

Formation of Nanostructured Fluorapatite via Microwave Assisted Solution Combustion Synthesis

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Statement of Purpose: This study presents a rapid microwave assisted solution combustion synthesis (MASCS) method for production of fluorapatite (FA) nanoparticles. FA has applications in dentistry and orthopedics. However, the conventional synthesis processes are tedious [1]. MASCS is able to synthesize unique structures including nanotubes, hexagonal crystals, nanowhiskers, and plate agglomerates by controlling the solution compositions and reaction times. We report here for the first time, the synthesis of FA nanotubes via the MASCS technique. The as-synthesized FA nanotubes presented “Y” shaped inner channels along the crystal axis. The channel formation is caused by the crystal growth and the removal of water soluble salts during process. The as-synthesized FA nanotubes showed cytocompatibility as the cells cultured with extracts from higher concentration of FA demonstrated greater growth rate. The as-synthesized nanotubes are believed to have potentials in gene/drug delivery.

Methods: The FA nanoparticles were synthesized using a microwave technique. The reaction solution was prepared by adding NaNO_3 , NaF or CaF_2 , $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, and KH_2PO_4 to 10 mL de-ionized water in 30 mL beaker with 200 rpm stirring. After 10 minutes of stirring, the mixture was covered and placed in a household microwave. The assembly was then heated at 100% power for 3, 5, or 7 minutes separately. Resulting substances were left to air-dry for 15 minutes. Air-dried products were magnetically stirred at 400 rpm in 500 mL of de-ionized water until the entire precipitate was dissolved. Finally, the solution was washed with approximately 2 L of de-ionized water and filtered. The filtrate was then placed in an 80°C oven overnight for further characterization and evaluation. SEM, TEM, XRD, and FTIR methods were used for physical characterizations. Finally, MC3T3 cells were used for *in vitro* studies.

Results: The SEM and TEM images of FA nanoparticles are shown in Figure 1. Figures 1(a) and (b) show the experimental conditions resulted in the formation of hexagonal FA nanotubes. Figures 1(e) and (f) show the physiological stability of hexagonal geometry and inner channels of FAs in SBF after 7 days. Apatite growth on the surface of bioactive materials is attributed to the nucleation sites on surface such as active Ca^{2+} sites of calcium phosphates [2]. FA is believed to require much longer time to create enough active Ca^{2+} sites for apatite deposition as compared to HA, since FA is more resistant to chemical attack. As a result, apatite formation is not significant on FA. Figures (c) and (d) show the presence of “Y” shaped inner channels in the FA crystal structure which are applicable in gene delivery. XRD and FTIR patterns of FA are shown in Figures 2 (a) and (b), and are in agreement with previously reported FA spectrum[3]. Figure 3 summarizes the kinetics of FA nanotube formation. Figure 4 illustrates higher growth rate of

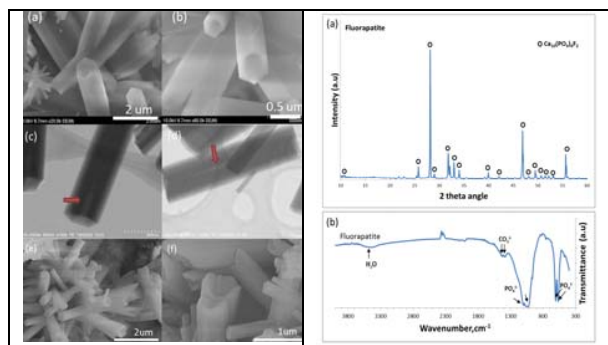


Figure 1. SEM images of as-synthesized FA nanoparticles: (a) and (b), after 7 days in SBF: (e) and (f), and TEM images of FA nanotubes: (c) and (d)

Figure 2. XRD (a) and FTIR (b) results of as-synthesized FA

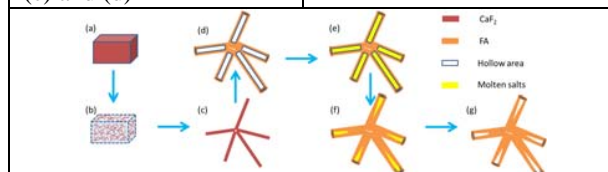


Figure 3. Kinetics of FA nanotube formation from



Figure 4. Effects of different concentrations of FA extracts on preosteoblast cells after culture for 1 and 7 days

preosteoblasts cultured in higher FA extract concentrations which shows that released F^- ions from FA can promote proliferation of preosteoblast cells.

Conclusions: Unique structures of FA including nanotubes, hexagonal crystals, nanowhiskers, and plate-like crystals can be synthesized by changing the processing parameters of the MASCS method (only nanotubes are presented here). The crystal features are controlled by reaction solution compositions and operation parameters. The as-synthesized FA nanotubes were shown to be stable in physiological conditions and were cytocompatible. In *in vitro* study, the cell proliferation rate was parallel to increasing concentration of FA extract. FA nanoparticles have potentials for numerous dental and biomedical applications.

References:

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