Effects of resin-based temporary filling materials against dentin demineralization

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This study investigated the *in vitro* anti-demineralization effects of resin-based temporary filling materials containing surface pre-reacted glass-ionomer (S-PRG) filler on dentin. Bovine root dentin specimens with a 3×3 mm experimental surface were divided into four treatment groups: DuraSeal (DU) as a control, S-PRG filler-free temporary material (S0), material containing 10% (S10) and 20% (S20) S-PRG filler. Each material was applied to 3×2 mm of the experimental surface, and the specimens were immersed in 8% methylcellulose gel demineralization system for one week at 37°C. Mineral profiles and integrated mineral loss (IML) of lesions induced on the surface (3×1 mm) adjacent to the materials were computed by transversal microradiography. S10 and S20 yielded thick surface layers and shallow lesion bodies, with significantly lower IML than DU and S0 ($p<0.05$, Tukey's test). These findings indicate that temporary filling resin-based materials containing over 10% of S-PRG filler content have anti-demineralization effects on adjacent dentin.

Keywords: S-PRG filler, Resin-based temporary filling material, Anti-demineralization, Dentin, Transversal microradiography

INTRODUCTION

Temporary resin-based filling materials are frequently used to fill prepared cavities during indirect restorations^{1–6}. These materials are easy to use, both in filling cavities and in removing before final restorations. Studies have suggested, however, that these materials lack sealing ability³ and that bacterial contamination can occur in the cavities^{3,7}.

Surface pre-reacted glass-ionomer (S-PRG) filler release various ions, including fluoride, strontium, sodium, boron, aluminum and silicate ions⁸. In particular, fluoride and strontium ions released by S-PRG filler may alter hydroxyapatite in dentin to fluoridated apatite, fluorapatite or strontium apatite during the course of demineralization and remineralization⁹. In addition, this filler may play important roles in mineral induction¹⁰. S-PRG filler consist of three layers: an outer surface-modifying layer that reinforces the glass-ionomer layer, a middle layer glass-ionomer layer that forms the glass surface, and an inner core consisting of multifunctional glass¹¹. Tooth coating material containing this filler was reported to have anti-demineralization activities against bovine enamel¹² and dentin¹³. Moreover, the composite resin containing this filler may reduce dental bacterial adherence and plaque formation¹⁴.

Temporary filling material with S-PRG filler has shown better marginal sealing ability, with a lower coefficient of thermal expansion, than other temporary

materials on the market¹⁵. The powder of this material contains polymethyl methacrylate (PMMA), 10% S-PRG filler and initiator, and the liquid contains benzyl benzoate, methyl methacrylate (MMA) and initiator. To date, however, the anti-demineralization effects of S-PRG filler containing resin-based temporary filling material have not been investigated. This study was therefore designed to investigate the anti-demineralization effects of the temporary filling materials on dentin *in vitro*. The null hypothesis was that the S-PRG filler containing temporary filling material did not have anti-demineralization effects on adjacent dentin.

MATERIALS AND METHODS

Preparation of dentin specimens

The experimental procedures are outlined in Fig. 1. The preparation procedure was performed by reference to previous reports^{9,13}. Cylindrical dentin roots were obtained from the lower central incisors of 2- to 3-yr-old cattle using a sectioning machine (Isomet Low Speed Saw, Buehler, Lake Bluff, IL, USA). Each cylinder was cut into halves longitudinally with a diamond-coated-wire sectioning machine (Well type 3242, Walter Ebner, Mannheim, Germany). Experimental surfaces, approximately 5×4 mm in size, were prepared by cutting 1 mm each from the buccal and lingual root surfaces with the wire sectioning machine and polished with 2000-grit waterproof abrasive paper (FUJI STAR, Sankyo Rikagaku, Saitama, Japan) to obtain flat surfaces.

The specimens were cleaned ultrasonically for 5 min with deionized water, and a 3×3 mm window on each specimen was made with sticky wax (New Sticky Wax,

GC, Tokyo, Japan).

Treatment of dentin experimental surfaces

Twenty-four dentin specimens were randomly divided into four groups of six each. Each group was treated with one of four different temporary filling materials (Table 1): DuraSeal (DU; Reliance Dental, Alsip, IL, USA; as control), S-PRG filler-free temporary filling material (S0; Shofu, Kyoto, Japan), temporary filling material containing 10% S-PRG filler (S10; PRG PROTECT SEAL, Shofu) and temporary filling material containing 20% S-PRG filler (S20; Shofu). Liquid and powder of each resin-based material were mixed with a small thin brush, and each was applied to a 3×2 mm area of each dentin sample as a thickness of approximately 500 µm (450–550 µm). Samples which had the materials with thickness under 450 µm or over 550 µm were excluded. All treatment procedures were performed by the same individual.

Demineralization

For each of the four experimental groups, we placed six specimens at the bottom of a plastic container (BOTTLE PP (120 mL), AS ONE, Osaka, Japan) and poured 40 mL of 8% methylcellulose gel (Methocel MC, Fluka, Buchs, Switzerland). After 24 h, 70 mL of acid buffer (50 mM acetic acid, 1.5 mM CaCl₂, 0.9 mM KH₂PO₄, pH 5.0) were poured to each container. All containers were kept for one week at 37°C¹³.

Transversal microradiography (TMR)

Three 300 µm thick sections were cut perpendicularly to the experimental surface using the diamond-coated-wire sectioning machine from each specimen. The sections

Fig. 1 Outline of experimental procedures.

Table 1 Materials used

Group	Material	Ingredients	Lot number	Manufacturer
DU	DuraSeal ^a	Powder: PMMA, BPO Liquid: MMA, DBP, DMPT	121411 (Powder) 120911 (Liquid)	Reliance Dental
S0	S-PRG filler-free temporary filling material	Powder: PMMA, Initiator, Others Liquid: Benzyl benzoate, MMA, Initiator, Others	120420 (Powder) 111013 (Liquid)	Shofu
S10	Temporary filling material containing 10% S-PRG filler	Powder: PMMA, S-PRG filler (mean dia. 3.0 µm, Filler contents: 10 wt%), Initiator, Others Liquid: Benzyl benzoate, MMA, Initiator, Others	120420 (Powder) 111013 (Liquid)	Shofu
S20	Temporary filling material containing 20% S-PRG filler	Powder: PMMA, S-PRG filler (mean dia. 3.0 µm, Filler contents: 20 wt%), Initiator, Others Liquid: Benzyl benzoate, MMA, Initiator, Others	120420 (Powder) 111013 (Liquid)	Shofu

PMMA: polymethyl methacrylate; BPO: benzoyl peroxide; MMA: methyl methacrylate; DBP: di-*n*-butyl phthalate; DMPT: *N*, *N*-dimethyl-*p*-toluidine; S-PRG filler: Surface pre-reacted glass-ionomer filler.

^aThe ingredients of DuraSeal were referred to Kawahara *et al.*⁵, and Tanaka *et al.*⁶.

were placed on a perspex holder in a droplet of water and covered with thin polyester sheets to avoid dentin shrinkage¹⁶. Together with an aluminum step wedge of 13 steps, ranging from 0 to 300 μm in thickness, the sections were radiographed on a high-resolution glass film plate (High-Resolution Plate, Konica Minolta, Tokyo, Japan) with a nickel-filtered Cu-K α source operated at 15 mA and 25 kV for 20 min (PW3830, Spectris, Surrey, UK).

The radiographic images were analyzed using a microscope/video camera/microcomputer setup and software (TMR2000, Inspektor Research System, Amsterdam, The Netherlands)¹⁷. The 300 μm area adjacent to the material was analyzed. The output parameters obtained were the mineral content *vs.* depth profiles of the lesions, lesion depth (LD), and integrated mineral loss (IML)^{18,19}.

Fluoride release

Metal molds (15 mm inner diameter, 1 mm thickness, $n=6/\text{group}$) were filled with S10 and S20, at a powder/liquid ratio of 2 g/mL, covered with celluloid strips on the top and bottom, and pressed by hand. The disks were polished lightly with 2000-grit waterproof abrasive paper to remove the resin-rich superficial layer. Each disk was immersed in 5 mL deionized water for one week at 37°C. A 0.3 mL of total ionic strength adjustment buffer (TISAB III, Thermo Electron, Beverly, MA, USA) was added to 3 mL of each eluate. Fluoride concentrations were measured with a combination fluoride electrode (Orion 9609BNWP ionplus Sure-Flow Fluoride, Thermo Fisher Scientific, Waltham, MA, USA) connected to a fluoride-ion meter (720Aplus, Thermo Fisher Scientific). The amount of fluoride released per unit surface area (cm^2) of the disk was computed.

Statistical analysis

IML and LD in the four groups were compared by one-way ANOVA, followed by Tukey's *post hoc* comparison test, and fluoride concentrations in two groups were compared by *t*-tests. All statistical analyses were performed using a statistical software package (SPSS-PC software version 10.1, SPSS Japan, Tokyo, Japan), with $p<0.05$ defined as statistically significant.

RESULTS

Representative TMR images and profiles

Figure 2 shows TMR images (a) and mineral profiles (b) of representative specimens in each experimental group. The TMR images of DU and S0 were very similar and showed an unclear marginal surface layer and a severely demineralized subsurface lesion. In contrast, the TMR images of S10 and S20 were similar and showed distinct surface layers, with greater radio-opacity of the lesion body than observed with DU and S0.

Mineral profiles of DU and S0 showed extremely low and indistinct peaks near the surface and heavy lesion bodies. However, the profiles of S10 and S20 showed well-defined peaks with a high volume percent of minerals in

Fig. 2 Representative TMR images (a) and mineral profiles (b).

The arrow indicates the dentin surface. (a) The TMR images of S10 and S20 were similar and showed clear surfaces (arrows), with the radio-opacity of the lesion bodies significantly greater than those of DU and S0. (b) In the microradiograms, LD was determined as the distance from the surface to the position at which the mineral content is 95% of that of the sound dentin, and IML was defined as the integrated difference between the microradiogram of the sample and that of the sound sample (grey area).

the near-surface region at depths of 5–10 μm and slight lesion bodies in deeper areas.

Average mineral profiles

The averaged mineral profiles of the four groups are shown in Fig. 3. The surface mineral peaks of DU and S0 were quite low, whereas those of S10 and S20 were almost 30–40 vol%. In addition, severely demineralized lesion bodies were observed in DU and S0, whereas mineral volumes in S10 and S20 were higher than those of DU and S0.

IML and LD

Table 2 shows intergroup comparisons of mean IML (vol% $\times\mu\text{m}$) and LD (μm) of the six sections in the four experimental groups. IML of S10 (2,979 $\pm$ 241) and S20 (2,814 $\pm$ 569) were significantly lower than those of DU (5,223 $\pm$ 341) and S0 (5,004 $\pm$ 404) ($p<0.05$), but there was no significant difference between S10 and S20 ($p>0.05$).

LD of DU (291 $\pm$ 28) was similar to that of S0 (272 $\pm$ 56) ($p>0.05$), but statistically higher than those of S10 (203 $\pm$ 29) and S20 (213 $\pm$ 46) ($p<0.05$).

Fluoride release

Experimental disks from specimens containing 10 wt% and 20 wt% of S-PRG filler released 0.55 $\pm$ 0.09 and 0.80 $\pm$ 0.16 $\mu\text{g}/\text{cm}^2$ fluoride ions, respectively, into deionized water. The difference between these two groups was statistically significant ($p<0.05$).

Fig. 3 Average mineral profiles formed in the four groups ($n=6$).

The surface mineral peaks of DU and S0 were extremely low, whereas those of S10 and S20 were almost 30–40 vol%. Severely demineralized lesion bodies were observed in the DU and S0 groups, on the other hand, mineral loss in deeper regions was less in the S10 and S20 groups. S10 and S20 had thick surface layers and slight lesion bodies.

DISCUSSION

The findings of this study rejected our null hypothesis. Briefly, the profiles of S10 and S20 consisted of well-defined peaks with a high volume percent of minerals in the near-surface region and slight lesion bodies in deeper areas. IML of S10 and S20 were significantly lower than those of DU and S0 ($p<0.05$), but there was no significant difference between S10 and S20 ($p>0.05$).

Between the time a tooth is prepared and the final restoration is completed, it is important that the patient be comfortable and that the tooth be protected and stabilized with an adequate temporary restoration²⁰. Many types of temporary restorations, such as hydraulic-setting cements, zinc oxide-eugenol cement and resin temporary restoration, have been used, and in particular, resin-based temporary materials has become popular. Because dentists could remove them easily with explorer, when they started to try-in of the casting on the tooth. However, they could not accomplish good marginal sealing ability, though it was very important to protect prepared cavity walls from bacterial contamination³. Recently developed resin-based temporary material containing S-PRG filler had better marginal sealing ability, with lower expansion in response to thermal stress, than conventional resin-based materials¹⁵.

As the resin-based temporary filling material, DuraSeal has spread at many dental general practitioners. Di-*n*-butyl phthalate (DBP) included in DuraSeal liquid was one of the most commonly used plasticizers^{5,6}, which were considered relatively non-toxic²¹. On the other hand, there was a report that the highest incidence of malformed fetuses occurred after administration with DBP on 13–15 days in rats²². S0, S10 and S20 contained benzyl benzoate instead of DBP. This would be why the manufacturer took the health considerations.

The temporary filling materials containing 10% or 20% S-PRG filler showed statistically less mineral loss at the adjacent dentin against demineralization than DuraSeal and filling material without S-PRG filler. Microradiographic analyses of the S10 and S20 groups showed increased mineral volume percent at the

Table 2 Intergroup comparisons of integrated mineral loss (IML) and lesion depth (LD)

Groups	IML (vol% $\times\mu\text{m}$)	LD (μm)
DU	5,223 (341) ^a	291 (28) ^a
S0	5,004 (404) ^a	272 (56) ^{a,b}
S10	2,979 (241) ^b	203 (29) ^c
S20	2,814 (569) ^b	213 (46) ^{b,c}

Mean ($\pm$ SD), $n=6$

Values with the same superscript letters did not show significant differences between groups.

IML of S10 and S20 were significantly lower than those of DU and S0 ($p<0.05$), but there was no significant difference between S10 and S20 ($p>0.05$).

DU: DuraSeal; S0: S-PRG filler-free; S10: 10% S-PRG filler; S20: 20% S-PRG filler.

surface and overall IML was reduced compared with S0, but these effects were not dose-dependent. Fluoride released by the S-PRG filler likely induces fluoroapatite-like precipitations in lesion bodies, inhibiting further demineralization²³. In contrast to our finding, showing that IML was similar for S10 and S20, a study investigating the amount of fluoride ions released into deionized water over one week by experimental denture base resins containing 10 and 20 wt% S-PRG filler found that the 20 wt% resin released significantly more fluoride ions than the 10 wt% resin²⁴. Also in this study, it was sure that temporary filling material containing 20% S-PRG filler released significantly more fluoride ions than that containing 10% S-PRG filler. However, in this investigation, demineralization was performed eight times of medium as fluoride releasing study. We assumed that fluoride ion released from the both materials in the demineralization medium would be much weakened to the level of same IML achieved. On the other hand, it will be necessary to confirm the anti-demineralization effect of these materials and determine filler volume requirement under the narrow space model simulating leakage.

Although IML differed significantly between S-PRG containing and non-containing materials, LD did not. LD, however, may be controlled by the pH of the solution rather than by the fluoride ion concentration²⁵, suggesting that LD may not be an accurate parameter for measuring demineralization.

Fluoride and strontium ions are released from S-PRG filler containing temporary filling materials and react with hydroxyapatite to form fluoroapatite, strontium apatite and/or fluoridated apatite⁹. Fluoride in saliva has been reported to provide more protection against demineralization than enamel-bound fluoride. Even at low pH, fluoride ion in saliva would increase the saturation degree of fluoroapatite, contributing to the remineralization of teeth^{26,27}. Strontium ion was also involved in the remineralization process of caries dentin²⁸, and the remineralization effect was affected by concentration of fluoride ions when used in conjunction with them²⁹. Moreover, silica and hydroxyapatite nanoparticles have been reported to infiltrate into demineralized dentin, acting as seeds within the collagen matrix³⁰. Therefore, we considered that the materials had anti-demineralization effects. Further investigations for clinical use of these materials are required.

CONCLUSION

The null hypothesis of this study was rejected for IML, indicating that the resin-based temporary filling materials containing 10% or 20% S-PRG filler achieved anti-demineralization effects on adjacent dentin.

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