Evaluation of the ion-releasing and recharging abilities of a resin-based fissure sealant containing S-PRG filler

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We evaluated the concentration of fluoride release at the initial stage and after recharging the fluoride from a resin-based sealant containing surface reaction-type pre-reacted glass-ionomer (S-PRG) filler and conventional resin-based sealants. The concentration of other ions released from the sealants in the surrounding distilled water was also examined. At the initial stage, fluoride was released from all sealants. For the sealant containing S-PRG filler, a significantly higher amount of Sr and B ions was detected relative to those from other sealants. After sealant samples were soaked in a sodium fluoride solution, a significant amount of fluoride and other ions was released from the sealant containing S-PRG filler. These findings suggest that sealants containing S-PRG filler enhance the enamel demineralization-inhibiting effect and the remineralization-promoting effect for long-term dental care.

Keywords: Fissure sealants, S-PRG filler, Fluoride release and recharge, Ion release

INTRODUCTION

Pits and fissures of immature permanent tooth are considered to be highly susceptible to the adhesion of microorganisms. Hence, a significant amount of tooth decay can occur at these sites. For preventing occlusal caries, pit and fissure sealants are frequently used for immature permanent teeth. When glass ionomer is used as a material in the sealants, it exhibits the property of fluoride release. Consequently, fluoride release by glass-ionomer cement restorations ensures an anticariogenic effect around the enamel and in adjacent teeth. Koga et al. reported that glass-ionomer sealants had the highest fluoride release and recharge in distilled water among four different types of sealants and that the conventional resin-based sealants did not show fluoride recharge. On the other hand, clinical studies have found the retention of glass-ionomer sealants to be significantly poorer than that of resin-based sealants. Furthermore, the glass-ionomer sealants exhibited significantly higher microleakage scores compared to the resin-based ones. For overcoming these limitations, a resin-based fissure sealant containing surface reaction-type pre-reacted glass-ionomer (S-PRG) filler was recently introduced.

With pre-reacted glass-ionomer (PRG) technology, a glass ionomer phase is formed on glass particles through the aqueous reaction of fluoroaluminosilicate glass with a polycarboxylic acid. The S-PRG filler can be recharged with fluoride. Recently, various materials such as composite resin, denture base resin, root canal sealer, and one-step adhesive have been incorporated into the S-PRG filler. These materials have revealed some aspects of its clinical value. Furthermore, S-PRG filler releases several types of ions (F, Si, and Sr) into either distilled water or lactic acid solution, and composite resin containing S-PRG filler exhibits antibacterial activity by the release of metal ions from the composite.

The purposes of this study were to compare the ability of fluoride release before and after fluoride recharging among the resin-based sealants with S-PRG filler and conventional resin-based sealants, and to examine the concentrations of other released ions from the sealants during the experimental period.

MATERIALS AND METHODS

Test specimens
We used BeautiSealant (BS; Shofu Inc., Kyoto, Japan), Delton FS° (DE; Dentsply Professional, York, PA, USA), and Teethmate F-12.0 (TE; Kuraray Medical Co., Osaka, Japan) as test materials (Table 1). All of these materials contain fluoride and are polymerized by light curing.

Specimens were prepared using a metal ring mold with an internal diameter of 15 mm and a height of 1 mm. The molds with the test materials were held between 2 micro cover glasses with a thickness of 0.12–0.17 mm. After irradiation on both sides (for 90 seconds at one side) using a visible light-curing unit (Gripilight II; Shofu Inc.), the specimens were gently polished with #600 waterproof abrasive paper (Sankyo-Rikagaku Co. Ltd., Saitama, Japan).

Fluoride release and recharging
Figure 1 shows the experimental procedure. Each sealant specimen was placed in 5 mL of distilled water (DW) for 15 days. Afterwards, the specimens were soaked in sodium fluoride solution (1,000 ppm and 9,000 ppm fluoride were used twice for each sample) for 5 minutes to recharge the fluoride, and the specimens were placed in 5 mL of DW again for 3 days. Then, the specimens were placed in 5 mL of DW for 2 days. Before and after the fluoride-release experiments were performed, the specimens were rinsed sufficiently with DW.

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**Measurement of released fluoride**

During the fluoride-release experiments, the fluoride-ion concentration of the DW was measured daily; 5 specimens were prepared for each test material. We added 0.5 mL of total ionic strength adjustment buffer (TISAB III; Thermo Fisher Scientific Inc., Waltham, MA, USA) to the DW and inserted a combination fluoride electrode (Orion 9609BN; Thermo Scientific Inc.) connected to a fluoride-ion meter (720A; Thermo Scientific Inc.) for measuring the released fluoride. After each measurement, the solution was discarded and replaced with fresh DW.

**Elemental analysis**

We performed an elemental analysis of the ions released from each specimen by using inductively coupled plasma atomic emission spectroscopy (ICP-AES; ICPS-8000; Shimadzu Co., Kyoto, Japan). This analysis was performed daily during the entire experimental period. The analysis was conducted after preparing calibration curves corresponding to each element (standard solution concentration; Na, Sr, B, Al, Si, and S: 0, 0.5, 5.0, and 20.0 ppm, respectively).

**Statistical analysis**

Differences in the daily released fluoride among the sealants (i.e., BS, DE, and TE) were tested for significance by one-way analysis of variance (ANOVA) at the $p<0.05$ level. When ANOVA showed a significant difference among the sealants, Tukey’s honestly significant difference (HSD) post-hoc test was used to identify the group difference. All analyses were performed with SPSS 15.0J for Windows (SPSS Japan, Inc., Tokyo, Japan).

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**Fig. 1** The experimental procedure of fluoride release and recharging.

Each sealant specimen was placed in DW for 15 days. Afterwards, the specimens were soaked in sodium fluoride solution (1,000 ppm and 9,000 ppm fluoride were used twice) for 5 minutes, and the specimens were placed in DW for 3 days. Then, the specimens were placed in DW for 2 days. The fluoride-ion concentration of the DW was measured daily ($n=5$).

**Table 1** Pit and fissure sealants used in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Main composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>BeautiSealant</td>
<td>S-PRG filler (40wt%), UDMA, TEGDMA</td>
<td>Shofu Inc., Kyoto, Japan</td>
</tr>
<tr>
<td>Delton FS⁺</td>
<td>Low viscosity monomers, TEGDMA, Bis-GMA, sodium fluoride</td>
<td>Dentsply Professional, York, PA, USA</td>
</tr>
<tr>
<td>Teethmate F-1₂₀</td>
<td>TEGDMA, HEMA, MDP, MMA-MF copolymer</td>
<td>Kuraray Medical Co., Osaka, Japan</td>
</tr>
</tbody>
</table>

Abbreviations: S-PRG: surface reaction-type pre-reacted glass-ionomer; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; Bis-GMA: bisphenol A-diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; MMA: methyl methacrylate; MF: methacryloyl fluoride

**Table 2** Means (ppm)±SD of fluoride released from pit and fissure sealants

<table>
<thead>
<tr>
<th>Materials</th>
<th>day 1</th>
<th>day 2</th>
<th>day 16</th>
<th>day 19</th>
<th>day 22</th>
<th>day 25</th>
</tr>
</thead>
<tbody>
<tr>
<td>BS</td>
<td>12.60±1.19a</td>
<td>3.22±0.58a</td>
<td>1.10±0.14a</td>
<td>1.10±0.31a</td>
<td>11.64±1.17a</td>
<td>15.84±3.25a</td>
</tr>
<tr>
<td>DE</td>
<td>45.80±5.46b</td>
<td>1.82±0.25b</td>
<td>0.79±0.09b</td>
<td>0.72±0.04b</td>
<td>3.17±0.39b</td>
<td>4.24±0.35b</td>
</tr>
<tr>
<td>TE</td>
<td>4.66±0.82a</td>
<td>2.60±0.45b</td>
<td>0.70±0.11b</td>
<td>0.65±0.18b</td>
<td>0.83±0.12c</td>
<td>0.96±0.24c</td>
</tr>
</tbody>
</table>

$n=5$

Values with different superscript letters denote statistically significant differences at $p<0.05$. 
RESULTS

Fluoride release
Table 2 shows the amount of fluoride released from each sealant specimen. On day 1, the fluoride ions were released into DW from the specimens at levels of 12.60, 45.80, and 4.66 ppm for the BS, DE, and TE samples, respectively. Significant differences were found among the sealants \((p<0.001)\). On day 2, the fluoride release decreased for all sealants. From days 3 to 15, the sealants showed consistently low levels of fluoride release, with averages of 0.92, 0.60, and 1.29 ppm for the BS, DE, and TE samples, respectively. On days 16 and 19, after the specimens were soaked in 1,000 ppm fluoride solution, fluoride release was slightly increased for the BS samples, and significant differences were found relative to the TE \((p=0.01\) for each day) and DE \((day\ 16: \ p=0.035,\ day\ 19: \ p=0.017)\) samples. On days 22 and 25, after the specimens were soaked in 9,000 ppm fluoride solution, fluoride release was significantly increased for the BS samples, and significant differences in fluoride release were found for the BS samples compared to other materials \((p<0.001\ for\ both\ days)\). The DE samples also showed some fluoride release, whereas the TE sample showed only low levels. After the samples were recharged with fluoride, the fluoride release from the BS and DE samples returned to the former concentration 2 days later.

Ion release
Figure 2 shows the amount of various ions released from each sealant specimen. At the initial stage, Sr, Na, B, and Si ions were detected in the BS (Fig. 2(a)). In particular, the release of Sr and B from the BS material was significantly increased relative to that of other materials. Remarkably, although a high concentration of Na ions was detected in the DE material (Fig. 2(b)), release of ions was not observed for the TE samples (Fig. 2(c)).

On days 22 and 25, after the specimens were soaked in sodium fluoride solution (9,000 ppm fluoride), the release of Na, Sr, Al, Si, and B ions was significantly increased in the BS (Fig. 2(a)). Although the Na ion concentration increased for the DE material after recharging with fluoride (Fig. 2(b)), release of ions was notably not found for the TE specimens (Fig. 2(c)).

DISCUSSION

Sealants have been developed to protect pits and fissures from caries by preventing the impaction of food and bacteria\(^{21}\). In addition, fluoride released from sealants prevents demineralization in the adjacent tooth structure\(^{22,23}\). Thus, sealants and topical fluorides are widely used in public dental programs as a primary prevention against dental caries\(^{24,25}\).

We investigated the concentration of fluoride release at the initial stage and after recharging the fluoride from a resin-based sealant with S-PRG filler. A previous study reported that denture base resin containing S-PRG filler

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We investigated the concentration of fluoride release at the initial stage and after recharging the fluoride from a resin-based sealant with S-PRG filler. A previous study reported that denture base resin containing S-PRG filler
released a great amount of fluoride on day 1, and the release of fluoride rapidly decreased with an increase in the immersion period by day 15\(^{14}\). Therefore, initial fluoride release might be decreased until 15 days. Thus, we soaked the sealant specimens in DW for 15 days to estimate an effect of fluoride recharging precisely.

According to Koga et al.\(^ {3,9} \), conventional resin-based sealants do not show the property of fluoride recharge. In this study, a considerable amount of fluoride was released from the resin-based sealant containing S-PRG filler after a recharge with a high concentration of fluoride. Even when a low concentration of fluoride was used for recharging, some amount of fluoride was released from the sealant with the S-PRG filler. Although the amount of fluoride released from the glass-ionomer cement in distilled water at the initial stage was approximately 10-fold higher than that of the sealant containing S-PRG filler (data not shown), the mechanisms underlying fluoride release and recharge abilities may be similar between the S-PRG filler and the glass ionomer.

The results of the present study indicate that the sealant containing S-PRG filler will be more effective than the other tested resin-based sealants in preventing dental caries when combined with daily tooth brushing using a fluoride dentifrice as well as with periodic dental office care that includes a professionally applied topical high concentration fluoride treatment.

Considerably higher amounts of Sr and B ions were released from the resin-based sealant containing S-PRG filler in comparison with those released from the other sealants. Although the mechanism of ion release from S-PRG filler is not completely understood, it is believed that the presence of a glass ionomer phase around the glass core of the filler is related to the ion release. Among the ions detected in this study, Sr is considered to play an important role in tooth mineralization\(^ {26, 27} \). A recently published study reported that Sr enhances enamel remineralization when used in conjunction with fluoride\(^ {29} \). Moreover, B, F, and Sr ions released from S-PRG filler may have antibacterial activity\(^ {29} \), but further analysis of these properties needs to be performed. Cattani-Lorente et al. reported that Na is present in the S-PRG filler composition and is released with Al\(^ {30} \). The release of Al is associated with enhanced F release, leading to increases in the formation of aluminofluoro complexes\(^ {31} \). In this study, on day 1, considerable amounts of fluoride and Na were released from the DE. Similarly, on days 22 and 25, considerable amounts of fluoride and Na were released from the BS. Thus, Na release might be increased in relation to fluoride release.

In conclusion, we found that the resin-based sealant containing S-PRG filler has superior abilities of fluoride recharge and of the release of various ions compared to the conventional sealants. The results of our study suggest that the sealant with S-PRG filler enhances the enamel demineralization-inhibiting effect and the remineralization-promoting effect possessed by fluoride and other ions.

REFERENCES


