Sonochemical synthesis and characterisation of hydroxyapatite nanopowders

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During the past few decades much effort has been devoted towards various routes to form nanoscale hydroxyapatite (HAp) powders[1] for biomedical applications due to its excellent osteoconductive and osteoinductive properties[2]. In the present work, ultrasound sonochemistry assisted processing was employed to synthesise nanocrystalline HAp powder from an aqueous mixture of nitrate or hydroxide of calcium and ammonium dihydrogen phosphate precursors. The influence of ultrasonic irradiation on the crystallinity, morphology and specific surface area were investigated by XRD, SEM, TEM, DTA-TG and FTIR. The reaction conditions such as pH and temperature were closely controlled to yield monophasic HAp nanoparticles and the level of ultrasonic irradiation was found to play an important role in the progress of the heterogeneous reaction leading to formation of HAp fine powder.

Experimental

Aqueous stock solutions of calcium hydroxide or nitrate were prepared for stoichiometric HAp with Ca/P molar ratio of 1.67 and pH was adjusted to a maximum of 12 by slow addition of ammonium phosphate. A commercial ultrasonic homogeniser was used to irradiate the solution at 40-60°C for a period of 5-120 min. The protocol was repeated for identical samples but without irradiation. The centrifuged suspension was dried and subsequently heated to a maximum sintering temperature of 1200°C.

Results

An equilibrium pH of 10 and a reaction temperature of 60°C were found be critical in the formation of phase pure HAp as confirmed by XRD. An optimum of 80% irradiation power for 5-10 minutes produced nanopowders of an average crystallite size of 25 nm, determined by the Scherrer equation of line broadening of (002) reflection, accompanied by increasing level of crystallinity with time. The FTIR spectra corresponded well with the characteristic O-P-O vibrational mode PO_4^{3-}. SEM and TEM studies showed the crystallite sizes of 10 nm diameter and 30 nm in length. The degree of agglomeration varied with the duration of irradiation.

Conclusions

The phase pure stoichiometric HAp can be synthesised at ambient pressure by sonochemical methodology. The results show that the sonochemistry significantly increased the synthesis efficiency when compared to other conventional precipitation routes. The technique is reliable in terms of monitoring and controlling the progress of the reaction kinetics with respect to pH, temperature and duration of irradiation treatment.

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