APPENDIX E-1

Preparation of CaP Powders

Stoichiometric calcium phosphate (CaP) was prepared by means of a precipitation reaction between 0.5 moles of calcium hydroxide, $Ca(OH)_2$, and 0.3 moles of orthophosphoric acid, H_3PO_4. Stoichiometric calcium phosphate was defined as synthetic calcium phosphate that had a Ca/P molar ratio approaching 1.67 and that, after sintering and calcining at temperatures between 800° and 1300°C, did not decompose to form secondary phases such as tricalcium phosphate or CaO. The precipitation reaction was carried out at room temperature, with the pH maintained at 10.5 by the addition of ammonium hydroxide solution. After complete mixing of the reactants, the suspension was aged overnight. The aged precipitate was filtered in air, dried at an elevated temperature, and then milled to form a powder.

Preparation of SiCaP Powders

The silicate-substituted calcium phosphate (SiCaP-30) was prepared in a manner that was analogous to the preparation of the stoichiometric calcium phosphate by the precipitation reaction between $Ca(OH)_2$ and H_3PO_4. The appropriate quantities of reactants to prepare the SiCaP-30 were calculated from the quantities used to prepare the stoichiometric calcium phosphate. The silicate-substituted calcium phosphate was prepared by an acid-base neutralization reaction. The quantities of reactants were calculated by assuming that silicon (or silicate) would substitute for phosphorus (or phosphate). Therefore, the number of moles of H_3PO_4 used in the preparation of the stoichiometric calcium phosphate was the same as the number of moles of (H_3PO_4 + Si acetate) used in the preparation of the SiCaP-30; the number of moles of $Ca(OH)_2$ was kept constant. The precipitation reaction was carried out at room temperature, with the pH maintained at 10.5 by the addition of ammonium hydroxide solution. After complete mixing of the reactants, the suspension was aged overnight. The aged precipitate was filtered in air, dried at an elevated temperature, and then milled to form a powder.

Preparation of Sintered Ceramics

Porous ceramic foams were manufactured from the milled precipitate with use of a foaming method. Briefly, polyvinyl alcohol was dissolved in double-distilled water at 40°C under conditions of continuous stirring. This binder solution was then combined with calcium phosphate powder with a particle diameter of 3.3 µm and a surface area of 13.4 m^2 per gram. The resulting slip was then placed in a mill pot with milling media (18-mm-diameter balls) and milled at a speed of 120 rpm for three hours. The foamed slip that was produced was then cast into a mold. The casting was dried in air at a temperature of 40° to 50°C for up to forty-eight hours. The dried green casting underwent a stepped heat treatment in a ventilated furnace for binder burn-out and densification by sintering. The casting was heated at a rate of 2.5°C/min to 500°C and held at this temperature for four hours. Once the binder had been burned out, the casting was heated at a rate of 2.5°C/min to a temperature of 1200° to 1300°C with no air flow and held for two hours. The sintered porous monolith was removed from the sintering furnace and mechanically crushed between two ceramic coated paddles. The material was subsequently passed through sieves for fractionation into 1 to 2 and 2 to 5-mm granules.