A NOVEL MICROWAVE ASSISTED PROCESS TO SYNTHESIZE PHOSPHATE NANOWHISKERS

S. Jalota, A.C. Tas and S.B. Bhaduri*
School of Materials Science and Engineering
Clemson University, Clemson, SC 29634

ABSTRACT

Calcium phosphate (single-phase HA, single-phase TCP, and biphasic HA-TCP) nanowhiskers were synthesized using a novel microwave-assisted "combustion synthesis (auto ignition) /molten salt synthesis" hybrid route. This work is an example of our "Synergistic Processing" philosophy combining the aforementioned three technologies while utilizing their useful aspects. An important part of this work is the assistance of microwave heating. If we were to perform the combustion of our initial solutions (for the biphasic and HA samples) under conventional heating, temperature distribution of the solution would be inhomogeneous. Since the synthesis of nanoparticles from their precursors in solutions consist of nucleation and particle growth, it is important that the particle growth is terminated. Those processes partly rely on the local temperature distribution within the solution. Any inhomogeneity in the temperature distribution would then cause broad distributions in particle size. Microwave irradiation should lead to homogeneous, i.e., molecular-level heating of the polar solution system, achieving automatic control of the above two processes required for the preparation of nanowhiskers or nanoparticles. The molten $NaNO_3$ provides a transient coating, which can be washed away, thus resulting in de-aggolmerated nanowhiskers.

Aqueous solutions containing $NaNO_3$, $Ca(NO_3)_2{\cdot}4H_2O$ and KH_2PO_4 (with or without urea) were irradiated in a household MW oven for 5 minutes at 600 watts of power. The as-synthesized precursors were then simply stirred in water at RT for 1 hour to obtain the nanowhiskers of the desired calcium phosphate bioceramics.

The nanowhiskers were characterized using X-ray diffraction, FTIR, and scanning electron microscopy. After 5 minutes of microwave heating but without wash in water, powder XRD data only showed the presence of $NaNO_3$ as the crystalline phase, however, as expected, FTIR data of the unwashed precursors revealed the presence of characteristic phosphate IR bands. But after washing, (1) monodispersed nanowhiskers of HA were obtained which were about one micron-long and 100 nm-thick; (2) biphasic samples contained around 60% TCP and 40% HA in which the volume percentage of the HA phase can easily be adjusted by varying the urea content in the starting solutions; (3) single phase TCP nanowhiskers were obtained by adjusting the cooling rate after heating in the microwave.

This study will open up new avenues for industrial (biomedical) applications and academic research on nano-scale hydroxyapatite and other calcium phosphates of biological relevance.