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Characterization of Beta-Tricalcium Phosphate (β - TCP) Produced at Different Process Conditions

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Abstract

β- TCP was synthesized by changing the process parameters and all samples were characterized in terms of density, porosity, XRD and FTIR analysis. Density rose and porosity decreased with rising of pH value and density found maximum (2.31 g/cc) at pH 10. Sharp peaks in the XRD diffractograms ensured the crystallinity of samples which increased after calcination and sintering process. Average crystal size was found 28.21 nm. IR spectrum at 943.19 cm^{-1} and 972.12 cm^{-1} appeared in FTIR analysis proved the presence of pure β- TCP exists in resulting samples. Performance to stress increased with pH and maximum compressive strength was found at pH 10. Calcination was found maximum 3.485 Nmm^{-2} at 900°C minimum 1.352 Nmm^{-2} at 800°C.

Keywords: Synthetic bone substitutes; Calcium phosphate ceramics; Hydroxyapatite; Beta-tricalcium phosphate; Process parameter; Wet chemical method

Introduction

Synthetic bone substitutes have been getting researcher's attention for the last few decades due to the disadvantages of the conventional autogenous and allogenous bone grafting. Calcium phosphate ceramics are most widely used as synthetic bone substitutes [1]. Among various calcium phosphate ceramics hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(OH)_2$) and Beta-tricalcium phosphate (β-TCP, $Ca_3(PO_4)_2$) are getting more concentration due to their biocompatibility and bioactivity [2-4]. The most significant feature of these materials is that they can construct direct chemical bond with bone tissue [5] and this interaction is related with and governed by the physical and chemical properties of the materials [6]. Both HAP and β- TCP are extensively used as bone substitute. HAP gets more importance for its use as bioactive and non biodegradable bone replacement material along with excellent mecahnical properties. But poor fracture toughness and wear resistance is being considered as the main shortcomings of HAP [7]. Whereas, β-TCP is bioresorbable and bioresorption occurs through osteoclastic activity [8,9]. Calcium phosphate powders can be synthesized through solid state process and wet chemical process that is neutralization process. Powders with different morphology can be obtained by neutralization process by varying process parameter. Studies show that particles with different physical properties such as surface area, particle size, density, porosity etc. affect the properties as well as performance of the final product [10]. As a result, the resulting powder can be used in different purpose depending on morphological conditions [11].

Though, β-TCP ceramic is very promising bone replacement material. But its poor mechanical strength and very fast resorption rate decreases its uses. The main objective of this study is to investigate the change in properties of porous beta tri-calcium phosphate produced in wet chemical method upon changing process parameters such as pH, concentration and calcinations temperature. In this study β-TCP powder was produced at different process conditions and the resulting powders were tested in terms of physical properties and mechanical performance.

Experimental

Powder synthesis

This β-TCP nano powders were synthesized by wet chemical process [12], where 500 ml of 0.4 M $(NH_4)_2HPO_4$ was added (drop wise) over 500 ml of 0.6 M $Ca(NO_3)_2.4H_2O$ for 3 hours which resulted in the formation of white precipitation. The pH of the suspension was maintained throughout the mixing process. The resulting white suspension was then stirred for 24 h by magnetic stirrer at room temperature which followed by filtration and the precipitate was washed with distilled water and then with 90% ethanol to improve the dispersion characteristic. It was then formed into a cake upon drying at 80°C for 24 hours and then crushed into powder and then calcined at 900°C for 3 hours. This sample was then preserved for characterization and termed as Sample S1 throughout the study.

Beside this, 9 different TCP samples were prepared by varring process parameters. These samples were: Sample S_2 (pH changed to 6 instead of 8), Sample S3 (pH changed to 2, this sample cannot be prepared as it get dissolved in this pH), Sample S4 (pH changed to 10), Sample S5 (Concentration of $(NH_4)_2HPO_4$ changed to 0.6 M instead of 0.4 M), Sample S6 (concentration of $Ca(NO_3)_2.4H_2O$ changed to 0.8 M instead of 0.6 M), Sample S7 ($(NH_4)_2HPO_4$ changed to 0.2 M), Sample S8 ($Ca(NO_3)_2.4H_2O$ changed to 0.4 M), Sample S9 (Calcined at 800°C) and Sample S10 (Calcined at 1000°C).

Characterization

Universal Testing Machine (FS-300, Testometric, England) was used to prepare TCP pellets from previously synthesized TCP powders which was then sintered in an electrical tube furnace (GSL-1600*40) in argon atmosphere at 1100°C temperature. Bulk density of the sintered TCP pelletis calculated from mass to volume ratio and that was used to calculate the porosity of sintered TCP [13], Total porosity = 1-M / (V × D). Where, M=mass of the sintered sample, V=volume of the sintered sample, D=density of the sintered sample. The phase analysis of the

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TCP was conducted in room temperature with Cu-Kα (λ=0.15406 nm) radiation in a continuous mode from $_2\theta$ range $_2$0-70° with a scanning rate of 2° per min by using Bruker: D8 ADVANCE XRD with SAX.

Subsequently average crystallite size of the TCP at 1100°C is calculated from X-ray line broadening using the Scherrer's equation [14] as:

$$D=\frac{0.89\lambda}{\beta\cos\theta} \quad (1)$$

Where, D is the crystallite size, λ is the X-ray wavelength (1.5406 Å) of Cu-Kα radiation, θ is the Bragg's angle and β is the full width at half-maximum (FWHM).

Chemical groups on the surface of calcined powders were analyzed by FTIR (PerkinElmer: Frontier FT-IR/NIR) in the wavenumber range 400 to 4000 cm^{-1}. In this regard the calcined powder was grinded with KBr.

Mechanical properties were observed using TMA (S11 TMA/ SS6300i).

Compressive strength wasmeasured by using Hertz equation [15]:

$$\sigma_{comp.}=\left(\frac{2P}{\pi DT}\right) \quad (2)$$

with the help of universal testing machine (Hounsfield H10K-S, U.K). Where, σ_{comp} is compressive strength, P is breaking load, D is diameter of the sintered compact, T is thickness of the sintered compact.

Results and Discussion

Bulk density and apparent porosity of different TCP samples were given in the Table 1. ResultS showed that the density was found maximum (2.31 g/cc) at pH 10 and it increased with the risen of pH value. Porosity was maximum (40.92%) when standard condition was followed. Both the density and the porosity found minimum (1.64 g/cc and 23.50% respectively) when $(NH_4)_2HPO_4$ conc. was 0.2 M. Figures 1-3 showed the XRD pattern of TCP samples sintered at different condition.

Sharp peaks in the diffractogram ensured that the products were well crystallized. The resulting graph was also well matched with the standard pattern of β-TCP or whitlockite with presence of a small amount of HAP in dried sample and very small amount of α-TCP. But, comparison with the whitelockite and sharpness of the graph indicates that crystallinity of all TCP samples increased after calcination and sintering procedure.

Average crystallite size of sintered β-TCP powder (computed by using Scherrer's formula) was found 28.21 nm. The IR characteristic peaks of phosphate groups appeared between 900-1160 cm^{-1}. The peak at 943.19 cm^{-1} and 972.12 cm^{-1} proved the presence of pure β-TCP. All the bands between 900-1200 cm^{-1} range represented the stretching mode of PO_4^{3-} group. Peaks at 725.2 cm^{-1} and 1209.37 cm^{-1} attributed to the presence of $P_2O_7^{4-}$ group, which was characteristic to calcium pyrophosphate phase. For all the samples similar types of graph was found that proved the presence of pure β-TCP in the working Samples (Figure 4).

Compression test determined the limit of performance of a material to stress. The maximum compressive strength was found for Sample 4 (pH 10) and minimum compressive strength was found for Sample 9 (calcined at 800°C). It is due to the fact that the porosity of TCP decreased with increased of pH as a result compressive strength increased. On the other hand, low calcination temperature resulted in poor density that was higher porosity which caused poor compressive strength (Table 2).

Thermal expansion coefficient value showed very small variation with changing the parameters. These values of thermal expansion co-efficient were listed in Table 3. The minimum value was 1.389×10^{-5} and

Sintered sample	Density(g/cc)	Porosity (%)
Sample S-1, where standard condition was followed	1.88	40.92
Sample S-2, where pH was 6 instead of 8	1.67	40
Sample S-4, where pH was 10 instead of 8	2.31	25.23
Sample S-5, $(NH_4)_2HPO_4$ conc. was 0.6M	2.08	26.57
Sample S-6, $Ca(NO_3)_2.4H_2O$ conc. was 0.8M	1.81	31.59
Sample S-7, (NH4)2HPO4 conc. was 0.2M	1.64	23.5
Sample S-8, $Ca(NO_3)_2.4H_2O$ conc. was 0.4M	1.76	38.89
Sample S-9, where calcination temp. was 800◦C	1.75	33.34
Sample S-10, where calcination temp. was 1000◦C	2.11	40.9

Table 1: Density and porosity of β-TCP samples.

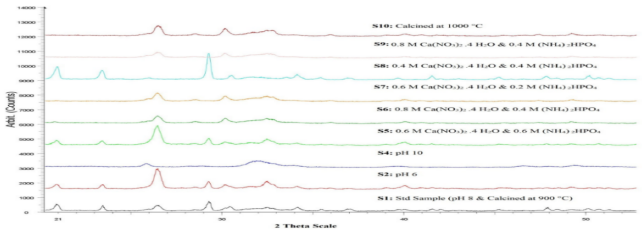

Figure 1: XRD pattern of dried TCP.

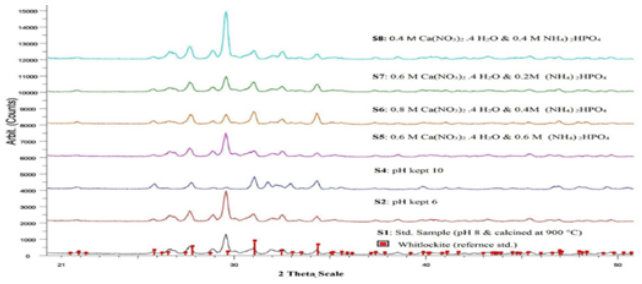

Figure 2: XRD pattern of calcined TCP.

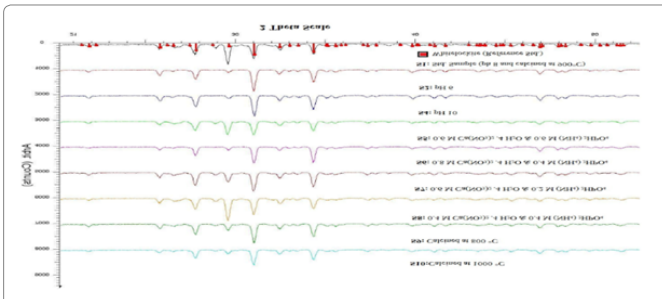

Figure 3: XRD pattern of sintered TCP.

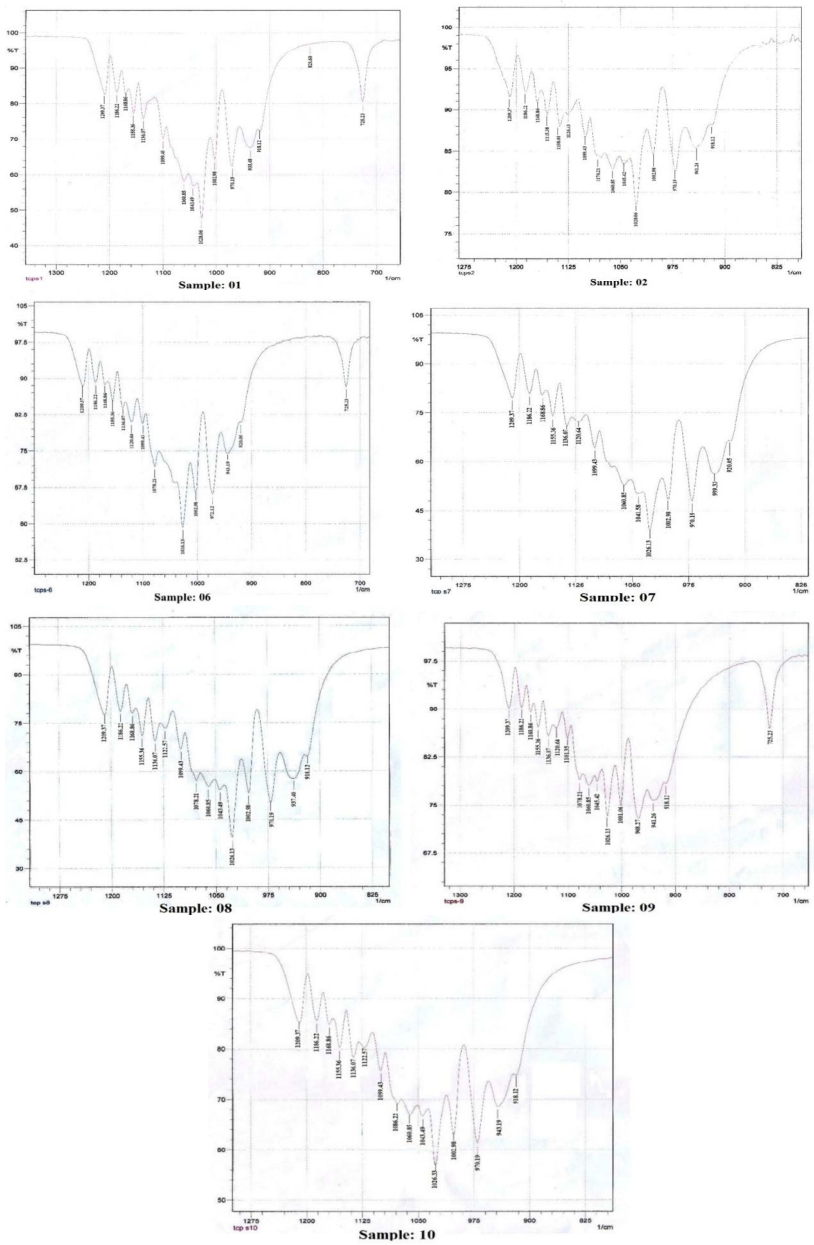

Figure 4: FTIR spectra of different samples of β-TCP powder (Samples 1, 2, 6-10).

Sintered sample	B- TCP (Nmm^{-2})
sample S-1, where standard condition was followed	3.485
Sample S-2, where pH was 6 instead of 8	1.812
Sample S-4, where pH was 10 instead of 8	13.402
Sample S-5, $(NH_4)_2HPO_4$ conc. was 0.6M	4.774
Sample S-6, $Ca(NO_3)_2.4H_2O$ conc. was 0.8M	8.858
Sample S-7, $(NH_4)_2HPO_4$ conc. was 0.2M	5.222
Sample S-8, $Ca(NO_3)_2.4H_2O$ conc. was 0.4M	3.804
Sample S-9, where calcination temp. was 800°C	1.352
Sample S-10, where calcination temp. was 1000 °C	1.614

Table 2: Compressive strength of β-TCP powder.

maximum value was 1.505×10^{-5} .Coefficient of Thermal Expansion of β-TCP powder obtained from TMA (Table 3).

Conclusion

Different samples of β-TCP nano powders were prepared by varying process parameter. All these parameters effects on the properties of the resulting β-TCP nano powders. Density and porosity changes with the pH changes and crystalinity changes with calcination. But, compressive strength found maximum at 900◦C.

Furthermore, widespread use required in situ test and site testing. For long term use both laboratory and full scale loading tests are required and needed to develop a database for assesment.

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Sintered sample	Thermal Expansion Coefficient (K^{-1})
Sample S-1, where standard condition was followed	1.481×10^{-5}
Sample S-2, where pH was 6 instead of 8	1.505×10^{-5}
Sample S-4, where pH was 10 instead of 8	1.389×10^{-5}
Sample S-5, $(NH_4)_2HPO_4$ conc. was 0.6M	1.538×10^{-5}
Sample S-6, $Ca(NO_3)_2.4H_2O$ conc. was 0.8M	1.471×10^{-5}
Sample S-7, (NH4)2HPO4 conc. was 0.2M	1.394×10^{-5}
Sample S-8, $Ca(NO_3)_2.4H_2O$ conc. was 0.4M	1.498×10^{-5}
Sample S-9, where calcination temp. was 800◦C	1.505×10^{-5}
Sample S-10, where calcination temp. was 1000◦C	

Table 3: Coefficient of thermal expansion of β-TCP powder.

different sample analysis by using their laboratory.

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