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Re-crystallization of bioactive glass mixed with various hardening agents

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ABSTRACT

Bioactive glass (BG) is a potential material for treating dentin hypersensitivity due to its high solubility. In this study, a commercial BG powder (PerioGlas®, PG) mixed with various hardening agents was applied in human dentinal tubule occlusions. X-ray diffraction (XRD), scanning electronic microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) were used to investigate the re-crystallization behavior of human dentin and the dentinal tubule occlusion efficiency by mixing PG with various hardening agents.

The major crystalline phases of PG mixed with phosphoric acid are $CaHPO_4{\cdot}2H_2O$ and $CaH_2P_2O_7$. PG mixed with 20 or 30 wt% phosphoric acid acted as a hardening agent and achieved a dentinal tubule penetration depth of 68–74 μm.

PG mixed with suitable phosphoric acid agents has a short reaction time and a good operability that is feasible for occluding dentinal tubules. PG mixed with hardening agents has a greater potential for treating dentin hypersensitivity than PG without hardening agents.

1. Introduction

In contrast to inactive glass, such as soda-lime glass or borosilicate glass, bioactive glass has well-known in vivo responses, including osteoconductivity, bonding to bone via ion release, and apatite layer formation [1,2]. Therefore, bioactive glass is used for bone reconstitution and tissue engineering [3–10]. Bioactive glass also has a competitive advantage in terms of tooth mineralization [11–14]. In earlier in vitro studies, bioactive glass was reported to induce dentin disc surface mineralization [11,15,16]. These studies suggest that bioactive glass could be helpful in human dentin re-crystallization, and there is the potential for bioactive glass to be used as a filler component of mineralizing restorative material [17]. Unfortunately, bioactive glass's relatively long reaction time reduces its feasibility as a dental re-crystallization agent. The re-crystallization process consists of mineral constituents slowly dissolving or precipitating into the dentin matrix [18]. The first study of the dentin re-crystallization of a bioactive glass was conducted by Wang et al. [19]. In this study, the tooth samples underwent artificial demineralization with EDTA (ethylene-ediaminete-traacetic acid) and were then treated with nanoparticulate bioactive glass and conventional micron-sized material (PerioGlas®, PG). The results showed that the tooth samples treated with nanoparticulate bioactive glass had a significant increase in the mineral content. This result meant that nanoparticulate bioactive glass was a rapid re-crystallization material and confirmed that the particle size and specific surface area of the nanoparticulate bioactive glass were critical factors. Because nanoparticulate bioactive glass has a low mechanical strength, it must be added to a second phase to form a composite to increase the mechanical strength [19]. In addition, investigations of bioactive glass-containing toothpaste show significant reductions in dentin permeability and excellent resistances to acid challenges, which can be beneficial for hypersensitivity and re-mineralization treatments [20,21].

We hypothesize that BG mixed with phosphoric acid will have a positive impact in human dentinal tubule occlusion and reduce the dentin penetration compared to the same treatment without phosphoric acid. However, an empirical study comparing the occlusion efficiency of BG mixed with various hardening agents in human dentinal tubules has not yet been performed.

This study explores the influences of dentin specimens smeared with PerioGlas® (PG) as a substrate, using phosphoric acid as the hardening agent, in the re-crystallization behavior and the human dentinal tubule penetration depth.

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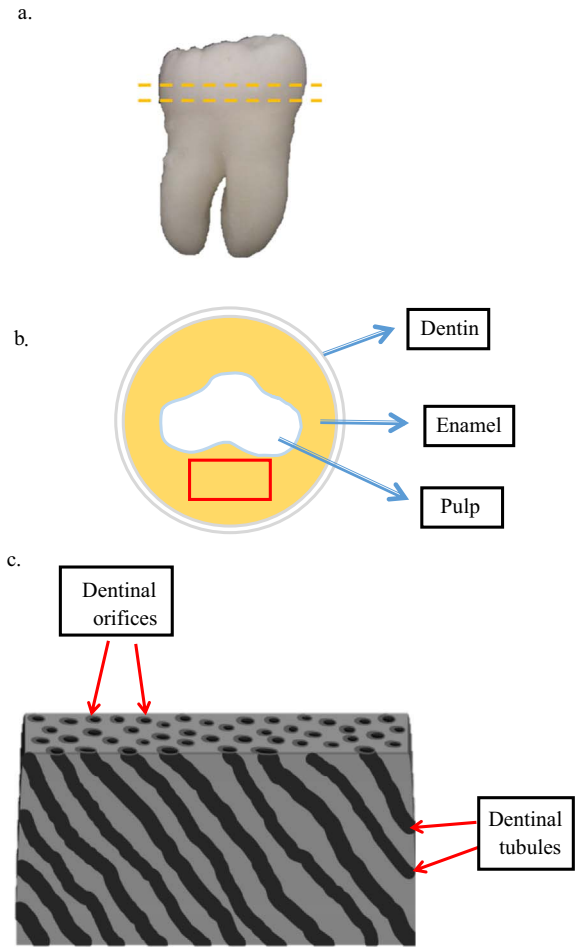

Fig. 1. Schematics of the dentin specimen.

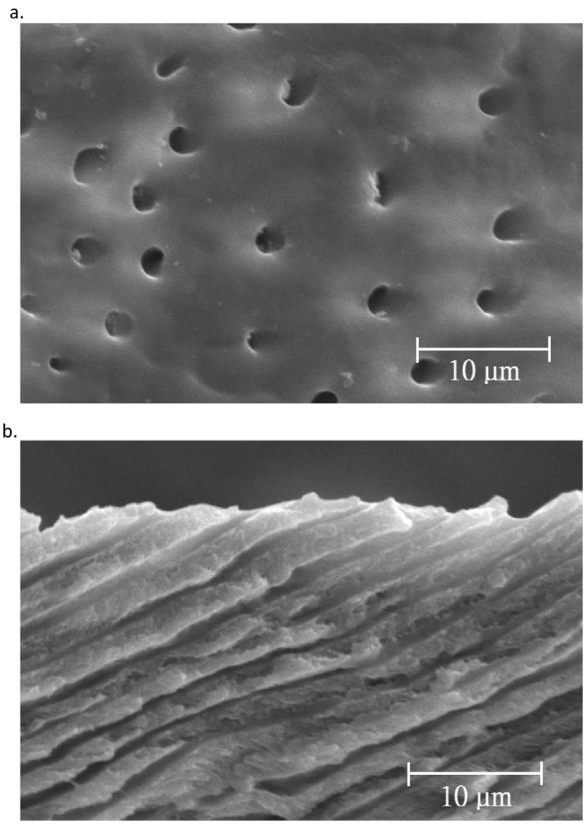

Fig. 2. SEM images of (a) the dentin surface and (b) the dentinal tubules after etching with 37% phosphoric acid to remove the smear layer.

2. Materials and methods

2.1. Dentin specimen pretreatment

In this study, human large molars were used as the dentin specimens. The dentin specimens were extracted from caries-free human large molars of healthy patients for surgical reasons. The teeth were obtained after approval by the Institutional Review Board of Kaohsiung Medical University Hospital, Kaohsiung, Taiwan. Dentin specimen pretreatments were referenced and modified from the study of Gandolfi et al. [22]. The surfaces of the human large molars were cleaned using the ultrasonic vibration method. The enamel on the human large molars' surfaces was removed after cleaning. Using a diamond saw blade, a parallel cut from 2 mm from a point along the tooth neck direction and a cut along the periphery were used to obtain a dentin specimen with a length of 3 mm, as shown in the yellow region in Fig. 1(a). Fig. 1(b) is a top view schematic of the human large molar, and Fig. 1(c) is a schematic of the dentin specimen [23]. The smear layer on the specimen surface was removed using 37% phosphoric acid, and the specimen was then rinsed with excess de-ionized water to remove the residual phosphoric acid. After drying, the dentin specimens were used as the experimental samples. The surface and cross-sectional area of a specimen are shown in Fig. 2 [23].

2.2. PerioGlas® (PG) formulation preparation

With a powder to liquid ratio of 0.05 g to 1ml, commercially available PerioGlas® (PG) powders were homogeneously mixed with various hardening agents, such as 20 wt%, 30 wt% or 40 wt% phosphoric acid (PA) agent, de-ionized water, and phosphate-buffered saline (PBS). The groups subjected to the various testing procedures used in this study are listed in Table 1.

Table 1
Groups of PG mixed with various hardening agents tested in this study.

Testing sample procedure	Abbreviation of group
PG mixed with de-ionized water	PGW
PG mixed with phosphate-buffered saline (PBS)	PGPBS
PG mixed with 20 wt%, 30 wt% or 40 wt% phosphoric acid (PA)	PG20PA, PG30PA, PG40PA

2.3. Dentin specimen preparation

The preparation and scanning electron microscope (SEM) images of the dentin specimens are shown in Figs. 1 and 2. Briefly, the upper dentin surface of each 1-mm thick sample was polished with 800-grit SiC paper for 1 min followed by ultrasonic cleaning for 10 min to obtain standard flat dentin surfaces with open human dentinal tubes. The PG formulations were smeared on the dentin specimen surfaces and incubated at 37 °C with 100% relative humidity, simulating the natural environment of the oral cavity. The occlusion time was 5–10 min. When the occlusion was complete, the dentin specimens were rinsed with a large quantity of de-ionized water for 20 s and submerged into anhydrous ethanol to stop any reactions. After being dried for 24 h, they were mechanically split open for occlusion efficiency analysis.