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Synthesis and Characterization of Arginine modified Nano Hydroxyapatite

Bhoomika V. Jogiya[1], Karan R. Rathod[1], Kashmira P. Tank[1], Bharat B. Parekh[2] and M.J. Joshi[1]

[1]Crystal Growth laboratory, Department of Physics, Saurashtra University, Rajkot 360 005, India
[2]PanditDeendayal Petroleum University, Gandhinagar, 382 007, India.

Corres. author : bhoomika.cpi@gmail.com

Abstract: Hydroxyapatite (HAP)findslarge number of applications in biomedical field.In the present study the synthesis of L-arginine modified HAP nano particles was carried out using the surfactant mediated approach and characterized by different techniques. The FTIR spectra revealed the presence of amino acid in HAP samples. The powder XRD study indicated no major change in the crystal structure and alternation of unit cell parameters. The average crystallite size of L-arginine modified HAP nano particles is smaller than pure HAP nano particles. The TEM images indicated change in the morphology of L-arginine modified HAP nano particles from pure HAP nano particles.
Keywords– Hydroxyapatite; L-arginine; FTIR; TEM; XRD.

Introduction and Experimental:

Bio-mineral Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) or HAP finds large number of applications in bio medical field[1-2].The functionalization of arginine on the surface of HAP improves its gene delivery efficacy [3]. The amino acids exhibit synergetic effect on structure, morphology and surface properties of HAP [4]. Considering the significance of amino acid functionalization, the authors have used different concentration of amino acid L-arginine to functionalize nano HAP and characterized the samples by powder XRD, TEM and FTIR spectroscopy.

The samples were synthesized by surfactant mediated approach using AR grade chemicals. The 5 ml Triton X-100 was mixed with100 ml of 0.3 M calcium nitrate hexahydrate solution, which was treated with aqueous ammonia to set pH 9. Thereafter,100 ml 0.18 M potassium dihydrogen phosphate having pH 9 was added to former solution at 60˚C temperature with continuous stirring resulted into white precipitates, which was washed with double distilled water and air dried. L-arginine modified HAP nano particles were synthesized by adding different molar L- arginine solution, i.e. 0.3 M, 0.6 M, 0.9 M and 1.2 M to the calcium nitrate solution before the addition of surfactant and ammonia. The synthesized samples were labeled as pure HAP, 1 Ar-HAP, 2 Ar-HAP, 3 Ar-HAP and 4 Ar-HAP, respectively.

The Powder XRDstudy was carried out on the Bruker AXS D8 Advance using Cu ($K\alpha_1$) radiation. TEM images were taken by TECNAIKA20 (Philips) at 200 kV potential.FTIR spectrum was recorded on Thermo scientific Nicolet 1510 in KBr media.

Result and Discussion:

Figure 1 shows characteristic broadening of the XRD patterns of pure and L-arginine modified nano particles of HAP. The unit-cell parameters were calculated using the Powder-X software, given in table 1, indicates monoclinic crystal structure, which corresponds to the reported HAP(JCPDF-76-0694). The unit cell parameters are changed slightly due to L-arginine functionalization. Table 1 shows the average crystallite size calculated for(002) plane using Scherrer's formula. The lower values of average crystallite size for L-arginine functionalized HAP is due to preferential adsorption of amino acid on surface of HAP and inhibiting its growth [4].Figure 2 shows the TEM images of pure and L-arginine modified HAP. The morphology of the HAP nano-particles changes from needle to spherical as concentration of L-arginine increases, which may be due to the adsorptionof amino acid on the surface of HAP and inhibiting its growth. Needle shaped particle are found with length between 10-20 nm while the spherical particles are found with radius less than 10 nm. But for the pure HAP only needle morphology observed with the size ranging from 30-60 nm.

Figure 3 shows the FTIR spectra of pure and L-arginine modified HAP samples. The absorption peaks located in the range of 3404-3440 cm^{-1} are due to the stretching mode of O-H. The absorption at 1034-1022 $cm^{-1}$is due to the asymmetric stretching mode of PO_4^{-3}, while a small absorption at 950 cm^{-1} is due to the stretching mode of PO_4^{-3}. The medium absorption peak observed between 570-600 cm^{-1}in every spectrum is due to the O-P-O bending mode [5]. An absorption observed at 1644 cm^{-1} is due the asymmetric stretching mode of NH_3 which is overlapped with vibration of H_2O at 1630 cm^{-1} [6], therefore the broadening in the absorption is observed, which increases as the concentration of arginine increases. The symmetric stretching of NH_3 group is observed at 1455 cm^{-1}.The band corresponds to symmetrical stretching of $CO_2$is also observed at 1460 cm^{-1}, which is due to the absorption of CO_2 from atmosphere [1]. Very weak absorption is observed between 2900-2800 cm^{-1} may be due to the stretching of C-H bond of L-arginine[6], which is not observed in the FTIR spectrum of pure HAP.

Table 1 Unit cell parameters and crystallite size

Sr. No.	Sample Name	Lattice parameters ($\alpha=\beta=90^\circ$, $\gamma=120^\circ$)			Crystallite size
		a(Å)	b(Å)	c(Å)	
1.	Pure HAP	9.280	18.830	6.885	33 nm
2.	1Ar- HAP	9.245	18.785	6.885	18 nm
3.	2Ar- HAP	9.300	18.850	6.860	20 nm
4.	3Ar- HAP	9.300	18.859	6.885	12 nm
5.	4Ar- HAP	9.290	18.860	6.910	13 nm

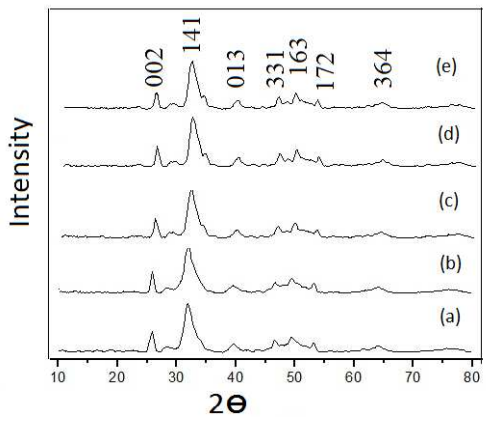

Figure 1 XRD patterns of (a) pure HAP (b)1Ar-HAP (c) 2 Ar-HAP (d) 3 Ar- HAP (e) 4 Ar-HAP

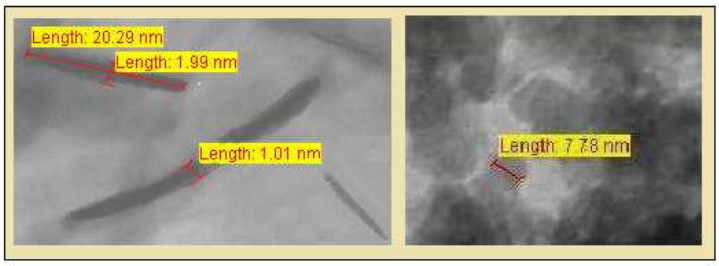

Figure 2 TEM images of (a) pureHAP(b) 4 Ar- HAP

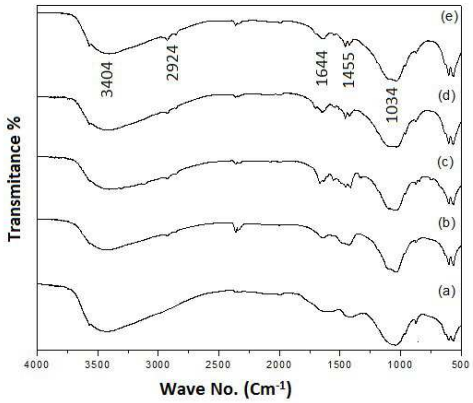

Figure 3 FTIR spectra of (a) pure HAP (b)1 Ar-HAP(c) 2 Ar -HAP (d) 3Ar-HAP(e) 4Ar-HAP

Conclusion:

Pure and L-arginine modified HAP nano particles were successfully synthesized by surfactant mediated approach. Due to adsorption of arginine on the surface of HAP, the inhibition of growth was observed in terms of reduction in the average crystallite size and changed in the morphology of nano particles. Change in the morphology of nano particles was observed from TEM images as the concentration of arginine increased. FTIR analysis confirmed the presence different functional group like O-H, P-O, N-H, C-H and C-O.

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