CHAPTER 5

CONCLUSION

5.1 CONCLUSIONS BASED ON THE SYNTHESIS OF HA POWDER

5.1.1 Synthesis of HA Without and With Microwave Irradiation

Nanocrystalline hydroxyapatite was synthesized by the wet chemical reaction between sea shells (CaCO$_3$) which were thermally converted to amorphous calcium oxide (CaO) and then to calcium hydroxide (Ca (OH)$_2$), and reacted with phosphoric acid (H$_3$PO$_4$). HA) was synthesized by the slow addition of phosphoric acid to calcium hydroxide (Ca (OH)$_2$) without microwave irradiation, and in another method microwave irradiation was carried out after the reactants were combined in a similar manner. In both the experiments, the Ca:P ratio of 1.67 was maintained, and the pH of the solution was maintained at 10, to avoid the formation of calcium deficient apatites.

It was observed that, during microwave irradiation, the process of ageing and formation of HA occurred rapidly, whereas in the first method without microwave irradiation, the HA formation occurred gradually. The morphology of the crystallites was rod-shaped in both the methods, but the HA prepared without microwave irradiation had a size of 101nm, and with microwave irradiation, the HA crystallite size was 68nm.
5.1.2 Synthesis of HA by Furnace Drying (FH) and Microwave Irradiation and Drying (MH) by Varying the Concentration of the Reactants

Two sets of experiments were done to synthesize nano hydroxyapatite from sea shell powder (CaO) by the wet chemical synthesis, in which the furnace drying of the obtained HA was done during the first experiment, and microwave irradiation and drying was done in the second. In both the experiments, the Ca: P ratio of 1.67 was maintained for the reagents, and the pH for the reaction was maintained at 10, by the drop-wise addition of ammonia. Both the methods yielded nanocrystalline hydroxyapatite, as evidenced from the XRD/HRSEM analyses.

It was observed that, when the concentration of the reactants decreased, there was random variation in the crystallite sizes ranging from 7nm- 34 nm, whereas, in the second experiment (MH), there was almost no variation in the size of the crystallites with decreasing concentration, and the crystallite sizes were almost the same for all concentrations, and in the range of 34nm -102 nm. Microwave irradiation leads to uniform crystallite sizes, as is evident from this study, at differing concentrations of the reactants, and is a comparatively easy method to synthesize HA.

5.2 CONCLUSIONS BASED ON THERMAL STABILITY STUDIES ON SYNTHESIZED HA

HA prepared from the previous experiment MH1 that had a crystallite size of 51 nm was used to conduct the thermal stability studies, using furnace and microwave heating to compare the decomposition of HA, and study the crystallite sizes.
It was observed that, the microwave heated HA showed no decomposition and yielded crystallite sizes of lesser and more uniform dimensions, than those of furnace heated HA at various heating temperatures. In furnace heating, at increasing temperatures, the size of the crystallites gradually increased from 39nm to 46nm, whereas in microwave heated samples, it was between 36-40nm. Only under a furnace temperature of 1000°C, appreciable amounts of \( \beta \)-TCP were found, and secondary phases such as CaO, \( \alpha \)-TCP and TTCP were never formed up to 1000°C. Microwave heating of HA at all temperatures of 180°C, 200°C, 220°C and 230°C yielded no degradation products and the phase purity of HA was maintained.

### 5.3 CONCLUSIONS BASED ON ELECTROSPUN COATING OF POLYSULFONE (PSU) –HA COMPOSITE ON Ti-6A-4V ALLOY

Nano HA synthesized from MH1, that had a crystallite size of 51nm was used to prepare composite electrospun coatings of PSU, with 1%, 2% and 3% HA on substrates of medical grade of titanium alloy Ti-6Al-4V of 2mm thickness and roughness, Ra~1µm. The morphologies of the nano fibres under different distances between the needle tip and the substrate have also been studied. The controlling parameters of electrospinning: electrical potential, flow rate, concentration of the solution (C) and the distance between the syringe tip and the substrate (d) were varied to study the variation of the coating characteristics.

It was observed that, a coating thickness of around 20 µm was obtained and the different conditions of the concentration of the solution were studied. A polymer concentration of 12% resulted in thinner fibres, but was accompanied by a large number of beads. However, at a higher concentration of 20%, the fibres were uniform with minimum beads. This was because, the
solvent of the solution jet with a higher concentration of 20% is easier to be dried, and hence, the bead sizes decreased. At constant conditions of flow rate (0.1ml/hr), DC electric potential (22kV) and C=20%, a higher value of ‘d’ (15 cm), resulted in thinner fibres having a larger surface area, than at a distance of 10 cm. At C=20% and d=12cm, almost bead free nanofibres were formed and for this condition, the bioactivity studies using a simulated body fluid (SBF) were conducted at different periods of time. The fibres formed were free from any pores, in view of the immiscibility of the water vapour present in air with PSU, enhancing the quality of the fibres.

Thus, nano HA has been synthesized from sea shells by wet chemical precipitation method with and without microwave radiation and by varying the concentrations of the reactants. Thermal stability studies of the synthesized HA have been done and a composite coating of nano HA-PSU has been made by electrospinning under varying conditions on Ti-6Al-4V alloy substrates. It was observed that HA processed by microwave irradiation and drying, yielded smaller crystallite sizes than furnace heated samples. HA processed by microwave irradiation at comparatively lower temperatures was not accompanied by any other secondary phases, since phase pure HA has higher biocompatibility and is more suited for in-vivo applications. The electrospun coating with high porosity and nano particles of HA has been observed to promote the growth of apatite crystals, which would eventually, undergo bioresorption, during which it decomposes by physiological means before the complete growth of natural bone over the implant. The nano fibres of PSU have not undergone corrosion over a prolonged period of 14 days, in view of their high quality, due to the absence of pores in the fibres. Hence, this research opens up the possibility of using electrospun coatings embedded with HA nano particles on titanium implants for clinical applications.