Synthesis of Hydroxyapatite Bone Mineral from Calcium and Phosphate Precursors via Sol-Gel Chemistry for Conformal Orthopedic Implant Coatings: Phase II

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INTRODUCTION

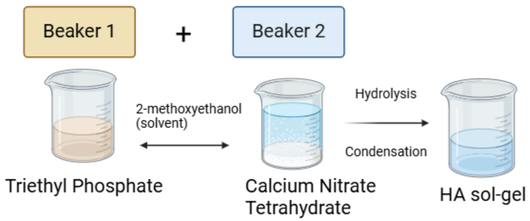
Given developments in advanced biomaterials, there is a growing interest in replacing metal with non-metal fixation devices. This study is a continuation of Phase I whereby the natural ceramic, hydroxyapatite (HA) was synthesized by sol-gel chemistry technology using calcium nitrate tetrahydrate and triethyl phosphate precursors. The goal of Phase II is to determine if it is possible to develop mechanically robust, conformal sol gel-derived HA coatings from heat treatment processes. Sol gel-derived HA coatings will be first made with the respective HA precursors and subsequently processed using external (muffle furnace) processes. The sol-gel derived coatings will then be characterized Fourier Transform Infrared Spectroscopy from which an estimation of coating mechanical quality will be determined.

The sol-gel process: a solution is formed from HA ceramic precursors that subsequently undergoes hydrolysis and condensation reactions that results in the formation of a 3D gel. The wet gel is dried to form a xerogel that can be transformed into ceramic films or coatings.

Hydroxyapatite: $Ca_{10}(PO_4)_6(OH)_2$

METHODS

1. HA Sol-gel Synthesis



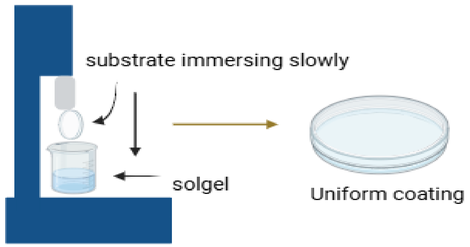
2. Sol-gel Characterization:

- Contact angle to determine wettability
- FTIR for identification of chemical bonds

$$\gamma^{SV} = \gamma^{SL} + \gamma^{LV} cos\theta$$

Fig 2. Young Dupre Equation

3. Dip Coating



4. Firing & Characterization

FTIR for identification of chemical bonds in fired coatings

DISCUSSION

Synthesizing a higher concentration of hydroxyapatite sol-gel and conducting dip coating soon after the sol-gel was synthesized produces greater quality of coatings that are uniform. A rate of 500 um/s became the determined withdrawal speed due to the proper coating and wettability it gave. The contact angle dictates a proper wettability due to it being less than 90˚. The FTIR of the sol-gel shows the hydroxyapatite precursors. The FTIR of the fired films shows the formation of hydroxyapatite forming after 5 hours under 800˚C with the visible phosphate group. However, after 2 hours there seems to be an incomplete reaction with HPO_4^{2-}.

NEXT STEPS

Doing XRD on the fired films will provide more information if hydroxyapatite was produced or not. Additionally, conducting more of a detailed process with dip coating can lead to a better withdrawal rate that would produce more of a uniform coating

RESULTS

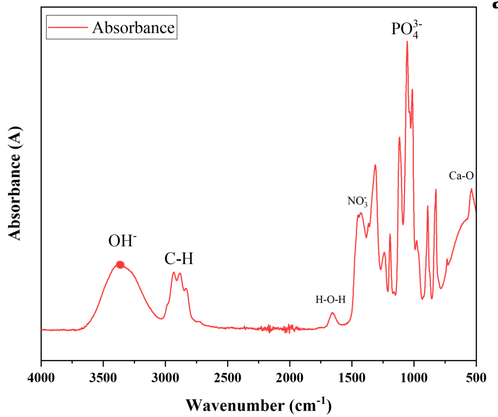


Fig 3. FTIR of 0.1 M sol-gel displaying functional groups

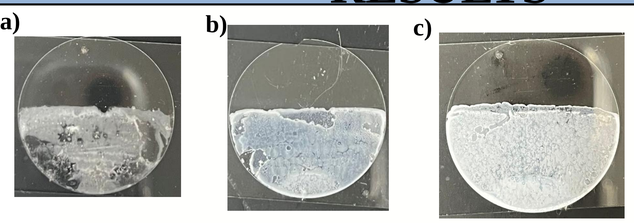
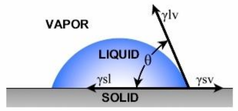


Fig 4. Dip coatings dried at 350˚C for 3 min. a) 40 um/s, b) 100 um/s, c) 500 um/s



γ^{SV} = solid surface free energy
γ^{SL} = solid/liquid interfacial free energy
γ^{LV} = liquid surface free energy

Average Contact Angle: 14.7˚ Average pH: 5

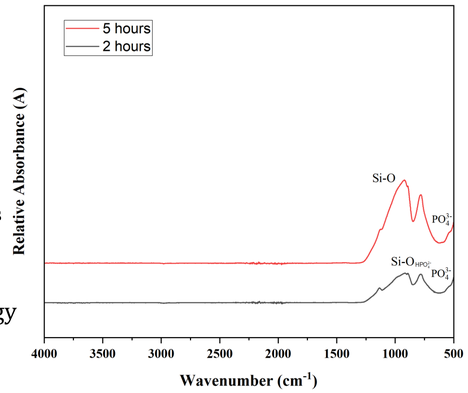


Fig 5. FTIR of fired 500 um/s dip coatings at 800˚C displaying functional groups

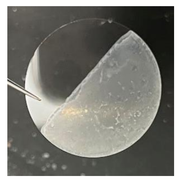


Fig 6. Fired 500 um/s dip coating after 2 hours

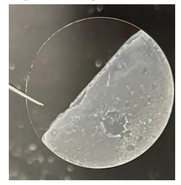


Fig 7. Fired 500 um/s dip coating after 5 hours

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FURI

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