Studies on Yttrium Incorporated Hydroxyapatite Nanocrystals Prepared by Sol Gel Method

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Abstract. Yttrium doped hydroxyapatite nanocrystals were synthesized by sol gel method for three different concentrations of yttrium (3%, 6% & 9%). XRD was used to examine structural parameters, SEM was used to examine surface morphology, and EDS was used to determine the elemental composition. XRD revealed that the crystallite size was 28 nm. When XRD data is compared to JCPDS data, the strength peaks from XRD data are found to be similar. Space group of the grown samples has been found to be P63/m. From the FTIR analysis the presence of HPO4 –was confirmed which reinforces that HAP was synthesized.

Keywords: Crystallite Size, Doping, FTIR, Hydroxyapatite, XRD.

1 Introduction

$(Ca_5(PO4)_3OH)$ is a biomaterial with a diverse set of uses. It's also known as bone mineral because it crystallises in a hexagonal system. It's filler that's used to patch amputated bone [1]. HAP is used to coat modern implants such as hip replacements, dental implants, and bone conduction implants [2, 3]. Porous HAP is a form of HAP that is used to deliver drugs to specific areas of the body, such as the bones [4, 5].

HAP has a low host reaction .When metal ions are doped in HAP, catalytic properties are observed [6, 7].Antibacterial activity is observed when yttrium is doped in HAP [8]. It slows bacterial growth in the mouth slightly. Human periodontal fibroblast development is accelerated by it [9]. It attracts water molecules with a high affinity. It boosts the performance of HaP-cultured cells [10]. It makes the proton environment more favourable.

2 Experimental Procedure

Solgel was used to make yttrium doped hydroxyapatite nanocrystals. In deionized water, diammonium hydrogen phosphate was dissolved and stirred at 90°C in a magnetic stirrer. Separately dissolved calcium nitrate tetrahydrate and Yttrium nitrate were added to deionized water. Drop by drop, the above two solutions were applied. Ammonium hydroxide was used

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to keep the P_H at 10. For six hours, the solution was held at 90°C. The precipitates were filtered and washed before being dried for 22 hours at 110°C and then calcined for 2 hours at 700°C.

3 Characterization

3.1 XRD

The phases of the samples were identified by powder X-Ray diffraction done at SAIF, Cochin with Cu Kα (λ = 1.5406 A0). The XRD data set was obtained using two ranges ranging from 100 to 1200 with a 0.020 phase scale. The obtained data was compared with JCPDS and crystal structure was confirmed as hexagonal with space group of p63/m. shows the XRD profiles of pure and doped hydroxyapatite samples. Table 1 shows the crystallite size as determined by XRD. The relationship between 2 and crystallite size is depicted in Figure 2.

Table 1. Crystallite size from XRD

S.No	Sample	2θ in degrees	Crystallite size in nm
1	Pure HAP	31.796	28.1205
2	3%Y doped HAP	31.807	28.1115
3	6%Y doped HAP	31.836	28.0864
4	9%Y doped HAP	31.858	28.0676

The size of crystallites was affected by yttrium doping. If the concentration of yttrium rises, the size of the crystallite shrinks. Because the ionic radius of yttrium (0.09nm) is smaller than that of calcium, this proves that Y^{3+} ions replace Ca^{3+} ions in the HAP. (0.1nm). Bragg's law states that as the interplanar distance decreases, the lattice parameter decreases. This also shows that as the concentration of Y increases, the crystallite size decreases.

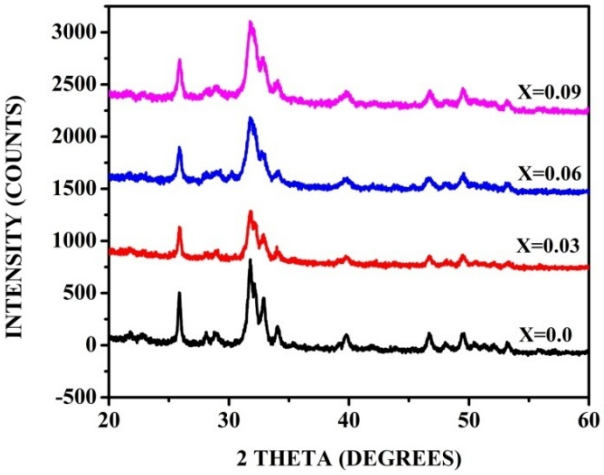

Fig. 1. XRD profiles for the pure and Yttrium doped Hydroxyapatite

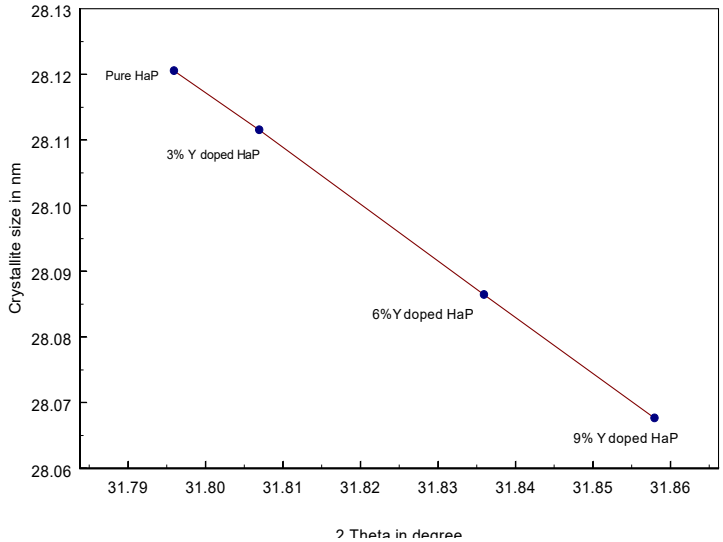

Fig. 2. Relation between 2θ and crystallite size of pure and Yttrium doped Hydroxyapatite

3.2 FTIR

Fourier transform infrared spectroscopy was used to validate the existence of functional groups. The spectrum was captured in the 4000-400cm-1 range. The spectrometer's resolution was 4cm-1. Many of HAP absorption peaks can be seen in FTIR spectra. Spectra were collected in the 472.56-3570.23 area. FTIR profiles of pure and doped Hydroxyapatite are shown in Figures 3a-3d. The FTIR peaks and their inferences are mentioned in Table 2.

Table 2. Peaks from FTIR and their corresponding inference

Sl. No.	Chemical group	pure hap	3%Y doped hap	6% Y doped hap	9%Y doped hap
1	OH^-	3423.65	3417.86	3514.93	-
2	CO_3^{2-}	1411.89	1413.82	1413.92	1415.75
3	Adsorbed water	-	2829.57	2833.43	2835.36
4	HPO_4^{2-}	-	877.61	876.61	875.68
5	PO_4^{3-}	601.79	603.72	603.72	603.72

Fourier transform-infrared spectroscopy is an analytical technique that uses the absorption or emission spectrum of materials to distinguish organic and inorganic materials. The existence of HAP is confirmed by FTIR performance. With a rise in yttrium concentration, we expect a decrease in the IR absorption bands of the phosphate groups of the samples.

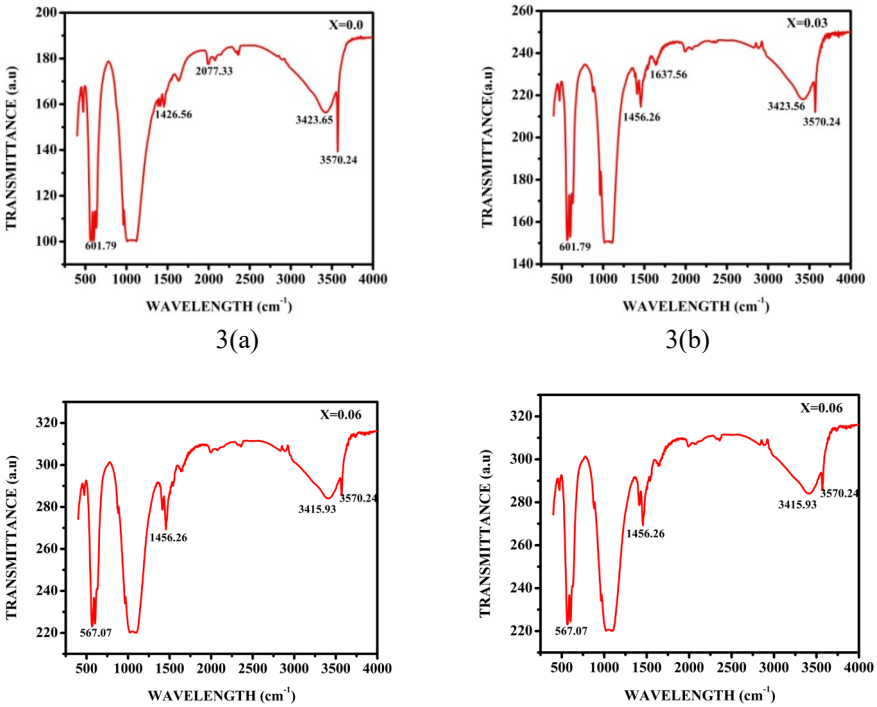

Fig. 3. FTIR profiles for the pure and doped Hydroxyapatite

3.3 SEM

SEM images of the samples were taken at International Research Centre, Kalasalingam University, Krishnan kovil. Figure 4 represents the SEM images of pure and doped hydroxyapatite.

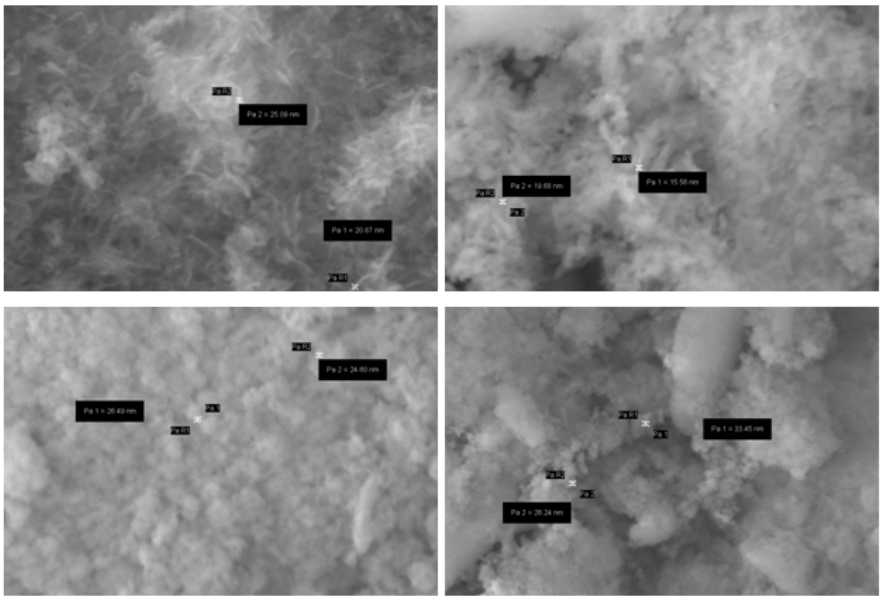

Fig. 4. SEM images of pure and doped Hydroxyapatite

3.4 EDS

Gandhigram Rural University performed the chemical characterizations of the samples. EDS representations of pure and doped hydroxyapatite are depicted in Figures 5a-5d. In pure and doped Hydroxyapatite, Table 3 shows the constituent elements.

Table 3. Constituent elements of pure and doped HAP

Sl. No.	Element	Pure HAP	3% doped HAP	6% doped HAP	9% doped HAP
1	O	38.81	62.05	42.24	14.94
2	C	-	14.76	15.23	24.68
3	Ca	42.26	20.12	18.27	2.67
4	P	16.32	10.03	9.57	5.55
5	Y	-	1.06	5.62	3.32

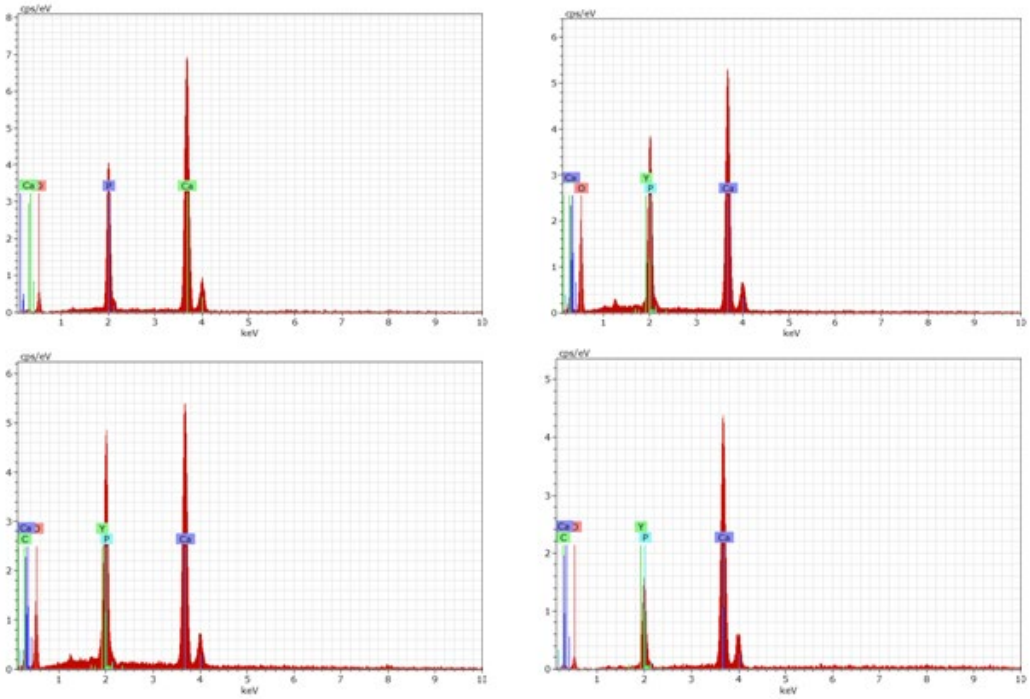

Fig. 5. EDS images of pure and doped Hydroxyapatite

4 Conclusion

The sol gel method was used to successfully synthesise yttrium doped hydroxyapatite nanocrystals in three different concentrations of yttrium (3 percent , 6 percent & 9 percent). The crystallite size was measured using XRD, and the crystallite size decreased as the Yttrium concentration increased. Using XRD data and JCPDS files as P63/m, the space category was calculated. The existence of an HPO4- group with a wavenumber in the order of 870cm-1 was confirmed by FTIR. SEM was used to examine the surface morphology, and EDS was used to determine the elemental composition.

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