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Effect of bioactive glass-containing resin composite on dentin remineralization

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ABSTRACT

Objectives: The purpose of this study was to evaluate the effect of bioactive glass (BAG)-containing composite on dentin remineralization.

Methods: Sixty-six dentin disks with 3 mm thickness were prepared from thirty-three bovine incisors. The following six experimental groups were prepared according to type of composite (control and experimental) and storage solutions (simulated body fluid [SBF] and phosphate-buffered saline [PBS]): 1 (undemineralized); 2 (demineralized); 3 (demineralized with control composite in SBF); 4 (demineralized with control composite in PBS); 5 (demineralized with experimental composite in SBF); and 6 (demineralized with experimental composite in PBS). BAG65S (65% Si, 31% Ca, and 4% P) was prepared via the sol-gel method. The control composite was made with a 50:50 Bis-GMA:TEGDMA resin matrix, 57 wt% strontium glass, and 15 wt% aerosol silica. The experimental composite had the same resin and filler, but with 15 wt% BAG65S replacing the aerosol silica. For groups 3–6, composite disks (20 × 10 × 2 mm) were prepared and approximated to the dentin disks and stored in PBS or SBF for 2 weeks. Micro-hardness test, attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) and field-emission scanning electron microscopy (FE-SEM) was investigated.

Results: The BAG-containing composite significantly increased the micro-hardness of the adjacent demineralized dentin. ATR-FTIR revealed calcium phosphate peaks on the surface of the groups which used BAG-containing composite. FE-SEM revealed surface deposits partially occluding the dentin surface. No significant difference was found between SBF and PBS storage.

Clinical Significance: Bioactive glass is a potentially useful material for remineralization of tooth structure. When incorporated into a resin composite, it may aid in remineralizing the adjacent demineralized dentin, thus preventing further destruction of the tooth.

Conclusion: BAG-containing composites placed in close proximity can partially remineralize adjacent demineralized dentin.

1. Introduction

Demineralization of exposed dentin is usually caused by acids from certain bacteria in the oral cavity [1]. Another factor causing dentin demineralization is phosphoric acid etching. When dentin is demineralized with phosphoric acid, collagen fibers become exposed and should subsequently be encapsulated with adhesive monomers to prevent collagen degradation over time. Otherwise, exposed, unprotected collagen fibers may be degraded by endogenous enzymes such as matrix metalloproteinases (MMP) or cysteine cathepsins [2]. To date, complete encapsulation of exposed collagen fibers with adhesive monomers has not been possible [3].

Remineralization of demineralized dentin may reduce collagen degradation [4], and specific agents have been used for this purpose. Fluoride is incorporated in some materials, such as varnish, gel, and certain restorative materials, in order to strengthen the tooth and make

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it more insoluble by transforming hydroxyapatite to fluoroapatite [5]. Although it has a high affinity for hydroxyapatite crystals, fluoride cannot react with the hydroxyapatite located in deeper areas because it is trapped by forming fluoroapatite crystals within the superficial layer of the tooth [6]. Casein phosphopeptide-amorphous calcium phosphate is another remineralizing agent. It has antibacterial and buffering effects on dental plaque, thereby interfering with the growth of dental plaque, decreasing demineralization, and promoting remineralization [7].

Bioactive glass (BAG) was first invented by Larry Hench in 1969 [8]. It can bind to both soft and hard tissues and stimulate tissue mineralization owing to its high bioactivity index ($I_B = 12.5$) [9]. It has been used for monolithic medical devices, bone regeneration, and toothpaste [10]. Recently, BAG showed the ability to remineralize enamel [11] and dentin [12], as well as low cytotoxicity to dental pulp cells [13] and antimicrobial activity against intraoral bacteria [14].

Many studies have focused on BAG-containing experimental products. When BAG is incorporated in a dentin desensitizer, it can reduce the movement of fluid across the dentin surface [15]. The adhesion of resin-modified glass ionomer to dentin has also been shown to be enhanced by BAG application [16]. A BAG-containing dentin adhesive improves the nano-mechanical properties of demineralized dentin [17]. As a composite filler, BAG releases calcium, and in certain formulations fluoride ions [18], increasing the elastic modulus of demineralized dentin [19]. BAG-containing resin composites show similar mechanical properties to conventional composite [20], and have antibacterial activities [21]. In addition, MMP activity within dentin was shown to be reduced when BAG was incorporated in an experimental resin cement [22].

Despite numerous studies, it is unclear whether BAG can remineralize adjacent demineralized dentin when incorporated in a resin composite. The purpose of this study was to investigate the dentin remineralization effect of BAG via micro-hardness test, attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) analysis, and field-emission scanning electron microscopy (FE-SEM). The hypothesis was that the BAG-containing resin composite would cause an increase in the micro-hardness of adjacent demineralized dentin, with evidence of new mineral formation at the surface.

2. Materials and methods

2.1. Preparation of dentin specimen

Thirty-three bovine mandibular incisors were sectioned with a low-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) perpendicularly to the long axis of the tooth to create disks with a 3 mm thickness. Two dentin disks were produced from each tooth, yielding sixty-six disks. Then, the lingual half was cut from each disk, leaving only the labial half. Each half disk was polished with silicon carbide (SIC) papers in ascending order from 180-, 320-, and 600-grit silicon carbide(SIC) to produce a standard smear layer.

2.2. BAG synthesis

BAG65S was prepared with a composition of 65% Si, 31% Ca, and 4% P (in mol%) via the sol-gel method as described by Davis et al. [18]. Briefly, calcium methoxyethoxide was first synthesized from methoxyethanol and calcium metal, and then combined with triethyl phosphate and tetraethyl orthosilicate under nitrogen gas to produce a homogenous solution. To this solution, 10% boron trifluoride etherate was added by drops over a 20 min. The solution was then stored in an incubator at 37 °C and 100% humidity for 7 days to create a monolithic product. The product was then completely hydrolyzed with deionized water. After 3 days, all the solvents were dried, and the prepared gel was rinsed with 100% ethanol. The gel was then heated in a furnace at 600 °C until a transparent glass was produced. The final glass was crushed to a powder, first by ball milling and subsequently by micronizing (Model OM2; Sturtevant, Hanover, MA, USA). The average particle size ranged from 0.04 to 3.0 μm, as determined with a laser diffraction particle-size analyzer (Beckman Coulter LS13 320, Brea, CA).

Table 1
The chemical composition of composite resin used in this study.

Composite resin	Chemical composition (wt%)	
Control composite without BAG	Matrix	combination of 50:50 BisGMA:TEGDMA (28), monomer CQ (0.4), photoactivator EDMAB (0.8), tertiary amine accelerator BHT (0.05), inhibitor
	Filler	Strontium glass (57) Aerosol silica (15)
Experimental composite with BAG	Matrix	combination of 50:50 BisGMA:TEGDMA (28), monomer
	Filler	CQ (0.4), photoactivator EDMAB (0.8), tertiary amine accelerator BHT (0.05), inhibitor Strontium glass (57) Aerosol silica (15)

BAG; bioactive glass, Bis-GMA; bisphenol A glycidyl methacrylate, TEGDMA; triethylene glycol dimethacrylate, CQ; camphoroquinone, EDMAB; 4-dimethylaminobenzoic acid ethyl ether, BHT; butylated hydroxytoluene.

2.3. Preparation of resin composites

The chemical composition of the two resin composites used in this study are shown in Table 1. Briefly, the composite matrix was made with a 50:50 mixture of bisphenol A-glycidyl methacrylate: triethylene glycol dimethacrylate (BisGMA: TEGDMA), 0.4 wt% camphorquinone as a photoinitiator, 0.8 wt% ethyl-4-dimethylamino benzoate as an accelerator, and 0.05 wt% butylated hydroxytoluene as an inhibitor. The resin was mixed with two types of fillers to produce different resin composites: 1) control = 57 wt% strontium glass and 15 wt% aerosol silica (OX-50, Degussa), and 2) BAG-containing experimental composite = 57 wt% strontium glass and 15 wt% BAG65S. Each composite was placed into a transparent acrylic mold (20 mm × 10 mm × 2 mm) and light-cured with a dental curing light having a radiant emittance of 600 mW/cm^2 for 40 s from top and bottom surface. Twenty-two composite disks were made for each of the control and experimental composites, respectively. The surface of each composite disk was polished with 180-, 320-, and 600-grit SIC papers in the same manner as for the dentin specimens.

2.4. Preparation of storage solutions

To investigate the effect of storage solutions on dentin remineralization, 27 mM HCO_3^- Tris-simulated body fluid (SBF) with a pH of 7.4 was prepared according to Tas et al. [23]. Phosphate-buffered saline (PBS, Sigma, St. Louis, Missouri, USA) with a pH of 7.4 was also used (Table 2).

Table 2
The chemical composition of the 27 mM HCO_3^- Tris SBF and PBS solutions.

Storage solution	Composition (amount, g/L)
27 mM HCO_3^- Tris SBF	NaCl (6.547), $NaHCO_3$ (2.268), KCl (0.373), $Na_2HPO_4{\cdot}2H_2O$ (0.178), $MgCl_2{\cdot}6H_2O$ (0.305), $CaCl_2{\cdot}2H_2O$ (0.368), Na_2SO_4 (0.071), and $(CH_2OH)_3CNH_2$ (6.057)
PBS	NaCl (8.0), KCl (0.2), Na_2HPO_4 (1.42), and KH_2PO_4 (0.24)

PBS: phosphate-buffered saline, SBF: simulated body fluid.