Prevention of acidic attack on tooth enamel surfaces using polishing paste containing ion-releasing filler

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The purpose of this study was to investigate the ability of polishing paste containing surface pre-reacted glass-ionomer (S-PRG) filler to prevent acidic attack on tooth enamel surfaces. Resin composites were filled in the standardized cavities and finished with silicon carbide paper. These specimens were divided into three groups: the unpolished “control” group, the “PRG” group polished with S-PRG paste, and the “DDP” group polished with diamond-containing polishing paste. Following polishing, the specimens were immersed in a lactic acid buffer solution for 28 days. Optical coherence tomography (OCT) signals were measured to obtain the signal intensity and width at 1/e at selected locations on the enamel surface adjacent to the restoration. Although signal intensity significantly increased in all groups, widths at 1/e did not change significantly in the PRG group. For both the control and DDP groups, signal intensity and width at 1/e increased and decreased over time, respectively.

Keywords: Polishing paste, Ion-releasing filler, Enamel erosion, OCT

INTRODUCTION

Direct restorations of teeth are now widely employed to restore both anterior and posterior lesions because of recent improvements in the mechanical properties and adhesive performance of such restorations. The final step in a direct restoration procedure involves finishing and polishing the resin composite surface to minimize gingival irritation, surface stains, and bacterial adhesion. In particular, the surface of resin composites has been shown to be susceptible to bacterial adhesion, which is one of the stages of biofilm formation in the marginal areas of restorations. Cariogenic biofilm formation is an issue of aetiological importance in secondary caries, and is one of the main reasons for the failure of resin composite restorations. Therefore, a smooth and glossy surface is required in the restoration, which is achieved by finishing and polishing procedures completed with the use of appropriate equipment.

Although resin composite restorations are now viable restoration options, failed restoration replacements remain a major issue for dentists, who may spend a considerable amount of time replacing the restorations or repeating the procedure. Secondary caries are the most common late complication in resin composite restorations. Such lesions are typically found adjacent to the restoration and were traditionally thought to be associated with the gap between the tooth and the restoration. Gap size and mechanical loading have been shown to be associated with lesion severity in oral condition models; however, the results of such models do not correspond exactly to the results obtained from in situ studies using restorative materials. Reducing the severity of lesion formation may lead to the longevity of the restorations; certain materials with remineralization potential could be used to achieve such effects.

Surface pre-reacted glass-ionomer (S-PRG) fillers are prepared using an acid-base reaction (with traditional glass ionomer cement) between fluoroaluminosilicate glass (base) and a polyacrylic acid in the presence of water; the preliminary product is a stable glass ionomer phase within the glass particles. A coating material which contains S-PRG filler has been reported to suppress demineralization of enamel and to inhibit plaque formation possibly by releasing fluoride, borate, strontium, silica, sodium, and aluminum ions. Indeed, compared to fluoroaluminosilicate glass filler materials, resin composites containing S-PRG filler release many types of ion at higher rates; they also have a modulation effect on oral acidity that causes the pH of the surrounding environment to become weakly alkaline upon contact with water or acidic solutions.

In order to manage the demineralization process of enamel with minimal intervention, various diagnostic techniques have been introduced to identify early mineral changes. For example, optical coherence tomography (OCT) has been applied to determine tooth mineralization with the advantage of being non-invasive and avoiding radiation-induced tissue damage. OCT observations can visualize the internal structure of demineralized lesions without destroying the specimen. Tooth enamel is composed of hydroxyapatite crystal, which is known to exhibit birefringence; both de- and re-mineralization processes alter the birefringence of tooth enamel with an anisotropic refractive index. The underlying principles of OCT clearly differentiate it from transverse microradiography, which has previously been thought of as the gold standard for detecting the mineral
Figure 1: Experimental treatments used on the enamel specimens restored with resin composites. PRG: surface reaction-type pre-reacted glass-ionomer (PRG Compoglass; Shofu) paste; DDP: diamond-containing (DirectDia) paste.

MATERIALS AND METHODS

Specimen preparation

Eighteen extracted bovine incisors were used in this study. Standardized cylindrical cavities (2 mm in diameter, 2 mm deep), for which all margins were in the dentin of the tooth, were prepared with a diamond point (202CR; ISO 021; Shofu, Kyoto, Japan) fixed within a high-speed handpiece (TwinPower Turbine P; J. Morita Mfg., Kyoto, Japan) with copious air-water spray. A super-fine-grained diamond point (SF202CR; Shofu) was used for finishing each preparation, and a new bur was changed after every six preparations.

The cavity walls were treated with self-etching primer (Clearfil SE Bond 2; Kuraray Noritake Dental, Tokyo, Japan) for 20 s, a bonding agent was applied, and a LED (light-emitting diode) curing unit (Pencure; J. Morita Mfg.) was used and irradiated for 10 s. A light-cured resin composite (Clearfil AP-X; shade A2, Kuraray Noritake Dental) was then condensed into the prepared cavity and a polyester transparent strip was placed over the sample. After light-curing for 30 s, the restorations were finished with wet SiC (silicon carbide) paper (Fuji Star Type DDC; Sankyo Rikagaku, Saitama, Japan) starting with #600-grit to #2,000-grit to standardize flat surface. After the polishing procedures were completed, the specimens were rinsed with tap water and air dried.

Finally, the specimens were divided into three groups as below (Fig. 1):

1) Control group: the specimens’ surfaces were not polished.
2) PRG group: the specimens’ surfaces were polished with a polishing paste containing S-PRG (PRG Compoglass; Shofu) using Super-Snap Buff Mini-Disks (Shofu) for 30 s.
3) DDP group: the specimens’ surfaces were polished with a polishing paste containing diamond particles (DirectDia Paste; Shofu) using Super-Snap Buff Mini-Disks for 30 s.

All polishing procedures were performed using a micromotor handpiece (Torqtech CA-DC; J. Morita Mfg.) with 5,000 rpm and a constant pressure of 0.5 N monitored by a digital balance (AT200; Mettler-Toledo, Greifensee, Switzerland). All restorations and polishing procedures were conducted by one operator.

For each group, the prepared specimens (n=6) were immersed in an undersaturated 0.1 M lactic acid buffer solution (pH 4.75, 0.75 mM CaCl$_2$•2H$_2$O, and 0.45 mM KH$_2$PO$_4$) for 10 min, and then were placed in artificial saliva (pH 7.0, 14.4 mM NaCl, 16.1 mM KCl, 0.3 mM MgCl$_2$•6H$_2$O, 2.0 mM K$_2$HPO$_4$, 1.0 mM CaCl$_2$•2H$_2$O, and 0.10 g/100 mL sodium carboxymethyl cellulose). These procedures were conducted twice daily (with a time interval of 10 h) over the 28-day test period. Between acid treatments, the specimens were stored in artificial saliva at 37°C.
**OCT measurements**

A time-domain OCT imaging system (J. Morita Tokyo Mfg., Saitama, Japan) was used in this study. We used superluminescent diodes (DL-CS3184B; DensLight Semiconductors, Singapore) as the light source for the time-domain OCT. Superluminescent diodes have a spectral bandwidth midpoint of 1,310 nm, a spectral bandwidth of 40 nm, and an optical output of 7.5 mW. The focused beam was coupled to a single-mode fiber-optic Michelson interferometer. In a fiber-optic Michelson interferometer, a focused beam was split into two, one was focused onto the specimen and scans the specimen in an X-Y longitudinal. The other beam was bounced off a reference mirror. Reflected light from the specimen was combined with reflected light from the reference mirror. These signals were amplified and demodulated by an amplifier. After that, the voltage from the lock-in amplifier was converted to a digital signal with a data acquisition board, before being processed using analysis software (Origin 9; OriginLab, Northampton, MA, USA).

The scanning probe of the time-domain OCT device was fixed 2 mm from the specimen surface. The scanning light beam was set perpendicular to the specimen surface, and changes in the specimen were determined based on the signal intensity values from the OCT images. The width between points, where the signal intensity decreased to a value of 1/e² and corresponded precisely to the peak signal intensity of the scanned profile line, was calculated. A width of 1/e² is equivalent to the distance between the two points where the intensity falls to 13.5% of the maximum value. Peak intensities and the widths at 1/e² were obtained for six points around the restoration of the maximum value. Peak intensities and the widths at 1/e² were obtained for six points around the restoration in each specimen, and these obtained data were averaged (mean values). Six specimens were examined in each treatment group. Measurements were taken at 23±1°C and 50±5% relative humidity. Data were collected before the polishing procedures and on days 0 (baseline), 1, 7, 14, 21, and 28 during the treatment period.

**Statistical analysis**

Statistical analyses were performed using a software (SigmaPlot 13; Systat Software, Chicago, IL, USA). First, the data for each group were tested for homogeneity of variance (Bartlett’s test) and normal distribution (SigmaPlot 13; Systat Software, Chicago, IL, USA). If those of the other groups.

| Polishing paste | Code | Main components | Manufacturer | Lot No. |
|-----------------|------|-----------------|--------------|---------|
| PRG Compogloss  | PRG  | glycerin, diamond powder (1 µm), aluminum oxide, S-PRG filler, purified water, viscosity agent | Shofu, Kyoto, Japan | 91701 |
| DirectDia paste | DDP  | glycerin, diamond powder (3 µm), viscosity agent, pH regulator, colorant | Shofu | 1217064 |

PRG: pre-reacted glass-ionomer; S-PRG: surface reaction-type pre-reacted glass-ionomer
DISCUSSION

In the present study, we assessed the ability of polishing paste containing S-PRG filler to prevent acidic attack on the enamel substrate around the margin of resin composite restorations. To this end, we used bovine teeth rather than human teeth; although human teeth are the ideal specimens in in vitro studies, bovine teeth are commonly used as human-teeth substitutes. While the comparative properties of human and bovine tooth tissues remains under consideration, current consensus recommends the continued use of bovine enamel as a substitute for human enamel\(^1\). One of the advantages of bovine teeth are their large size, which makes fabricating test specimens much simpler. Previous studies have compared the use of bovine and human teeth for research purposes. For example, one review concluded that bovine teeth can be considered as a suitable substitute for human teeth during in vitro bond strength tests of adhesive systems\(^2\). Another study compared the acid-mediated dissolution of human and bovine enamel in vitro under conditions representative of erosion, and it was concluded that bovine enamel can be used as a substitute for human enamel in erosion studies over moderate exposure periods\(^3\). Nevertheless, when interpreting the results of studies with bovine teeth, the differences in their chemical and physical properties relative to human teeth must be taken into account.

In our study, we examined the inhibitory effect...
of polishing paste containing S-PRG filler on enamel demineralization when the restored specimens were immersed in a pH 4.75 lactic acid buffer solution. Acid production by oral bacteria dissolves the mineral phases of the enamel surface and facilitates the formation of tooth caries; lactic acid is the major acid that causes caries, hence our choice of buffer solution, followed by acetic and propionic acids\(^2^1\). Given the concept of "critical pH", the most important method for relating changes in pH to caries activity across treatment groups is to classify values according to a hypothetical critical decalcification pH level of 5.0\(^2^2\).

When light is incident on a tooth substrate, four phenomena occur: interaction between the tooth and light flux, specular reflection at the surface, diffuse light reflection at the surface, and absorption and scattering of the flux within the tooth\(^2^3\). Therefore, accurate determination of optical constants from OCT image data is vital because these optical constants describe the optical field-sample interaction\(^2^4\). Intensity is generated by two mechanisms in OCT images: (1) the intrinsic birefringence of the enamel rotates the phase angle of the incident light between two orthogonal axes as the light propagates through the enamel without changing the degree of polarization and (2) depolarization scrambling from scattering reduces the degree of polarization. The second mechanism is used to measure the severity of demineralization\(^2^5\). Therefore, areas of demineralization show increased reflectivity in OCT images, which results in higher peak intensities. This observation is contingent on the inverse relationship between the intensity value of light backscattering and the mineral content within the enamel surface\(^2^6\). Reflectivity from the lesion area can be directly determined and then used as a measure of lesion severity\(^2^7\): shallow lesions show a loss of penetration depth in OCT images, which is correlated with mineral loss and can be detected using LSM. In the present study, following 21 days of treatment, both the control and DDP groups showed a strong reflection on the enamel surface due to the strong depolarization caused by light scattering at the acid-roughened enamel surface. A similar increase in the intensity of reflection at the enamel surface was observed in the DDP group from Day 21 to 28, with the signal showing backscatter along the depth beyond the enamel surface. This was probably the cause of the masking subsurface scattering and creation of speckles in our obtained images.

When attempting to measure lesion depth and severity, the high dynamic range of reflectance makes it difficult to define the cutoff point when using OCT to measure demineralization. Furthermore, determination of signal fall off is complicated by the exponential attenuation of light as it propagates to the tooth substrate. Previous studies proposed that the depth at which the peak signal intensity decreases by \(1/e^2\) can be used to determine the cutoff signal intensity\(^2^8\). In our study, the \(1/e^2\) values in the DDP and control groups decreased as the treatment period progressed, but no change in \(1/e^2\) was observed in the PRG group. Furthermore, lower signal intensity with a wider \(1/e^2\) value was recorded in the PRG group relative to the other groups on Day 28. These results show that in PRG group the OCT signal was generated by light that traveled over a longer pathway than that which was observed in the DDP and control.

![Fig. 2 LSM images of the resin composite/enamel interface of specimen surfaces subjected to the three experimental treatments.](image-url)

**Control group:** unpolished; **PRG group:** polished with surface reaction-type pre-reacted glass-ionomer (PRG Compogloss) paste; **DDP group:** polished with diamond-containing (DirectDia) paste.

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| Control group | PRG group | DDP group |
|--------------|-----------|-----------|
| Resin composite | Enamel | Resin composite | Enamel | Resin composite | Enamel |

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\(^2^1\) Dent Mater J 2021; : –
groups. It is possible that the presence of ions from the PRG filler in the polishing paste may have strengthened the enamel surface and protected it from acid attack\(^3\), ultimately leading to changes in the optical properties of PRG specimens\(^4\). The LSM observations of the PRG group revealed the relatively smoother enamel surfaces of specimens, and these results were in agreement with the data we collected from specific measurements. We therefore suggest that when S-PRG filler is incorporated into polishing paste formulations, the ions released from the glass-ionomer phase contribute to the suppression of tooth surface demineralization.

The presence of antibacterial ions, such as silver, magnesium, strontium, zinc, and gallium ions, has an inhibitory effect on bacterial growth\(^5\). Therefore, most resin composites without such ions do not have the antibacterial characteristics of glass-ionomer cement, which is due to fluoride release\(^6\), and therefore have no protective effect against biofilm formation or secondary caries. To reduce the risk of tooth decay around resin composite restorations, materials that enhance remineralization and protect against acidic attack can be used\(^7\). The ability of fluoride to prevent acidic attack is based on its potential to precipitate fluorapatite with lower solubility\(^8\). In the present study, the S-PRG filler acted as fluoride-releasing material; it released aluminum, borate, fluoride, sodium, silica, and strontium ions, among which strontium and boron ions are known to inhibit the growth of oral bacteria\(^9\). In addition, Uo et al. reported that the local structure of strontium in enamel after immersion in S-PRG filler eluate was similar to that of strontium-containing hydroxyapatite\(^10\). The combination of strontium and fluoride ions improves the crystallinity of carbonated hydroxyapatite\(^11\). Therefore, fluoride and strontium ions released from the S-PRG filler in the polishing paste used here may have contributed to improving the remineralization of the enamel substrate and protecting it from acid erosion. Fluoride is also known to facilitate the conversion of hydroxyapatite to fluorapatite or fluoridated apatite on the demineralized root dentin surface. It has been reported that extra fluoride application enables remineralization of a demineralized lesion even in the absence of fluoride in dental materials\(^12\). Indeed, uptake of fluoride from the outer tooth surface is important for tooth remineralization and prevention of acidic attack. Thus, the combination of fluoride and strontium ions can inhibit the dissolution of hydroxyapatite by the acids produced by cariogenic bacteria. Furthermore, strontium can act as a substitute for calcium during precipitate formation and has a synergistic caries-control effect with fluoride\(^13\).

Among the other ions released from the S-PRG fillers, silica may also play an important role in mineralization of the tooth substrate by promoting hydroxyapatite formation through the induction hydroxyapatite nucleation\(^14\). In an aqueous environment, there is a rapid exchange of sodium ions (Na\(^+\)) with hydrogen cations (H\(^+\) or H\(_3\)O\(^+\)) from the solution. There is also a loss of soluble silica in the form of Si(OH)\(_4\) to the solution due to the breaking of Si-O-Si bonds and formation of Si-OH (silanol) at the glass-solution interface. Condensation and repolymerization of a SiO\(_2\)-rich surface is eliminated by alkali and alkaline earth cations. When Ca\(^{2+}\) and PO\(_4\)\(^{3-}\) groups migrate to the S-PRG fillers through the SiO\(_2\)-rich surface, a CaO-P\(_2\)O\(_5\)-rich film forms on the tooth surface and then crystallizes into hydroxy carbonate apatite. It has been suggested that the chemical reactions that promote apatite formation may also help protect against demineralization in early carious lesions\(^15\). In future studies, it will be necessary to conduct further research into the relationships among the content of dental materials and the diffusion or release of fluoride.

In a previous study, the clinical performance of refurbished resin composite restorations was compared to that of untreated restorations, and it was concluded that no significant differences existed in the survival curves of the refurbished and untreated groups\(^16\). The main reasons for this observation were secondary caries and fracture of the tooth, neither of which are failures of the materials themselves but are rather related to the operator’s and patient’s characteristics. In contrast, we have shown that by using polishing paste containing S-PRG filler not only can the luster and marginal adaptation of defective resin composite restorations be improved but also the enamel margins become protected against acidic attack.

**CONCLUSION**

Based on our results, we conclude that polishing paste containing S-PRG filler appears to inhibit demineralization of the enamel around resin composite restorations. However, because demineralization of the enamel structure in the oral environment may be different to that in the conditions tested here, further in vivo research will be required to prove the clinical effectiveness of S-PRG filler in polishing paste.

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**CONFLICT OF INTEREST**

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is mentioned in this article.

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