



Evaluation of reinforced strength and remineralized potential of resins with nanocrystallites and silica modified filler surfaces

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ABSTRACT

Surface-modified dicalcium phosphate anhydrous particles that were treated with an ion-rich solution and a silane-coupling agent were evaluated as fillers for resin composites. The physiochemical properties of these composites were characterized. The properties of the specimens as reinforcements, which were modified using various surface conditions and 30% and 50% filler to composite mass ratios (30% and 50%) were measured before and after they were immersed in water for 24 h. All groups were of the same strength and showed no significant changes after immersion. However, the groups showed a significant increase in the modulus after 24 h of immersion. The filler surfaces with nanocrystallites had the highest modulus, whereas the fillers treated with silanization had the lowest ion concentration in the solution and highest remineralization ability after immersion. The strength and brittleness were increased by the modified fillers with nanocrystallites on the surfaces and by the increased amount of fillers in the resin composites. Filler surfaces that were modified with silica hindered interfacial interactions and consequently had better flexibility and less brittleness during the light-curing process. Surface modifications of reinforced particles using nanocrystallites and silica films have superior potential applications in restorative medicine.

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1. Introduction

The two major challenges when using dental restorative composite resins are secondary caries and restoration fractures [1]. With the advances in nanotechnology and biomaterials, novel dental composite resins have been developed to prevent secondary caries and, moreover, to regenerate tooth structure [2]. Calcium phosphates (CaPs) have been studied and used as reinforcements in the dental resin matrix for the prevention of caries in composite resin fillers. For example, hydroxyapatite (HA; $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), is the structural prototype of the major mineral component in the enamel and the cortical parts of teeth and bones. HA is the final stable product in the precipitation of high concentrations of calcium (Ca^{2+}) and phosphate ions, namely, H_2PO_4^- , HPO_4^{2-} , and PO_4^{3-} , existing in solutions from neutral or basic environments (pH of 7 to 9 at 37 °C) [3]. Dental composite resins composed of CaP fillers in a resin matrix have been proposed; these have the potential to remineralize carious enamel and dentin lesions *in vitro* [4–6]. The fillers have similar

functions as those of the re-mineralizing agents in anti-sensitivity toothpaste. These agents deliver supersaturated ions to the surface to promote the restoration of demineralized areas. Restorative materials that contain CaPs as reinforcements in a polymer matrix are referred to as “smart composites” in several studies [7].

Skrtric et al. [8] evaluated the mechanical strength of amorphous CaPs and found that this weak filler could only be used as a preventive sealant. Another group used HA combined with silica whiskers as the filler for dental resin composites and substantially improved the mechanical properties of composites filled with calcium phosphate cement (CPC) particles without whiskers [9]. Recent studies performed by Xu et al. [10–14] demonstrated the mechanical properties and the ion-releasing ability at different pH values of tetracalcium phosphate (TTCP) and anhydrous dicalcium phosphate (DCPA), which are the main ingredients of CPC, when these were mixed separately with silica whisker fillers of varied particle sizes and filler-to-matrix mass ratios. The results suggest that the combination of releasing nano-fillers with stable and strong reinforcing fillers may yield a resin matrix composite with both stress-bearing and caries-inhibiting capacities; however, such combined materials are not yet available in dental restoration to date [15].

Difficulties arise in the long-term remineralization abilities of the materials and their performances when used in a dynamic oral environment. Recent studies have investigated the effects of relatively small amounts of CaP fillers, which were hardly exposed to the top surfaces of the restorative resins [9–15]. Furthermore, their dissolution

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causes rapid ion exhaustion prior to surface layer remineralization. In our previous studies [16,17], we characterized a novel modified surface treatment method using TTCP to reinforce CaP bone cements. The few noteworthy studies on the remineralization potential of composite resin on DCPA ceramics recommended the use of reinforcement and modified resin bonding agents that contain hydroxyl groups and silicates for increased, durable bond strengths. The objectives of the present study were to characterize the physiochemical effects of different amounts of the filler based on the unmodified and modified surface pretreatment of DCPA with nanocrystallites or further silanization and to evaluate the remineralization abilities at the early stages of immersion. These applications are intended to minimize or prevent the cycle of secondary caries and restoration failure.

2. Materials and methods

2.1. Treatment of nanocrystallite film

The particle surfaces of the DCPA crystallites were treated using our previously developed method with some modifications [18]. DCPA powder (CaHPO_4 , Alfa Aesar GmbH & Co. KG, Karlsruhe, Germany) was used, which had a particle distribution size of (1 to 3) μm and a purity of 98%. Crystallite formation on the filler surfaces was performed using 5 g of DCPA powder mixed in 40 mL saturated solution with a molar calcium-to-phosphate ratio of 2.0 and operated at a stabilized lower pH value between 4.5 and 5.0 for 20 min at room temperature (26 °C). After the solid film of nanocrystallites was deposited on the powders, the particles were vacuum filtered, washed twice with deionized water, rinsed with 95% alcohol, and then dried in an oven. To examine the effects of the surface coating on the particles, a drop of the surface-treated powder that was dispersed in ethanol was placed on a #325 mesh carbon grid (3 mm in diameter) and allowed to dry. The specimen was then coated with a thin carbon film for electrical conductivity during transmission electron microscopy (TEM). A JEOL JEM-3010 electron microscope was used at 200 kV. The observed patterns were indexed, identified in detail, measured, and compared with the d value in the Joint Committee on Powder Diffraction Standards (JCPDS). The observed diffraction patterns were compared with those of known CaP phases and other generated electron diffraction.

2.2. Silica modification

A solution of 100 mL cyclohexane solvent with 4 (v/v%) (3-mercaptopropyl) trimethoxysilane (MPTMS) and 2 (v/v%) *n*-propylamine (Alfa Aesar GmbH & Co. KG, Karlsruhe, Germany) was used as the saline-coupling agent for the silica modification of the particle surfaces. DCPA powder (5 g) was added to the colloidal solution with rapid agitation for 30 min at room temperature. The temperature was then raised to 60 °C, and the solvent was further removed by drying the samples in a vacuum. The effects of the deposited organosilane films on the filler surface were evaluated by Fourier-transform infrared spectroscopy (FT-IR) analysis (Nicolet 6700; Thermo, MA, USA).

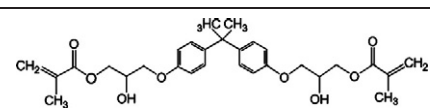
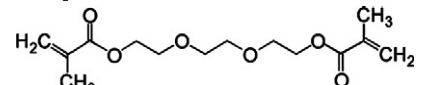
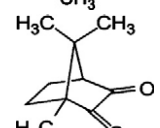
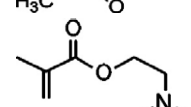
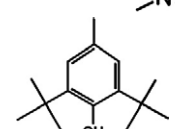
2.3. Resin composites

The resin matrix was mainly composed of bisphenol-A diglycidyl methacrylate (bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) monomers. The free radical polymerization initiation system in the photocurable composite resin used the light initiator camphoroquinone (CQ, Sigma-Aldrich Co., Buchs, Switzerland) with dimethylaminoethyl methacrylate (DMAEMA) as the accelerator and butylated hydroxyl toluene (BHT) as the photostabilizer. The chemicals used in the present study were all obtained from Sigma-Aldrich, and their molecular formulas are listed in Table 1. The resin matrix was composed of 48.75 wt.% bis-GMA, 48.75 wt.% TEGDMA, 1.0 wt.% CQ, 1.0 wt.% DMAEMA, and 0.50 wt.% BHT. To form a resin composite, the organic matrix and inorganic fillers of the unmodified and surface-treated DCPA featuring the filler-based resin composites with filler-to-composite ratios of 30 wt.% and 50 wt.% were prepared in a dark room and mixed with a magnetic stirrer until the colloid was well formed. The resin composite was uniformly mixed and then loaded into a 10 mL syringe for injection into the mold. The syringe was covered in aluminum foil to prevent any possible reactions induced by room light. The resin composites were used within 24 h after preparation.

2.4. Physiochemical analyses

To evaluate the effects on the strength of the particle surfaces that were modified with (w) or without (w/o) nanocrystallites, silica, and

Table 1
Materials used as the matrix in the dental composite resins.

Materials, abbreviations and chemical formula	Structural formula
Bisphenol A diglycidyl methacrylate, Bis-GMA (2,2-bis[p(2'-hydroxy-3'-methacryloxypropoxy)phenyl] propane)	
Triethylene glycol dimethacrylate, TEGDMA 2-[2-[2-(2-methylprop-2-enoyloxy)ethoxy]ethoxy]ethyl 2-methylprop-2-enoate	
Camphoroquinone, CQ 1.7.7-trimethylbicyclo-[2.2.1]-hepta-2,3-dione	
Dimethylaminoethyl methacrylate, DMAEMA Methacrylic acid 2-(dimethylamino)ethyl ester	
Butylated hydroxyl toluene, BHT 2,6-Bis(1,1-dimethylethyl)-4-methylphenol	

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