



In-vitro formation and growth kinetics of apatite on a new light-cured composite calcium phosphate cement



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ABSTRACT

Calcium phosphate cements are used as synthetic bone grafts with several advantages such as biocompatibility, osteoconductivity, and moldability. In this study, the synthesis of a bio-cement starting from calcium hydroxide (Ca(OH)₂) and Monocalcium Phosphate Monohydrate (MCPM) was investigated. A 6 wt% Na₂HPO₄ aqueous solution along with a modified polymeric resin (RIVA(SDI)[®]) were adopted as the variable liquid phase in self- and light-cure cement groups. XRD analysis and FTIR spectroscopy were used to study the phase composition. The composite microstructure was characterized by scanning electron microscopy (SEM) and the degradation rates were measured by atomic absorption spectroscopy (AAS) analysis. In addition, the effect of soaking time of the cement in simulated body fluid (SBF) on the final phase and morphology was studied. The results showed that soaking the composite in SBF has a significant influence in phase transformation into hydroxyapatite, but following a slower kinetic in light-cured composite cements. Evidences of crosslinking reactions in light-cured cements were observable, which at the same time can legitimize slower apatite formation and faster biodegradation of these composite cements.

1. Introduction

Calcium phosphates (CaPs) are considered ideal for bone substitution as they resemble the bone mineral phase. The matrix biomaterial should possess appropriate mechanical stability to support cell growth. When a flexible easy-shaping synthetic organic polymer and a spontaneously depositing apatite layer of ceramics are coalesced, the resulting organic-inorganic composite can cover most requirements of an ideal biomaterial. Hydraulic CaP cements usually consist of one or more calcium phosphate precursor powder along with an aqueous liquid that form a homogenous paste after mixing. The setting mechanism in most CaP cements results in either precipitated hydroxyapatite (HA) at pH > 4.2, or brushite (dicalcium phosphate dihydrate, DCPD) at pH < 4.2, as the most stable end products [1]. Majority of the osteoconductive bioactive hydraulic CaP cements slowly set in low temperatures and show weak mechanical properties [2–4]. However, manipulations after mixing and during the precipitation can lead to malconfiguration of crystals, inhomogeneity of the cement, and substantial fall in mechanical strength [5].

Light curable CaP cements are potential alternatives that allow delicate crafting during clinical application [6–9]. The conventionally

available resin modified glass ionomers have been qualified to the minimal setting exotherm, fast acid neutralization and gradual release of useful ions [10]. The resin phase is a biocompatible material widely used in light-cure glass ionomer dental cements for lining and canal sealing [11]. On the contrary to common hydraulic CaP cements, light curing CaP cements which set through photopolymerization of a resin modified glass ionomer cement show superior mechanical properties, reduced setting time, decreased early moisture sensitivity, and extended working time [12–14]. Photoinitiators play an important role in photopolymerization process through the absorption of UV or visible-light energy, transforming it into radicals or ions, and inducing chain-growth polymerization. Cross-linking the oligomers in the curing process initiated by photoinitiators results in a network thermoset polymer [15].

In this study, a resin-modified glass-ionomer cement (RMGIC) was used as the liquid phase of a new composite calcium phosphate cement. The structural development and morphology of the synthesized bio-cements were investigated.

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2. Materials and methods

A mixture (1:2M ratio) of dried (120 °C, 24 h) reagent-grade Monocalcium Phosphate Monohydrate (MCPM) (Sigma-Aldrich, USA) (MW = 252.07 g/mol) and Ca(OH)₂ (Merck, Germany) (MW = 74.10 g/mol) were milled with 12–15 ml absolute ethanol in a planetary ball mill at 180 rpm for 3 h. The mixture was dried at 80 °C for 12 h and then sieved to obtain a homogenous powder mixture (average particle size below 40–45 μm). The final Ca/P molar ratio was 1.5.

2.1. Synthesis of LP₅₀ sample (hydraulic self-cure)

According to the previous studies LP50 sample was prepared as a commonly reported composition [16]. The cement powder mixture was further processed by mixing a phosphate solution (6 wt% Na₂HPO₄). The aqueous phosphate solution was prepared by dissolving 1.2 g disodium phosphate in 20 ml double distilled water. The liquid/Powder ratio (L/P) was taken to be 0.5 (w/w). The cement samples were prepared by mixing the powder and liquid in a mortar for about 1 min. Equal pellet shaped samples were dried overnight at room temperature.

2.2. Synthesis of LP30 and LP100 samples (composite light cure)

The polymeric phase of a light-cure glass ionomer dental cement (RIVA®, SDI, Australia) was adopted as the liquid part of calcium phosphate cement. These glass ionomer cements are composed of a fluoroaluminosilicate glass powder along with a modified polymeric resin. The LP30 and LP100 samples were fabricated according to the instructions for preparation of the dental cement [17].

The modified polymeric resin consists of polyacrylic acid/tartaric acid (PA/TA) and 2-hydroxyethyl methacrylate (2-HEMA). Dimethacrylate cross-linkers and acidic monomers are also parts of the precursors of this resin.

The liquid resin was mixed with the previously processed cement powder mixture in two different proportions (liquid/powder (w/w): 0.3 and 1) to yield homogenous pastes for each of the LP₃₀ and LP₁₀₀ sample groups. The pellet-shaped specimens were photopolymerized by sequential irradiation of both sides for 40 s with visible light source at room temperature (according to manufacturer's datasheet).

2.3. In-vitro bioactivity assessments

Each sample was incubated in 6 ml SBF at 37 °C for 7 and 14 days. The solution was refreshed every other day. At each time point the specimens were gently rinsed with distilled water and dried overnight at room temperature for FTIR, XRD, and SEM analysis. The reacted solutions were conserved in 4 °C for AAS analysis.

2.4. X-ray diffraction (XRD)

The structural changes in the specimens were evaluated by X-ray diffraction (PHILIPS, PW1730). The XRD patterns were recorded in the 10° ≤ 2θ ≤ 80° interval at 2°/min scan speed and 0.05° step size.

2.5. Fourier transform infrared spectroscopy (FTIR)

FTIR was performed to detect the functional groups such as phosphate (PO₄³⁻), hydroxyl (OH), and carbonate (CO₃²⁻) that are assignable to apatite formed on the cement surface after immersion in SBF solution. It also provides more information about setting mechanism. A thin layer of each sample was scratched off and embedded in potassium bromide (infrared grade KBr) pellet. The structure of the grinded samples was examined by FTIR spectroscopy (Bruker vector 33). The signals were collected at 2 cm⁻¹ resolution in the range of 400–4000 cm⁻¹.

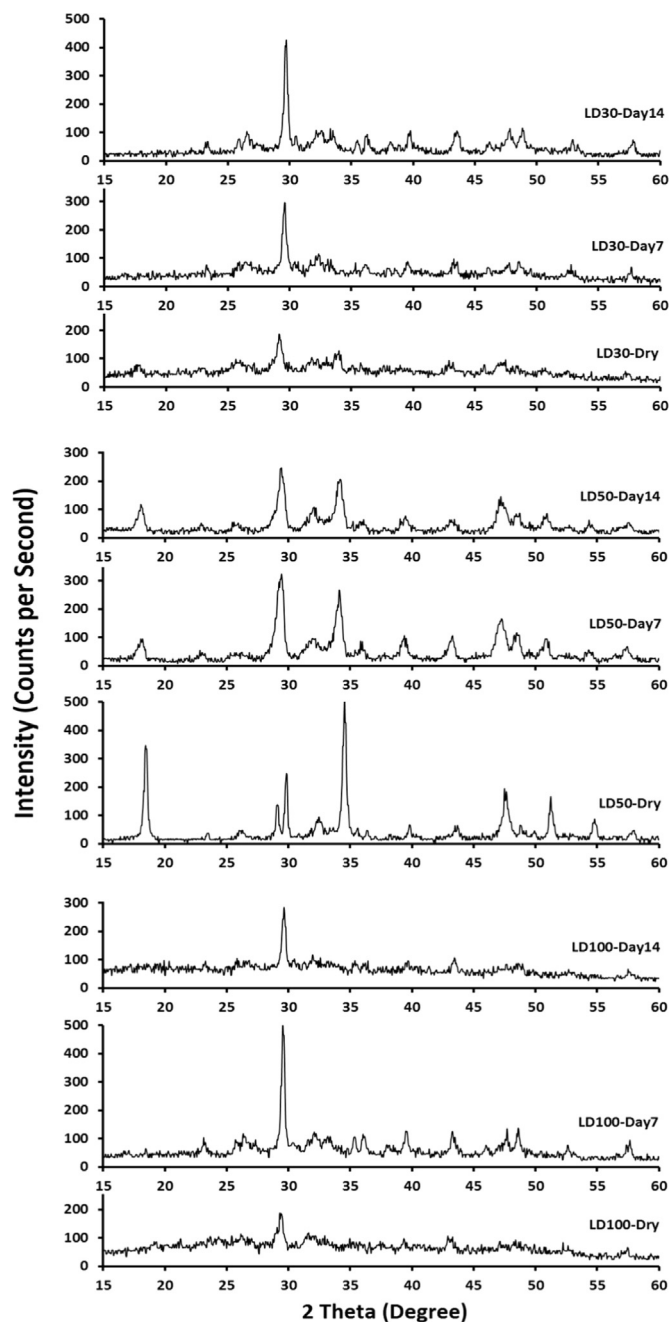


Fig. 1. XRD pattern of LP50, LP30, and LP100 samples before and after immersion in SBF for 7 and 14 days.

2.6. Scanning electron microscopy (SEM)

The surface microstructures of gold-palladium coated samples (at a thickness of around 450 Å using a 150 rotary-pumped sputter coater (Quorum Technologies)) were observed at 5.0 kx under a scanning electron microscope (MIRA3 TESCAN 2XMU) at 15 kV.

2.7. Atomic absorption spectroscopy (AAS)

Changes in SBF pH and Ca concentration were measured by a pH meter (Metrohm 691) and atomic absorption spectroscopy (AAS, GBC-932⁺), respectively. After a short centrifugation at 250 rpm for precipitation of the suspending particles, the SBF solution was filtered using a Whatman® paper. The clear solution was assessed using a Calcium (Ca) lumina hollow cathode lamp at 422.7 nm.

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