Chemical and X ray Diffraction Analyses of Calcium Phosphate Used for Discrete Crystalline Deposition

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Statement of Purpose: NanoTite™ (Implant Innovations, Inc., Palm Beach Gardens, FL, USA) is an implant surface modification featuring discrete crystalline depositions (DCD™) of nanometer-scale calcium phosphate (CaP) particles processed by sol gel application over the microtopography of dual acid-etched titanium alloy (Osseotite®, Implant Innovations, Inc.). The two-part objective of this study is (1) to confirm that the CaP particles in the colloidal solution used for the DCD process have a predetermined crystallinity and chemistry that are not altered during the application process to the titanium surface and (2) to qualitatively assess the size and shape of the CaP particles deposited on the implant surface using Field Emission Scanning Electron Microscopy (FE-SEM).

Methods: The X-ray Diffraction (XRD) analysis was performed using a Scintag XDS2000 diffractometer (Scintag, Inc., CA, USA) on the CaP powder used as a raw material in preparing the colloidal solution. The size of the CaP nanometer-scale particles in the colloidal solution ranges from 20nm to 100nm. The Certificate of Analysis from the manufacturer of the CaP powder states that the Ca/P ratio is 1.6. XRD was also carried out on a CaP powder sample which was obtained by drying the colloidal solution used in the DCD process. The crystallinity of the samples were identified through comparison with the built-in JCPDS powder diffraction database. An FE-SEM, model JEOL JSM-6700F (JEOL USA, Inc. Peabody, MA) was used to obtain high resolution imaging on the NanoTite implant surface in order to visualize the nanometer-scale CaP crystals.

Results/Discussion: The XRD analyses for both the source material sample used for preparing the colloidal solution and for the dried CaP sample that had been used in the DCD process show 100% crystallinity within the detection limits of the instrumentation and no amorphous content was detected. This result indicates that there was no change in the crystallinity due to the DCD process and therefore the chemistry of the CaP particles was not altered during the sol gel application to the titanium oxide surface layer. Figure 1 shows the XRD pattern for the dried CaP powder sample to be purely crystalline in nature.

The FE-SEMs of the implant surface show discrete depositions of CaP crystals the size, shape and structure of the CaP crystals visually appear to be in the size range from 20nm to 100nm which is in agreement with the Certificate of Analysis from the manufacturer of raw CaP powder. Figure 2 shows a representative FE SEM image (30,000X) of the implant surface.

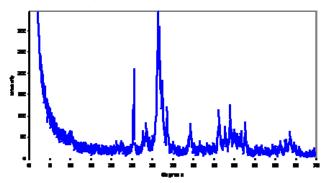


Figure 1. XRD Pattern For Dried CaP Powder Sample Which Shows It to be Purely Crystalline

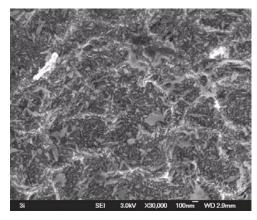


Figure 2. High Magnification FE SEM Image of Implant Surface Showing Size and Shape of CaP Crystals

Conclusions: The crystallinity of the CaP particles in the raw material and the dried sample obtained from the colloidal solution used in the discrete crystalline deposition process was confirmed by the x-ray diffraction analysis to be purely crystalline in nature. This verifies that there was no change to the CaP crystallinity before and after the deposition process. The qualitative analyses of the implant surface, visualized with high resolution FE-SEM imaging showed that the CaP crystal size remained unchanged. By incorporating highly crystalline CaP crystals into a nanoscale textured surface, the biological benefits of hydroxyapatite and the osseogenic potential of surfaces with nanotopographical features can be realized *in vivo*.