Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Biomimetic fibronectin/mineral and osteogenic growth peptide/mineral composites synthesized on calcium phosphate thin film

C. Chen,[a,b] Z.Y. Qiu,[a] S.M. Zhang*[a] and I-S. Lee*[b]

[a] Advanced Biomaterials and Tissue Engineering Center, Huazhong University of Science and Technology, Wuhan, China.

[b] Atomic-Scale Surface Science Research Center, Yonsei University, Seoul, Republic of Korea.

* E-mail: smzhang@mail.hust.edu.cn; inseop@yonsei.ac.kr.

General

The osteogenic growth peptide (OGP) is a 14-amino acid growth peptide (Ala-Leu-Lys-Arg-Gln-Gly-Arg-Thr-Leu-Tyr-Gly-Phe-Gly-Gly) initially isolated from the osteogenic phase of postablation regenerating bone marrow. The synthetic linear peptide OGP was synthesized with the Fmoc system according to the standard solid-phase peptide synthesis methodology.

For each experiment, commercially pure titanium (grade IV, Supra Alloys Inc., Camarillo, CA, USA) was used as a substrate after machining into disk of 10 mm in diameter and 2 mm in thickness. After sonication in acetone and then deionized water the samples were dried in a nitrogen stream prior to the deposition process. Evaporates of calcium phosphate were prepared by sintering mixtures of powdered CaO (Cerac, USA) and hydroxyapatite (Alfa, USA) at 1200 °C for 2 h in air. Thin calcium phosphate film deposition was conducted by using an IBAD system. Using a cryopump (OB-10, Helix Technology, Mansfield, MA, USA), the chamber was evacuated to a pressure of 10^{-7} Torr. Subsequently, Ar gas (P = 10^{-4} Torr) was introduced to the chamber. An electron beam (Telemark, Fremont, CA, USA) at 8.5 kV and about 0.1 A was evaporating the powder mixtures, and the end-hall type ion gun (Mark II, Commonwealth Scientific, Alexandria, VA, USA) was simultaneously applied to the implant substrate surface to assist the deposition. The voltage was fixed at 130 V and the current level was gradually increased up to 1.0 A. The substrate was rotated at 8 rpm during deposition in order to improve uniformity of coating layer. Heat treatment after deposition was performed at 350 °C with a heating rate of 5 °C min^{-1}. The heat treatment was held for 1 h, and then the samples were cooled to room temperature in a furnace. For the cross section observation by SEM, the calcium phosphate deposited silicon wafers were prepared in the same way as titanium. It was considered that the calcium phosphate films deposited on the silicon substrates were identical with that on the titanium substrates.

Equipment and techniques

The precipitated layers were analyzed using a JEOL 6500F SEM with an INCA Energy 300 Microanalysis EDS system. ATR-FTIR spectra were collected using a VERTEX 70 ATR-FTIR.

TF-XRD data were collected on a Bruker D8-FOCUS X-Ray diffractometer using Cu-Kα radiation (λ = 1.540598 Å) at 23 °C over the 2θ range 1-80° with a step size of 0.01° and a step duration 5 s. Rietveld refinement analysis was conducted by means of software package GSAS in conjunction with EXPGUI. A shifted Chebyshev function was used to refine the background. To visualize the cross sections of OGP spatial distributions, a ratio of 7:1 of OGP: fluorescein isothiocyanate (FITC)-OGP was used. The peptides were incorporated as previously described. The treated samples were viewed using confocal microscopy (MRC-1024 MP, BioRad) at an excitation wavelength of 488 nm. Using the BioRad Radiance 2000 LaserSharp imaging program, a series of images was taken in 1 μm intervals using 60 × magnification oil immersion. Side depth profiles through the thickness of the OGP/mineral layer were obtained by stacking the series of images.

Supplementary figure and table captions (listed)

Figure S1 Amount of FN or OGP incorporated on each sample after as-deposited samples immersed in DPBSF or DPBSO solutions for determined intervals.

Figure S2 Rietveld refinement for TF-XRD patterns: (a) FN/mineral sample, and (b) OGP/mineral samples. The crosses (+) mark the observed pattern, and the solid lines (-) are the calculated diffraction pattern. The lower trace is the difference pattern between the observed and calculated patterns.

Table S1 Unit cell parameters, disagreement factors of apatite for mineral with and without biomolecules from TF-XRD Rietveld refinement.

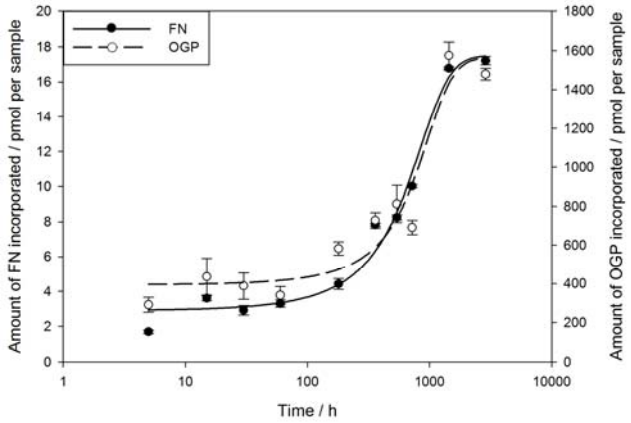

Figure S1 Amount of FN or OGP incorporated on each sample after as-deposited samples immersed in DPBSF or DPBSO solutions for determined intervals.

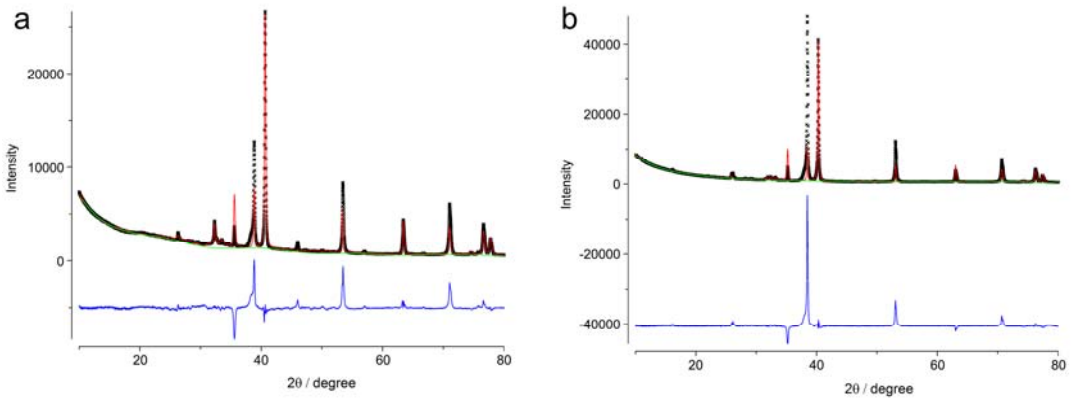

Figure S2 Rietveld refinement for TF-XRD patterns: (a) FN/mineral sample, and (b) OGP/mineral samples. The crosses (+) mark the observed pattern, and the solid lines (-) are the calculated diffraction pattern. The lower trace is the difference pattern between the observed and calculated patterns.

Table S1 Unit cell parameters, disagreement factors of apatite for mineral with and without biomolecules from TF-XRD Rietveld refinement.

Sample	*a* (Å)	*c* (Å)	R_{wp}	R_p
Mineral	9.378	6.870	0.2180	0.1061
FN/mineral	9.405	6.890	0.1536	0.0802
OGP/mineral	9.369	6.882	0.2190	0.1388