

Effect of Conventional and Microwave Sintering On Nanostructured Hydroxyapatite (Hap) for Biomedical Application

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ABSTRACT: Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) is hexagonal in nature. Hydroxyapatite is also commonly known as Hap which has hexagonal structure, its chemical formula is $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ and its empirical formula increased by 2 times of the chemical formula. Molecular weight of HAP is 502.31 gm, hardness is 5 and density of HAP is 3.08. HAP is naturally available but only disadvantages is contains the impurities which is not suitable for biomedical application so researchers are interested to produce pure HAP by artificially. So only numerous process are introduced like Molten salt solution, co-precipitation, hydrothermal, solvo thermal, solution combustion, wet chemical method and precipitation process etc. sol-gel process is chosen in the present work. Raw material is used Calcium chloride and Phosphoric acid. The as prepared HAP nano powder is sintered in the muffle furnace and microwave sintering at 800°C and Melting temperature of HAP is 1250 °C. It has vast applications in the field of bio medical such as implants of bone, bone tissue, ear, eye and dental application because of its good biocompatibility. In both the cases particle size were as low as 30 nm to 80 nm have been measured and calculated manually. The complete phase of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ has been identified by X-ray Diffraction pattern some of the peaks matches with JCPDS data 09-0432. More details are discussed in detail about the formation of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ and their morphological behaviour under different parameters.

KEYWORDS: HAp, co-precipitation, probe sonicator

I. INTRODUCTION

Sol gel process is typical process which is used to get complex shapes, pure powder and ultrafine powders of ceramics products. In this ceramics various groups are derived based on their applications and field including bio-ceramics. Hydroxyapatite is also a bio-ceramics material and its chemical formula is $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ [1]. One of the calcium phosphate based bio-ceramics material and it is similar to the inorganic teeth and human bone composition. Artificially prepared HAp is recommended to replace the human bone and teeth because it has good properties like biocompatibility, bioactive, plasticity, adherence, Nano structure, osteoblast and osteoconductivity. Biological tissues present on the bone, teeth, enamel and dentine contain calcium phosphate based mineral in it and poisons ratio of the HAp is $0.27 \approx 0.3$ so it is suitable for the implantation [2, 3].

There are various process are there which can be adopted to get the synthesized HAp such as co-precipitation process, precipitation process, hydrothermal, solvo-thermal, emulsion method, molten salt synthesis, mechano-chemical method, microwave synthesis, electrochemical deposition and sol gel process. In the present studies the research is carried out by preparing in sol gel process to get pure HAp [3, 6]. Structure and composition has high ion exchange capacity and a property initially depends on the synthesizing process. Moreover by precipitation process prepared HAp was examined which has good anion and cation exchange property [4]. High surface area, high volume fractions and nano grains of grain boundaries improves the mineralization, proliferation and adhesion to osteoblast tissue. Sol-gel process has more advantages than other conventional process due to high surface area, high reactivity in low process temperature and homogenous mixture of HAp [5].

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II. MATERIALS AND METHODS

Materials:

Calcium chloride, phosphoric acid, gelation agent, ammonium hydroxide, surfactant, ethanol and distilled water.

Experimental procedure

Initially calcium chloride dissolved in DI and ethanol, phosphoric acid dissolved in DI, surfactant dispersed in ethanol and gelatin also dissolved in water individually. Then calcium chloride solution, surfactant and gelatin are mixed together at a constant speed of the magnetic stirrer was maintained and then phosphoric acid solution added slowly by vigorous stirring of the mixture. Ammonium hydroxide added to maintain the pH 10. stirred mixture filtered for 2 days by using ordinary filter paper in a funnel filtered. Filtered sample is dried for 24 hrs continuously and annealed at a temperature of 800°C for 1 hr in muffle furnace and for half an hour in microwave furnace. Annealed sample is characterized by XRD and SEM.

III. RESULT AND DISCUSSION

Characterization

At room temperature characterization were carried they are XRD and SEM. XRD phase are examined by Rigaku D/max2500 X-ray diffractometer with Cu K α radiation at a scanning with a speed 6(°)/min in the range of 10°–90°. as prepared HAp sample is directly verified phase by getting peaks from XRD analysis. Operating voltage at 40 kV and 200 mA current passed respectively. Morphological and elemental analysis of the powder are investigated by SEM (scanning electron microscope, ZEISS EVO 50) and here elemental analysis together because of EDX (energy dispersive X-ray, Oxford instruments Inca Energy 350) equipped in SEM only and operated at 30kV, diameter of the sample 0.15-0.20 μ m, MTI 24T desktop hydraulic pressing machine under the pressure of 10-20mpa. The powder is coated with a conductive layer of gold for 20s using a Denton Desk V coater and after that nano structures were observed.

XRD Analysis

Fig 1 and 2 are the XRD graphs of the HAp. As prepared sample is annealed in muffle and microwave furnace to investigate difference in phases obtained by the annealing environment may change the formation of the phase due to reaction caused by different condition. HAp formations were identified by comparing the peaks using JCPDS data and ICDD data. JCPDS card no 09-0432, narrower peaks indicate micro crystalline structure, and broader peak indicates nano particle size. Narrower peak also indicates without other extra peaks. HAp sintering normally above 1100°C and which forms phases of HAp but more than 1200°C starts to decompose. In this present studies as prepared powder is sintered at 800°C which also shows some unreacted phase of CaO, Ca(OH)₂, CaCO₃ and some peaks of calcium phosphate group phase are present. by increasing the annealing temperature may increase crystallinity of HAp and pure HAp phase also inversely increase.

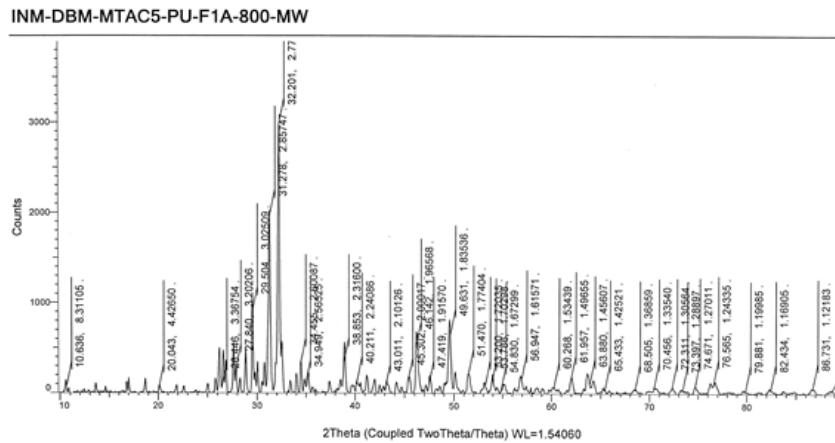


Fig 1: sintered sample in microwave furnace

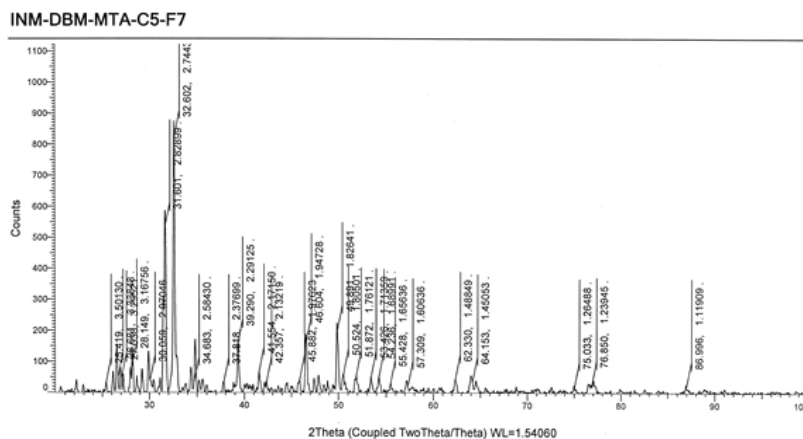


Fig 2: Sample is sintered in the muffle furnace

SEM Analysis

Fig 3 shows a) and b) sample was sintered in the muffle furnace which shows the presence of lumps like structure image a) show rice network structure (SEM mag 5.00kx) and b) shows the lumps like structure due to the role of temperature (SEM mag 30.0 kx), c) and d) sintered in the microwave furnace which shows honey comb structure due to role of temperature while synthesizing of the sample. Image c) shows the clusters of the buds like structure (SEM mag 10.0kx) and d) unclear structure (SEM mag 25.0kx). Annealed sample at 800°C for half an hour and 1 hr which made difference in grain growth of the sample made difference in the structure.

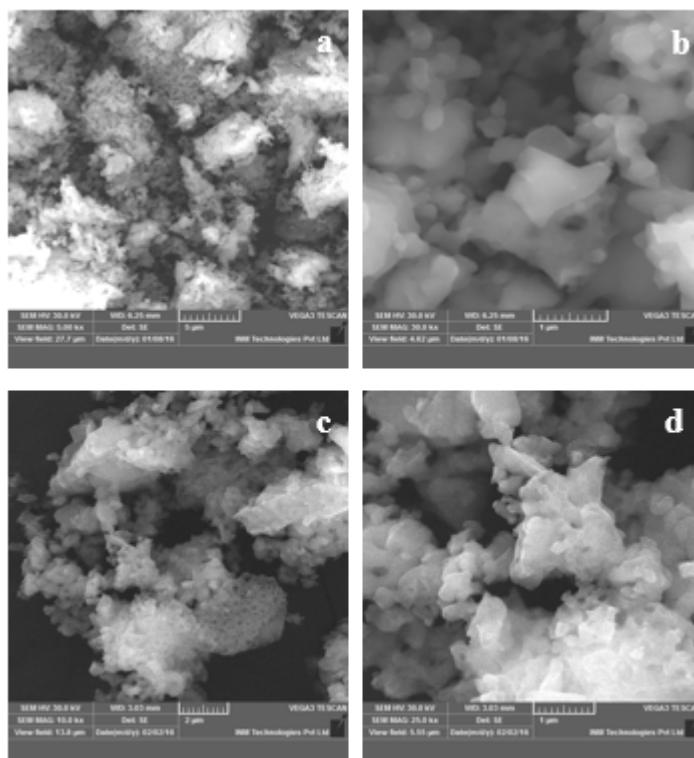


Fig 3: SEM images of microwave and muffle furnace sintered sample

IV. CONCLUSION

Sol-gel process reduced additional step of grinding and calcination temperature. Some phases of the CaO, Ca(OH)₂ and CaCO₃ completely not occurred phase of HAp and some are calcium phosphate phase. Nano particles are obtained. Obtained particle size range is 30-80nm. Nano particles grains are increased by increasing annealing dwelling time in a furnace, controlling grain growth during reaction by adding surfactant and by increasing reaction time in synthesizing.

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