

# Deposition of calcium phosphate coatings using condensed phosphates ( $P_2O_7^{4-}$ and $P_3O_{10}^{5-}$ ) as phosphate source through induction heating



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## ABSTRACT

In present work condensed phosphates ( $P_2O_7^{4-}$  and  $P_3O_{10}^{5-}$ ) were used as phosphate source in induction heating to deposit calcium phosphate coatings. The phase, morphology, and composition of different phosphate-related coatings were characterized and compared using XRD, FTIR, and SEM analyses. Results showed that  $P_2O_7^{4-}$  formed calcium pyrophosphate hydrate coatings with interconnected cuboid-like particles. The as-deposited calcium tripolyphosphate hydrate coating with  $P_3O_{10}^{5-}$  was mainly composed of flower-like particles assembled by plate-like crystals. The bioactivity and cytocompatibility of the coatings were also studied. Moreover, the feasibility of using hybrid phosphate sources for preparing and depositing coatings onto magnesium alloy was investigated.

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## 1. Introduction

Surface modification is essential to enhance the biocompatibility and osteoconductivity of inert orthopedic devices. Coating a base material with a thin layer of calcium phosphate (CaP) is a typical and effective approach used to enhance strong bone bonding to the implant in vivo [1]. Numerous techniques, such as biomimetic coating, microwave-assisted wet-chemical deposition, and plasma spraying, can be used to successfully deposit CaP coatings onto the implant surface [2–4].

Induction heating (IH) is a convenient and effective method for deposition of CaP coatings onto conductive materials [5]. CaP coatings can be successfully deposited within hours through electromagnetic induction heating to substrates immersed in aqueous solutions containing  $Ca^{2+}$  and  $PO_4^{3-}$ . This technology provides stronger coating binding force and faster coating rate than biomimetic coating. In our previous study, we determined the effects of temperature, processing time, and  $Mg^{2+}$  doping on CaP coating properties [6,7]. Increasing the temperature from 70 °C to 100 °C and prolonging processing from 0.5 h to 8 h could increase coating density and adhesion force. Moreover,  $Mg^{2+}$  doping could induce the formation of micropores and promote cell adhesion and proliferation. CaP contains both Ca- and P-based components. Replacement of either Ca or P component can alter the

properties of CaP. In particular, Ca-based components can be modified through  $Mg^{2+}$  doping.

This paper is inspired by our long-standing efforts to apply condensed phosphates for generating CaPs [8,9]. Condensed phosphates are more important in some biomolecules, such as guanosine-5'-triphosphate and adenosine triphosphate, than regular  $PO_4^{3-}$ . The phosphate chain of these molecules can be broken down by enzyme hydrolysis to release  $PO_4^{3-}$ . Supplementation of condensed phosphates in bone formation promotes the differentiation and maturation of osteoblasts [10,11] but inhibits the activity of osteoclast cells [12]. The properties of CaP coatings can be controlled by modifying the composition of aqueous solutions for synthesis [2,13]. However, condensed phosphates can be degraded under hydrothermal treatment; specifically, heat is generated around the coil during IH. Therefore, the influences of replacing  $PO_4^{3-}$  with condensed phosphates on CaP coatings deposited through IH must be investigated.

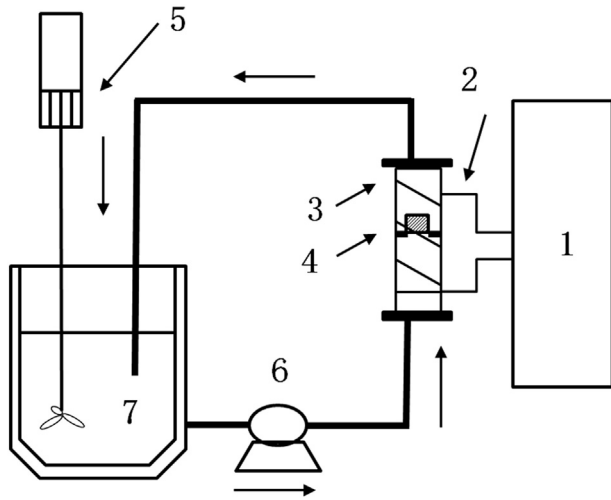
## 2. Material and methods

### 2.1. IH coating system set-up

IH coating deposition was performed using SP-15 high-frequency induction power with a frequency of 15 kHz (Shenzhen SuanPin Power Co. Ltd). The experimental setup for induction heat deposition is similar to that in reference [5]. This system is composed of an induction current power, which consists of a copper coil with appropriate water cooling, a

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**Fig. 1.** Schematic graph of IH coating system: (1) induction powder, (2) copper coil, (3) glass tube, (4) sample, (5) agitator, (6) pump, and (7) coating solution.

glass tube of 20 mm diameter inside the coil, a peristaltic pump, a feeding tank, and tubes that connect the feeding tank to the bottom of the glass tube and the top of the glass tube to the tank for the residual solution. A narrowed area of 16 mm diameter is inside the glass tube and supports the samples in a vertical position. The schematic of the coating system is presented in Fig. 1.

## 2.2. IH coating deposition onto carbon/carbon substrates

All chemicals were purchased from Aladdin (Shanghai, China). The coating solution used in this study was prepared by dissolving given amounts of reagent-grade  $\text{CaCl}_2$  and different phosphate sources into distilled water (Table 1). The pH of solution was adjusted to 4.5 by adding 1 M HCl to obtain a transparent solution.

Cylindrical carbon/carbon (C/C) composites with 8 mm diameter and 10 mm length were prepared by Northwest Polytechnique University in China. Samples were pretreated in high-pressure steam in a 50 mL autoclave with 40 mL of 2 M  $\text{H}_2\text{O}_2$  solution at 160 °C before coating. The cylinders were removed from the  $\text{H}_2\text{O}_2$  solution, rinsed ultrasonically with deionized water, and dried in air. The C/C sample was subsequently placed into a tube and coated for 4 h by using different solutions. The C/C samples were rewashed using deionized water to remove uncoated powder on the surface. The samples were then immersed in an autoclave containing 20% ammonia water at 130 °C for 6 h to mature the deposited crystals.

The coated samples were characterized using X-ray diffraction (XRD, D/MAX2500, Rigaku, Japan), Fourier transform infrared spectroscopy (FTIR, PROTÉGÉ 460, Nicolet, USA), and scanning electron microscopy (SEM, SUPRA 55, Zeiss, Germany) analyses. The bioactivity of the coatings was studied in vitro by soaking the samples in simulated body fluid (SBF) for up to 2 weeks; SBF was replaced every other day [14].

**Table 1**  
Compositions of aqueous coating solution using different phosphate source.

Sample	$\text{CaCl}_2$ (g)	Phosphate source (g)		$\text{H}_2\text{O}$ (mL)	Ca/P
Ca–P	17.4	$\text{NH}_4\text{H}_2\text{PO}_4$	10.8	1000	1.67
Ca–P2	17.4	$\text{Na}_4\text{P}_2\text{O}_7$	12.5	1000	1.67
Ca–P3	17.4	$\text{Na}_5\text{P}_3\text{O}_{10}$	11.5	1000	1.67

**Table 2**  
Compositions of aqueous coating solution using hybrid phosphate sources.

Sample	$\text{CaCl}_2$ (g)	$\text{NH}_4\text{H}_2\text{PO}_4$ (g)	$\text{Na}_4\text{P}_2\text{O}_7$ (g)	$\text{Na}_5\text{P}_3\text{O}_{10}$ (g)	$\text{H}_2\text{O}$ (mL)	Ca/P
Ca–P–P2	17.4	7.2	4.6	–	1000	1.67
Ca–P–P3	17.4	7.2	–	3.83	1000	1.67

The incubated samples were examined using SEM. Moreover, the cytocompatibility of the deposited coatings was evaluated by seeding 10,000 MC3T3-E1 osteoblast cells on the coated sample surface. The osteoblast cells were provided by Lei Yang, Soochow University. The samples were further incubated for up to 7 d, and the medium was refreshed every other day. The number of cells in each group on days 1 and 7 was measured through MTT assay to determine cell density.

## 2.3. IH coating deposition using solution of hybrid phosphate sources

The feasibility of depositing hybrid composite coatings onto the C/C sample surface was studied using solutions containing hybrid phosphate sources (Table 2). Coating was conducted in the same way as described above. The as-prepared samples were characterized using XRD and SEM analyses.

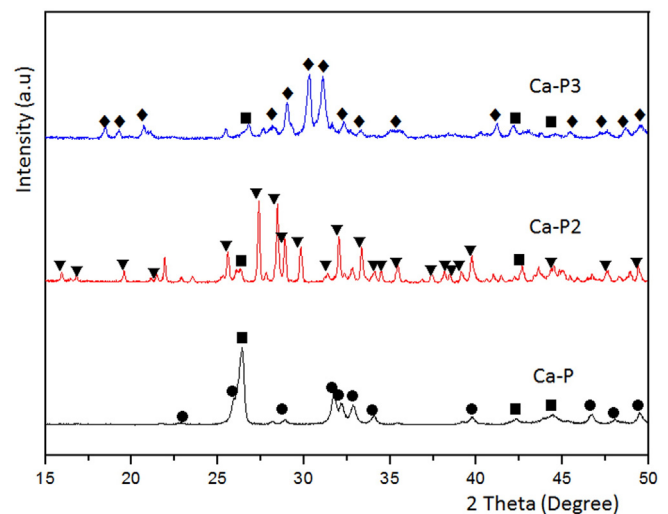
## 2.4. IH coating deposition onto magnesium substrates

Mg alloy (AZ31) square with 15 mm side length was placed into the IH coating system. Solutions containing  $\text{PO}_4^{3-}$ ,  $\text{P}_2\text{O}_7^{4-}$ , or  $\text{P}_3\text{O}_{10}^{5-}$  were used to coat Mg alloys by following the steps described above. The coated samples were compared visually.

## 3. Results

### 3.1. IH coating deposition onto carbon/carbon substrates

The XRD patterns of the coated samples are presented in Fig. 2. After deposition for 2 h, the characteristic diffraction peaks on the surface of hydroxyapatite (HA,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , PDF#09-0432) crystal almost



**Fig. 2.** XRD patterns of the coated C/C composite samples, “■” refers to carbon, “●” refers to HA, “▼” refers to CPPH, and “◆” refers to unknown phase.

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