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Reinforcement of experimental composite materials based on amorphous calcium phosphate with inert fillers

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ABSTRACT

Objectives. The aim of this study was to examine the influence of the addition of glass fillers with different sizes and degrees of silanization percentages to remineralizing composite materials based on amorphous calcium phosphate (ACP).

Methods. Four different materials were tested in this study. Three ACP based materials: 0-ACP (40 wt% ACP, 60 wt% resin), Ba-ACP (40 wt% ACP, 50 wt% resin, 10 wt% barium-glass) and Sr-ACP (40 wt% ACP, 50 wt% resin, 10 wt% strontium-glass) were compared to the control material, resin modified glass ionomer (Fuji II LC capsule, GC, Japan). The fillers and composites were characterized using scanning electron microscopy. Flexural strength and modulus were determined using a three-point bending test. Calcium and phosphate ion release from ACP based composites was measured using inductively coupled plasma atomic emission spectroscopy.

Results. The addition of barium-glass fillers (35.4 (29.1–42.1) MPa) (median (25–75%)) had improved the flexural strength in comparison to the 0-ACP (24.8 (20.8–36.9) MPa) and glass ionomer control (33.1 (29.7–36.2) MPa). The admixture of strontium-glass (20.3 (19.5–22.2) MPa) did not have any effect on flexural strength, but significantly improved its flexural modulus (6.4 (4.8–6.9) GPa) in comparison to 0-ACP (3.9 (3.4–4.1) GPa) and Ba-ACP (4.6 (4.2–6.9) GPa). Ion release kinetics was not affected by the addition of inert fillers to the ACP composites. **Significance.** Incorporation of barium-glass fillers to the composition of ACP composites contributed to the improvement of flexural strength and modulus, with no adverse influence on ion release profiles.

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1. Introduction

Contemporary restorative dental medicine requires not only esthetic materials which restore tooth structures, but also materials which are able to heal carious-affected hard dental tissue [1]. Over the last few decades, efforts have been increased to produce bioactive materials able to reverse the carious process and to remineralize caries-affected tissue. Amorphous calcium phosphate (ACP) based composite resins are intended as remineralizing/anti-demineralizing agents. ACP is well known as a direct precursor of hydroxyapatite (HA) and has a major role in the biomineralization processes of teeth and bones [2,3]. In an aqueous environment, ACP composite materials release calcium and phosphate ions, providing supersaturating concentrations sufficient to trigger the apatite build-up [4,5] and remineralize demineralized enamel [6,7].

At the same time, those characteristics pose limitations in clinical applications when durability or resistance to crack and deformation upon load are required. Bioactive ACP particles do not have a reinforcing role such as silanized glass or silica fillers, which are used in most of the commercially available dental composite materials. ACP particles are not silanized, as this has an adverse effect on calcium and phosphate ion release without providing any advancement in mechanical properties [8]. Hence, the amount of ACP had to be reduced to the minimal level, which also preserved the remineralizing properties without additionally undermining the mechanical properties. This has led to the optimization of the composition to 40 wt.% of ACP [4].

Flexural strength of composite materials is mostly dominated by the degree of conversion of the organic matrix [9,10], filler volume [11] and the filler to matrix interfacial relationship [12]. In conventional composites, filler particles increase strength, stiffness and decrease dimensional changes [13], while their silanization ensures better resin to filler interaction and deflects the fracture line. This contributes to higher flexural strength and an overall improvement of mechanical properties [14,15]. The filler load is directly correlated to the flexural strength and modulus, as stated by many authors [11,16–18]. However, fillers without silanization do not provide sufficiently high flexural strength for composite materials [12]. Composites with silanized fillers show higher flexural strength in comparison to those with unsilanized fillers [12,15,16]. A lack of silanized reinforcing fillers in ACP based composite resins have insufficient mechanical properties as a consequence [8].

This group of authors has recently tested the influence of the addition of silanized nanosilica to the ACP composite resin formulation. The study proved that the principle of the admixture of inert fillers is successful in improving flexural strength and increasing the level of calcium and phosphate ion release in comparison to ACP composites without non-releasing fillers [19]. However, it was emphasized that the agglomeration of silica nanoparticles, due to their large surface area, may contribute to the hydrolytic degradation. This in turn enhances ion release, but it also might negatively affect the long-term mechanical properties. Similar conclusions were drawn in another study which examined the effect of various types of

silanized fillers on the degree of conversion of ACP composite resins [20]. Taking into account that the random clustering of ACP particles in resin matrix is also recognized as one of the reasons for diminished strength of ACP composites [5], any additional agglomeration is an undesired property. Different authors agree that the agglomerated particles could act as strength controlling flaws which initiate crack and consequently lead to fracture [21,22].

In contrast, conventional glass microfillers have a lower surface area than nanofillers and do not show the tendency to agglomeration. Thus, it is expected that their addition to ACP composites might provide better interaction to the resin phase and higher strength and modulus. At the same time, it is necessary to examine if the inert glass fillers interfere with the ion release kinetics. The present study was aimed to investigate the effect of glass fillers of various sizes and degrees of silanization on ACP based composite resins. The null-hypothesis was that the addition of fillers does not have an influence on flexural strength, flexural modulus and ion release of ACP based composites.

2. Materials and methods

2.1. Materials

2.1.1. Synthesis of zirconia ACP fillers

The synthesis of Zr-ACP fillers followed the procedure by Skrtic et al. [23]. Zr-ACP precipitated instantaneously in a closed system at 23 °C upon rapidly mixing equal volumes of a 800 mmol/L $\text{Ca}(\text{NO}_3)_2$ solution, a 536 mmol/L Na_2HPO_4 solution that contained a molar fraction of 2% $\text{Na}_4\text{P}_2\text{O}_7$ as a stabilizing component for ACP, and an appropriate volume of a 250 mmol/L ZrOCl_2 solution (mole fraction of 10% based on the calcium reactant) [24]. The reaction pH varied between 8.6 and 9.0. The suspension was filtered; the solid phase was washed subsequently with ice-cold ammoniated water and acetone and then lyophilized. ACP fillers were kept in a desiccator to avoid exposure to humidity and premature conversion to apatite until being used in composites.

2.1.2. Formulation of resin

The experimental resin was the same for all ACP based materials containing 62.8 wt% of ethoxylated bisphenol A dimethacrylate (EBPADMA; Esstech, PA, USA), 23.2 wt% of triethylene glycol dimethacrylate (TEGDMA; Esstech), 10.4 wt% of 2-hydroxyethyl methacrylate (HEMA; Esstech), 2.6 wt% of methacryloxyethyl phthalate (MEP; Esstech), 0.2 wt% of the photo oxidant camphorquinone (CQ; Aldrich, WI, USA) and 0.8 wt% of photo reductant ethyl-4- (dimethylamino) benzoate (4E; Aldrich). Using a magnetic stirrer, the monomers and photo activators were mixed (in the absence of blue light) until a uniform consistency was achieved.

2.1.3. Tested materials

The compositions of the three ACP composite materials used in this study, as well as the glass-ionomer control are shown in Table 1. The ACP fillers, silanized glass fillers (Table 2) and resin were mixed in lightproof containers in a dual asymmetric centrifugal mixing system (Speed Mixer TM DAC 150 FVZ,

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