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% methycellulose 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 restorations1-6. 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 cavities4,7.

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 reports1,6. 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, Suitama, 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˚C.

**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

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**Table 1  Materials used**

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

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.

*The ingredients of DuraSeal were referred to Kawahara et al., and Tanaka et al.
were placed on a perspex holder in a droplet of water and covered with thin polyester sheets to avoid dentin shrinkage. 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-Kα 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). 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).

**Fluoride release**
Metal molds (15 mm inner diameter, 1 mm thickness, n=6/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 (cm²) 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 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%×μm) and LD (μm) of the six sections in the four experimental groups. IML of S10 (2,979±241) and S20 (2,814±569) were significantly lower than those of DU (5,223±341) and S0 (5,004±404) (p<0.05), but there was no significant difference between S10 and S20 (p>0.05).

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

**Fluoride release**

Experimental disks from specimens containing 10 wt% and 20 wt% of S-PRG filler released 0.55±0.09 and 0.80±0.16 μg/cm² fluoride ions, respectively, into deionized water. The difference between these two groups was statistically significant (p<0.05).

**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 restoration. 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 contamination.

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 materials.

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, which were considered relatively non-toxic. 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 rats. 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

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

<table>
<thead>
<tr>
<th>Groups</th>
<th>IML (vol%×μm)</th>
<th>LD (μm)</th>
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<td>DU</td>
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<td>213 (46)</td>
</tr>
</tbody>
</table>

Mean (±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 fluorapatite-like precipitations in lesion bodies, inhibiting further demineralization. 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% resin. Also in this study, it was found 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 weaken 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 concentration, 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 fluorapatite, strontium apatite, and/or fluoridated apatite. 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. Strontium ion was also involved in the remineralization process of caries dentin, and the remineralization effect was affected by concentration of fluoride ions when used in conjunction with them. Moreover, silica and hydroxyapatite nanoparticles have been reported to infiltrate into demineralized dentin, acting as seeds within the collagen matrix. 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.

ACKNOWLEDGMENTS

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