric ratio of (Ca+Zr)/P in HAp. After mixing 100 ml of suspension was transferred to 190 ml Teflon® liner and sealed in stainless steal autoclave. For sintering, powders are pressed at 175 MPa in 6 mm pellets.



Particle size distribution of powders

Sample	Green Density (g/cm³)	Sintered Density (g/cm³)
0%ZrHAp	1.72	2.79
5%ZrHAp	1.85	2.05
10%ZrHAp	1.96	2.08
15%ZrHAp	1.93	2.11
20%ZrHAp	2.14	2.86

Results and Discussion

Phase analysis of all powders shows pure HAp phase. FTIR spectrum of 20%ZrHAp indicate presence of ZrO_2. Broadening in peaks in XRD and FTIR is due to structural disordering of HAp with ascending Zr content. All powders are strongly agglomerated with similar PSD as is shown on distribution plot. ICP AES analysis shows that 10%ZrHAp is close to theoretic HAp stoichiometry which is in accordance with FTIR and XRD results…

Sample	Ca/P mol.%	(Ca+Zr)/P mol.%	Zr/Ca mol.%
10%ZrHAp	1.58	1.64	4.3
20%ZrHAp	1.28	1.54	18

ICP AES analysis results