Hydrothermal synthesis of biocompatible $CaTiO_3$ particles

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Extended Abstract

Of late, synthetic grafts have presented themselves as worthy alternative source for continuous bone healing to both the conventional autogenous and allografts. This is due to their reasonable production costs (while harvesting cost is very steep in the conventional methods) and the avoidance of the pathogenic transmission risk as posed in allografts. One of the essential requirements for synthetic grafts is its biocompatibility and this can be achieved through hydrothermal process. Furthermore, this technique of synthesis is also capable of producing a narrow distribution of particle sizes besides its ease in implementation and thus, the reason for its employment in this study.

In the current work, orthorhombic submicro- and nanoparticles of $CaTiO_3$ were synthesised via a simple hydrothermal process using TiO_2 (p25) as the precursor in alkaline media containing excess NaOH and $CaCl_2$. The as-prepared powdery materials were washed with distilled water until the pH value became neutral and dried in oven at 50°C for 24 hours. The dried samples were characterised by X-ray diffraction analysis (XRD) and Scanning Electron Microscopy (SEM).

NaOH in excess provided a basic environment (pH > 12) for the dissolution of TiO_2 and its subsequent conversion to $CaTiO_3$. The formed Ti-OH complexes reacted with the Ca^{2+} ions in the solution to form the perovsite, for example, at 120°C for 24 hours. The chemical reaction involved is shown below.

$2NaOH + CaCl_2 + TiO_2 \rightarrow CaTiO_3$ (or $CaO{\cdot}TiO_2$) $+ 2NaCl + H_2O$

The most interesting outcome of this synthesis was that orthorhombic calcium titanate particles with three different mutual edges were generated (shown in Figures 1a and 1b). The crystalline lattice of the produced perovskite was also orthorhombic which was confirmed by the X-ray diffraction angles at 23.1°, 33°, 39°, 41°, 47.3° and 59° (shown in Figure 2).

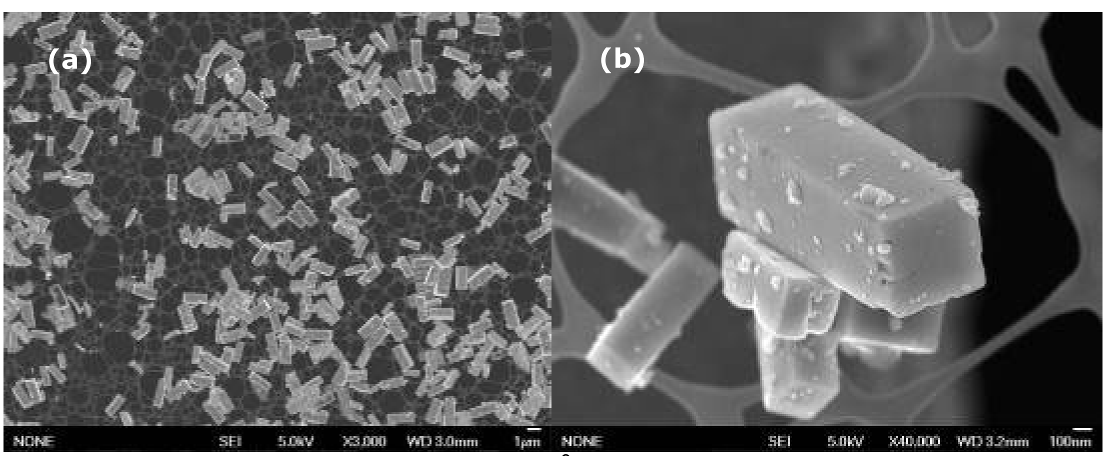

Figure 1 (a) Uniform $CaTiO_3$ generated at 120°C for 24 hours (b) Orthorhombic particles with three different mutual edges

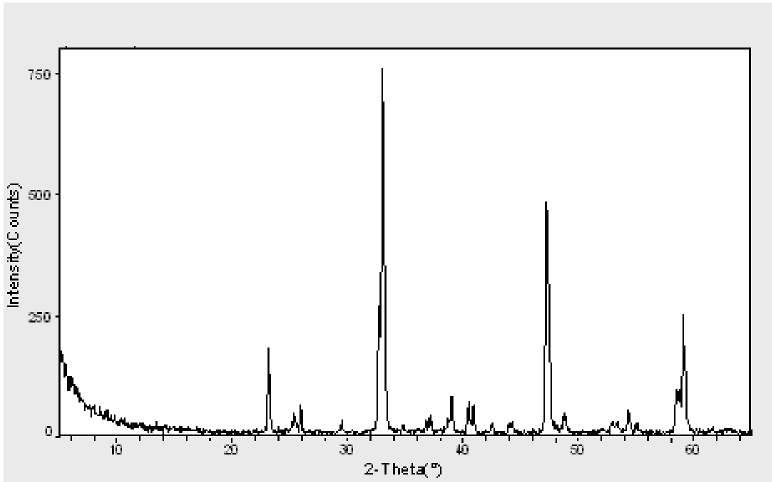

Figure 2. XRD pattern of the perovskite produced at 120°C for 24 hours using P25 TiO_2 and excess NaOH and $CaCl_2$

The size control of the perovskite particles could be achieved by adjusting the amount of NaOH added. For example, when the number of moles of sodium hydroxide was less than 20 mmol than that of calcium chloride in the synthesis, orthorhombic perovskite particles with shorter length were generated as shown in Figure 3. However, the reaction temperature was raised to 200°C for the formation of shorter perovskite. Thus, the formation of shorter orthorhombic particles was more preferable at less basic solution.

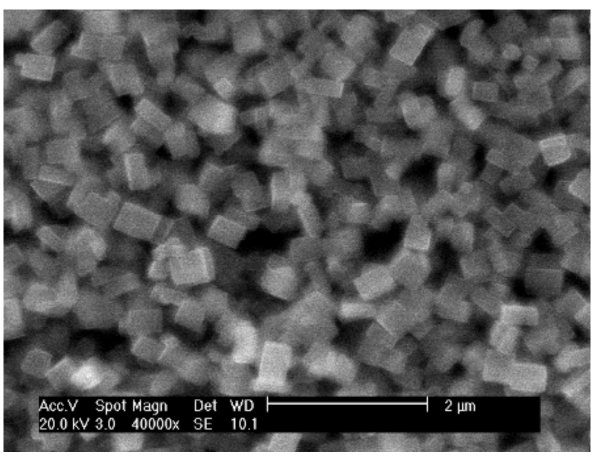

Figure 3. The length of the orthorhombic particles reduced at less amount of NaOH.

When NaOH was not added to the mixture of $CaCl_2$ and TiO_2, only anatase was collected after the process. No trace of perovskite peaks is observed in Figure 4.

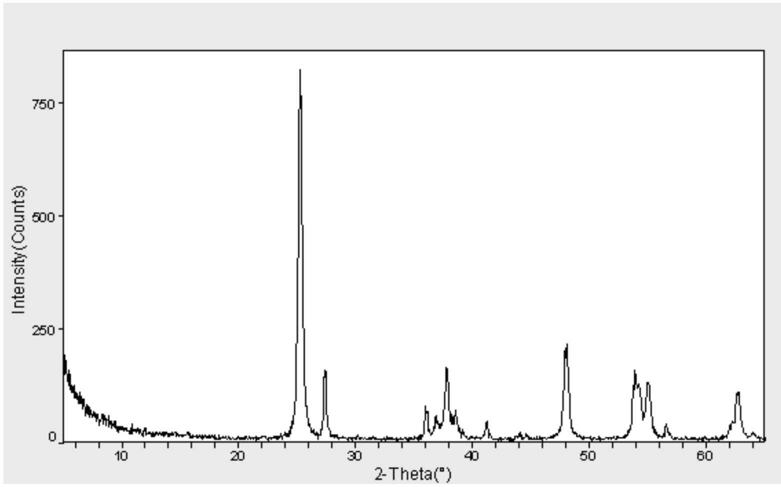

Figure 4. XRD pattern for anatase collected after for 24 hours hydrothermal process at 120°C using only P25 TiO_2 and $CaCl_2$

In conclusion, orthorhombic $CaTiO_3$ particles with narrow size distribution were successfully generated through hydrothermal method. The essential requirement for the formation of $CaTiO_3$ was the presence of NaOH. The amount of this alkaline media also affected the size of the perovskite generated. The formation of longer orthorhombic perovskite was more preferable at pH value of 12. The biocompatibility and osteoconductivity of these novel submicro- and nano-perovskite will be tested in future research[1].

[1] Ohtsu N, Sato K, Saito K, Hanawa T, Asami K, Evaluation of Degradability of $CaTiO_3$ Thin Films in Simulated Body Fluids, Mater Trans 45 (2004), pp. 1778-1781