



Bioactivity and corrosion properties of gelatin-containing and strontium-doped calcium phosphate composite coating



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ARTICLE INFO

Article history:

Received 8 April 2013

Received in revised form 1 June 2013

Accepted 2 June 2013

Available online 13 June 2013

Keywords:

Electrodeposition

Bioceramics

Strontium

Gelatin

Corrosion

Bioactivity

ABSTRACT

To improve coating corrosion resistance and bioactivity, strontium (Sr) and gelatin (GLT) were simultaneously incorporated in calcium phosphate (Ca–P) to form Sr–Ca–P/GLT composite coating on titanium (Ti) by electrodeposition. The surface morphology, chemical composition, phase identification, bond strength, corrosion resistance, and cytocompatibility of the films were studied. Results revealed that the Sr–Ca–P/GLT layer was rough and inhomogeneous, with floral-like crystals or flake agglomerate morphology. The Sr–Ca–P crystals were Sr-doped apatite (hydroxyapatite and brushite), and Sr²⁺ ions and GLT were homogeneously distributed in the Ca–P coating. The thickness of the composite coating was almost 10 μm without delamination and/or cracking at the interface. The bond strength of the composite coating was 5.6 ± 1.8 MPa. The Sr–Ca–P/GLT coated Ti had lower corrosion rates than bare Ti, suggesting a protective character of the composite coating. Osteoblast cellular tests demonstrated that the Sr–Ca–P/GLT composite coating better enhanced the in vitro biocompatibility of Ti than Ca–P coating.

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1. Introduction

Bioactive coatings such as various ceramic calcium phosphate (Ca–P) compounds are highly significant for modifying the surface of metal implants. These coatings are widely used in the surface modification of titanium (Ti) and its alloys to stimulate the direct attachment of the surrounding hard tissue and to restrain the release of corrosion products into the human body [1–10]. However, the clinical applications of Ca–P in medical implant fields are restricted by its comparatively slow rate of biological interactions [11]. Thus, the preparation of Ca–P enriched with biologically active ions and organic molecules is gaining increased interest. Numerous studies have shown that strontium (Sr)-doped Ca–P (Sr–Ca–P) shows super-osteoinductive activity and higher solubility than Ca–P [1,6,9]. Moreover, Ca–P/gelatin (GLT) composites receive considerable attention as promising biomaterials because of their similarity in composition to native bone and good biocompatibility [12,13]. However, few studies have been conducted on the simultaneous incorporation of Sr ion and GLT into Ca–P coating. Thus, the corrosion of Ti can be reduced and the bioactivity can be improved by preparing Sr–Ca–P/GLT composite coating.

Electrodeposition (ED) is attracting considerable attention as a superior method of preparing inorganic composite coatings and organic protective coatings [14,15]. In this study, we simultaneously incorporated Sr²⁺ ions and GLT into electrodeposited Ca–P coatings for enhanced corrosion resistance and cell–biomaterial interactions. The structural, chemical, mechanical, and corrosion behaviors of the new composite coating were studied. The in vitro cellular responses of the Sr–Ca–P/GLT composite coating were also assessed using mouse osteoblasts (MC3T3-E1).

2. Materials and methods

2.1. Preparation of Sr–Ca–P/GLT composite coating

ED was conducted using an LK2005A (China) electrochemical workstation. An electrolytic deposition apparatus with saturated calomel, Ti, and platinum electrodes were used as the reference, working, and counter electrodes, respectively. The electrolyte was prepared with a theoretical (Ca + Sr)/P ratio of 1.67 for apatite (Ca:Sr molar ratio = 9:1), and strontium-substituted Ca–P with 10 mol% Ca²⁺ replaced by Sr²⁺ was also designed. The Sr–Ca–P/GLT composite coating was conducted in an electrolyte consisting of 1 g/L GLT (Sigma), 4.2 × 10^{−3} M Sr(NO₃)₂, 3.78 × 10^{−2} M Ca(NO₃)₂·4H₂O, and 2.5 × 10^{−2} M NH₄H₂PO₄ under the following parameters: pH 4.2 ± 0.5, 1 × 10^{−3} A/cm² current density, 10–20 min, and 50 ± 0.5 °C. All aforementioned reagents were analytical grade.

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2.2. Composite coating characterization

A JEOL JSM-6490LV scanning electron microscope (SEM) was employed to detect the surface morphology. Quantitative element analysis was identified by energy-dispersive X-ray analysis (EDS) (GENESIS 2000 XMS). A BEDE D1 SYSTEM X-ray diffraction (XRD) apparatus was employed for phase identification. The apparatus was operated with a $\text{CuK}\alpha$ radiation at 35 kV and 35 mA. The structural and molecular composition analysis was conducted by Fourier transform infrared spectroscopy (FTIR NICOLET NEXUS 670).

2.3. Bond strength test

The bond strength (BS) between the as-electrodeposited Sr–Ca–P/GLT coating and the Ti substrate was evaluated using an electronic universal testing machine, following the American Society for Testing Materials international standard F1044 [2]. Five replicates were conducted. The counter Ti substrate was attached to the surface of the Sr–Ca–P/GLT coating using epoxy. After heating in an oven at 120 °C for 2 h, the samples were tested for tensile strength at a constant speed of 1 mm/min until fracture. The adhesive BS was calculated as failure load/sample area (0.785 cm²).

2.4. Potentiodynamic polarization test

The simulated body fluid (SBF) was prepared in the light of Kokubo's recipe, as listed in Table 1 [16]. The corrosion behavior of the Sr–Ca–P/GLT composite coating in SBF solution at 37 ± 0.5 °C was studied by way of potentiodynamic polarization test (Electrochemical Workstation, LK2005A, China). The platinum sheet was designed as auxiliary electrode, a saturated calomel electrode (SCE) as reference electrode and the uncovered and covered Ti as working electrode with an exposed surface area of 1 cm². The polarization curves of the Sr–Ca–P/GLT covered and uncovered Ti were obtained at a scan rate of 10 mV/s.

2.5. Cell experiments

Murine osteoblasts (MC3T3-E1) were used to study the osteoblastic cell response on specimens. The cells were cultured at 37 °C in a humidified 5% CO₂ atmosphere in α -minimal essential medium (MEM- α Hyclone) added with 1% penicillin/streptomycin (GIBCO) and 10% Fetal Bovine Serum (FBS, HyClone). For comparison, Ca–P coated Ti was designated as a control. For the cell assay, the specimens were thoroughly sterilized in an autoclave at 121 °C for 25 min before the cell experiments. Cell numbers were determined by counting via a haemocytometer, and then adjusted to the concentration of 5 × 10⁴ cells mL⁻¹.

When MC3T3-E1 cells were cultured for 1 day, the culture medium with non-adhered cells was carefully removed from the wells, and then rinsed with PBS buffer. The cells on the specimens were fixed with 2.5% glutaraldehyde for 2 h, and cleaned 2 times

Table 1
Amount of material needed to prepare 1 L of SBF.

Salt	Amount	Purity (%)	Formula weight
NaCl	8.306 g	99.5	58.44
NaHCO ₃	0.352 g	99.7	84.01
KCl	0.225 g	99.0	74.56
K ₂ HPO ₄ ·3H ₂ O	0.230 g	99.0	228.23
MgCl ₂ ·6H ₂ O	0.311 g	99.0	203.30
1 M-HCl	40 ml	–	–
CaCl ₂	0.293 g	99.0	110.98
Na ₂ SO ₄	0.072 g	99.0	142.04
(CH ₂ OH) ₃ CNH ₂ (Tris)	6.063 g	99.8	121.14
1 M HCl	0–0.2 ml	–	–

with PBS. Afterward, each specimen was graded dehydration with 30%, 40%, 50%, 60%, 70%, 80%, 90%, 95%, and 100% ethylalcohol for 10 min, respectively. Then the specimens were critical point dried using liquid CO₂ and then sputter-covered with gold film for SEM analysis.

After 3 days and 6 days of co-incubation, the culture medium was removed from each culture well, and the samples were transferred to new 24-cell culture plates. Subsequently, the cells were incubated with a tetrazolium salt solution, 3-[4,5-dimethylthiazol-2-yl]-2,5-diphenyltetrazolium bromide (MTT) for 4 h, and then the MTT solution was removed. Next, the dimethylsulfoxide (DMSO) was added into each well. The cell viability was evaluated at 490 nm on a spectrophotometric microplate reader.

3. Results and discussion

3.1. Characterization of as-deposited composite coating

The XRD pattern of the as-deposited composite coating is shown in Fig. 1a. The patterns show a mixture of HAP and DCPD (Brushite, CaHPO₄·2H₂O) phases that match the standard XRD patterns (JCPDS Nos. 09-0432 and 72-1240, respectively). DCPD, which is thermodynamically unstable and comparatively soluble under physiological conditions, is a superior precursor material for generating HAP [8]. The presence of an amorphous phase with broad peaks within the 2 θ regions of 30° to 35° was found in the pattern. This phase represents the amorphous organic component (GLT) deposited with calcium phosphate [13].

Fig. 1b shows the FTIR spectra of the as-deposited composite coating. The peaks at 3425 cm⁻¹ are attributed to HPO₄²⁻,

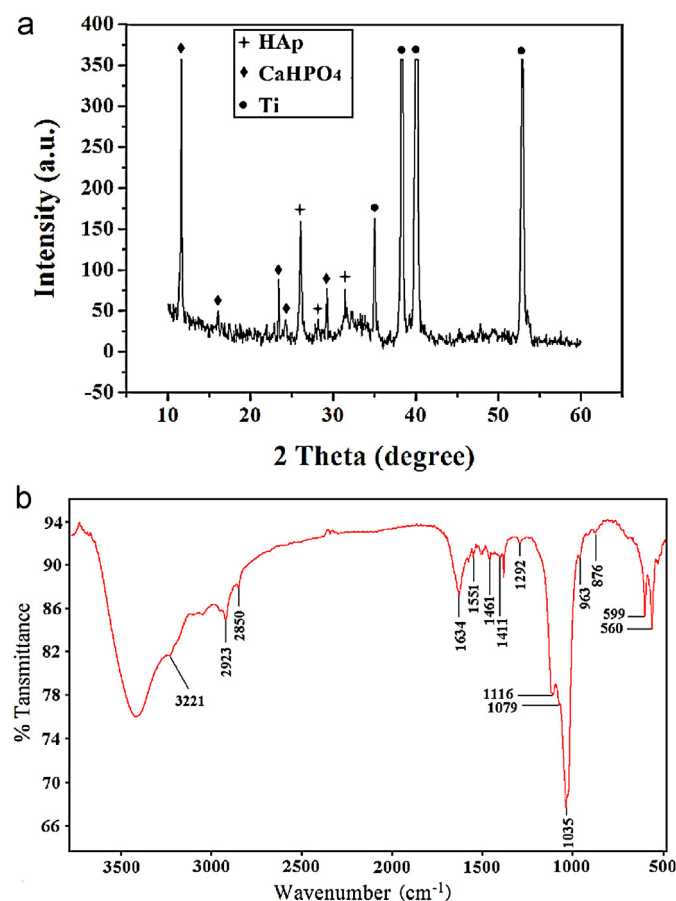


Fig. 1. (a) XRD patterns of Sr–Ca–P/GLT composite coating. (b) FTIR spectra of deposits scraped from Ti substrate.

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