Effect of Resin Coating on Dentin Bonding of Resin Cement in Class II Cavities

Shamim SULTANA¹, Toru NIKAIĐO¹, Khairul MATIN¹², Miwako OGATA¹, Richard M. FOXTON³ and Junji TAGAMI¹²
¹Cariology and Operative Dentistry, Department of Restorative Sciences, Graduate School, Tokyo Medical and Dental University, 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113-8549, Japan
²Center of Excellence Program for Frontier Research on Molecular Destruction and Reconstruction of Tooth and Bone, Tokyo Medical and Dental University, 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113-8549, Japan
³King's College London Dental Institute at Guy's, King's College and St. Thomas' Hospitals, London, UK

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This study was designed to evaluate the efficacy of resin coating on the regional microtensile bond strength (MTBS) of a resin cement to the dentin walls of Class II cavities. Twenty mesio-occlusal cavities were prepared in human molars. In 10 cavities, a resin coating consisting of a self-etching primer bonding system, Clearfil SE Bond, and a low-viscosity microfilmed resin, Protect Liner F, was applied. The other 10 teeth served as a non-coating group. After impression taking and temporization, they were kept in water for one day. Composite inlays were then cemented with a dual-cure resin cement, Panavia F 2.0, and stored in water for one day. Thereafter, MTBSs were measured. Two-way ANOVA (p<0.05) revealed that the MTBS of resin cement to dentin was influenced by resin coating, but not by regional difference. In conclusion, application of a resin coating to the dentin surface significantly improved the MTBS in indirect restorations.

Keywords: Regional bond strength, Indirect restoration, Dual-cure resin cement

INTRODUCTION

The indirect fabrication of composites is widely used not only for the esthetic treatment of posterior and anterior teeth, but also to conserve tooth structure in the case of large defects. Direct composite restorations are preferred to indirect composite restorations because they require only minimal intervention during cavity preparation — even in posterior restorations. However, polymerization shrinkage of direct composites under confined conditions generates stress at the tooth-restoration interface, which may lead to gap formation, postoperative sensitivity, and secondary caries. Unfortunately, current resin cements do not always provide good bonding performance to dentin compared with dentin bonding systems for direct resin composites.

To overcome the lackluster bonding performance to dentin, a resin coating technique was developed in the early 1990s. In this technique, a hybrid layer and a tight sealing film are produced on the dentin surface with a dentin bonding system and a low-viscosity microfilmed resin. It enables coverage and protection of the prepared dentin immediately after cavity preparation. Therefore, this technique has the potential to minimize pulp irritation and postoperative sensitivity. Further, a resin coating can provide a resin cement with high dentin bond strength and good interfacial adaptation of composite inlays. Therefore, the resin coating technique is a key to achieving minimal intervention with indirect resin composites.

Dentin moisture, as well as regional difference, are important factors that may affect dentin bonding. The bond strength of the cavity floor dentin of Class I³⁴ and Class II³⁴ restorations have been evaluated in direct composite restorations. There have also been some studies on the relationship between resin coating and resin cement bond strength. However, there is little information on the regional bond strength of resin cement to resin-coated dentin. Therefore, the purpose of this study was to evaluate the efficacy of a resin coating on the regional (i.e., occlusal and proximal) microtensile bond strength (MTBS) of a resin cement to the dentin walls of Class II (MO) cavities.

MATERIALS AND METHODS

Specimen preparation

Specimen preparation is illustrated in Fig. 1. Twenty non-caries human third molars were used for this study. Mesio-occlusal (MO) cavities with slightly rounded internal line angles were prepared using a regular-grit diamond bur (207CR, Shofu, Kyoto, Japan), and cavity surfaces were finished with a superfine diamond bur (SF 207 CR, Shofu) mounted in an air turbine handpiece under water coolant. Dimensions of the occlusal cavities were approximately 4 mm wide and 2.5 mm high. The mesial gingival margin of the proximal cavity was located 1.5 mm above the cementoenamel junction. Height of the proximal cavities depended on the crown height.

The prepared teeth were randomly divided into two groups. For one group, the cavities were coated
Impression taking

1 day in water

2.5 mm

MO cavity

1.5 mm

Resin coating

Resin cement

Temporary material

Indirect inlay

Fig. 1 Specimen preparation for microtensile bond strength test.

Table 1 Materials, batch numbers, and compositions

<table>
<thead>
<tr>
<th>Material</th>
<th>Batch No.</th>
<th>Components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dentin bonding system</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>00565B</td>
<td>Primer: MDP, HEMA, hydrophilic dimethacrylates, water, photoinitiator</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Bond: MDP, HEMA, Bis-GMA, photoinitiator, microfiller, functional monomer</td>
</tr>
<tr>
<td>Low-viscosity microfilled resin</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Protect Liner F</td>
<td>0060A</td>
<td>Bis-GMA, TEGDMA, microfillers, photoinitiator</td>
</tr>
<tr>
<td>Indirect resin composite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Estenia (DA3)</td>
<td>00239A</td>
<td>Hydrophobic methacrylates, 92 wt% (82 vol%) fillers. Ultra-fine fillers (0.02 μm particle size) loaded into a microfilled (2 μm particle size) resin matrix.</td>
</tr>
<tr>
<td>Resin cement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Panavia F 2.0</td>
<td>011120</td>
<td>ED primer II:</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Primer A — MDP, HEMA, chemical initiator, water, 5-NMSA</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Primer B — 5-NMSA, chemical initiator, water</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Panavia F 2.0:</td>
</tr>
<tr>
<td></td>
<td></td>
<td>A Paste — Quartz glass, microfiller, MDP, methacrylate, photoinitiator</td>
</tr>
<tr>
<td></td>
<td></td>
<td>B Paste — Barium glass, NaF, methacrylates, chemical initiator</td>
</tr>
</tbody>
</table>

All materials were manufactured by Kuraray Medical Inc., Tokyo, Japan.

MDP = 10-methacryloxydecyl dihydrogen phosphate; HEMA = 2-hydroxyethyl methacrylate; Bis-GMA = bisphenol A diglycidymethacrylate; TEGDMA = triethylene glycol dimethacrylate; 5-NMSA = N-methacryloyl 5-aminosalicylic acid; NaF = Sodium fluoride
with a combination of a self-etching primer bonding system, Clearfil SE Bond (Kuraray Medical, Tokyo, Japan), and a low-viscosity microfilled resin, Protect Liner F (Kuraray Medical). Table 1 lists the batch numbers and compositions of the materials used. According to the manufacturer’s instructions, SE Primer was first applied to the cavity for 20 seconds and gently air-dried. SE Bond was then applied, mildly air-dried, and light-cured for 10 seconds using a conventional halogen light curing unit (XL 3000, 3M ESPE, Seefeld, Germany). Thereafter, Protect Liner F was placed on the cured adhesive surface using a brush-on technique and light-cured for 20 seconds. The other 10 cavities remained as a Non-Coated group. After cavity preparation, the roots of all the teeth were embedded in a silicon impression material (Exafine Putty Type, GC, Tokyo, Japan).

Impression of each cavity was taken using a combination of an agar (Aromaloid, GC) and an irreversible hydrocolloid (Aroma Fine DFII, GC). The impression was then cast in a Type III stone (Zo Gypsum, GC). After impression taking, the cavities were temporized with a water-setting temporary filling material (Cavit-G, 3M ESPE) and stored in water at 37°C for 24 hours. Composite inlays were fabricated on the working casts using an indirect resin composite (Estenia, Kuraray Medical) according to the manufacturer’s instructions. The inlays were polymerized with a halogen light curing unit (Alpha Light II, J. Morita, Kyoto, Japan) for three minutes, and then heat-cured at 110°C in an oven (KL-100, Kuraray Medical) for 15 minutes.

The temporary filling material was removed with a spoon excavator, and the cavity cleaned with an alcohol-soaked cotton pellet for 10 seconds. Following this, trial insertion of the composite inlays prior to cementation was performed to check their fit. An etchant of 37% phosphoric acid gel (K-etchant, Kuraray Medical) was applied to the fitting surface of the inlay for 10 seconds, rinsed, and gently air-dried. The inlay was then silanized using a mixture of Clearfil SE Bond Primer and Porcelain Bond Activator (Kuraray Medical) applied to the surface of the inlay for five seconds and mildly air-dried. Before cementation, the cavity surface of the non-coating group was treated with ED Primer II (Kuraray Medical) for 30 seconds and gently air-dried.

Next, the resin-coated surface was treated with 37% phosphoric acid gel (K-etchant) for 10 seconds, rinsed, and dried to remove any debris on the surface. A mixture of ED Primer II was then applied for five seconds and gently air-dried.

To simulate the interproximal contact area, an adjacent tooth was used during light curing. For cementation, equal amounts of the two pastes of a dual-cure resin cement (Panavia F 2.0, Kuraray Medical) were mixed together and placed on the fitting surface of the inlays. The inlays were seated in the cavities using hand pressure. After removal of any excess cement, the cement was exposed to light (XL 3000, 3M ESPE) from the occlusal, buccal, and lingual directions for 20 seconds each. After cementation, the margins of the restorations were finished with a superfine diamond bur (SP207CR, Shofu) and then stored in water at 37°C for 24 hours.

Microtensile bond strength test

The occlusal and proximal surfaces of each specimen were ground with a carborundum point (2 HP, Shofu) for additional resin composite build-up. Ground surface of the inlay was treated with 37% phosphoric acid gel (K-etchant) for 10 seconds, rinsed, and gently air-dried. A mixture of SE Primer and Porcelain Bond Activator was then applied for five seconds and mildly air-dried. SE Bond was then applied, gently air-dried, and light-cured for 10 seconds. A resin composite (Clearfil AP-X, Kuraray Medical) was built up to a height of 3 mm in two increments. Each increment was light-cured for 20 seconds (XL 3000).

Each specimen was sectioned perpendicular to the bonded interface occlusally or proximally to obtain a thickness of 0.7 mm. Occlusal and proximal slabs were obtained from different restorations. Eventually, 4–5 occlusal slabs or 3–4 proximal slabs were obtained from one specimen. Each slab was trimmed and shaped along the adhesive interface with a superfine diamond bur (V16ff, GC) to obtain an hourglass shape. The proximal box was discarded to get the occlusal slab, while the first cut slab was discarded due to its irregular shape. The narrowest portion at the adhesive interface was then trimmed to approximately 1 mm² for the MTBS test. Width and thickness were then measured with digital calipers to calculate the bonded surface area.

Each specimen was attached to a Bencor Multi-Testing apparatus (Danville Engineering, San Ramon, CA, USA) with cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA) and placed in a benchtop material tester (EZ Test, Shimadzu, Kyoto, Japan) for MTBS test at a crosshead speed of 1.0 mm/min. Number of samples in each group was 11. Data were statistically analyzed using two-way ANOVA at a 5% level of significance.

Scanning electron microscopic (SEM) observation

After MTBS testing, the fractured specimens were fixed in 10% neutral buffer formalin. Both the dentin and composite sides of the fractured samples were desiccated, gold sputter-coated, and observed with a SEM (JSM-5310LV, JEOL, Tokyo, Japan) to confirm the fracture mode.

Fracture mode was classified into one of the following four categories — A: Partial adhesive
failure, where remnants of resin cement remained on the dentin surface; B: Cohesive failure within resin cement; C: Complete and partial adhesive failure including cohesive failure of the resin cement at resin coating-resin cement interface; D: Partial adhesive failure at resin coating-resin cement interface where remnants of resin cement remained on the coating surface.

To examine the dentin-composite interface, three bonded specimens were prepared for each group in the same manner as described in the sample preparation for MTBS testing. The specimens were stored in water at 37°C for 24 hours. Bonded assemblies were then sectioned into two halves using a low-speed diamond saw microtome (Isomet, Buehler, Lake Bluff, IL, USA) and embedded in a self-curing epoxy resin (Epon 815, Nissin EM, Tokyo, Japan). The specimens were subsequently polished with silicon carbide papers under running water, and polished to high gloss with abrasive disks and diamond pastes of decreasing abrasiveness down to 0.25 μm. At each step, the specimens were cleaned ultrasonically. Polished specimens were subjected to argon ion beam etching (EIS-1E, Elionix, Tokyo, Japan) for five minutes at 0.2 mA and 1 kV to disclose the interfacial structure, and then gold sputter-coated.

RESULTS

Microtensile bond strengths and fracture modes

Microtensile bond strengths and their fracture modes are summarized in Table 2. Two-way ANOVA revealed that the MTBSs of resin cement to the dentin walls of Class II cavities were influenced by resin coating (F=29.22, p=.0001), but not by dentin region (F=0.467, p=0.499). There was no interaction between resin coating and dentin region (F=0.009, p=0.997). Indeed, the resin coating group provided statistically higher MTBSs to dentin than those of the non-coating group (p<0.05). Some specimens debonded during trimming for MTBS testing in the non-coating group, which were discarded from the calculation. However, no specimens debonded in the resin coating group.

Figure 2 shows the SEM views of typical fracture modes for the coating and non-coating groups. For the non-coating group, the fracture mode was mainly partial adhesive failure (category A). In the resin coating group, the key fracture mode was complete and partial adhesive failure including cohesive failure of the resin cement at the resin coating-resin cement interface (category C).

SEM observation of the interface

Figure 3 shows the SEM views of the dentin-composite interface. The SEM pictures revealed good

<table>
<thead>
<tr>
<th>Non-coating</th>
<th>Occlusal</th>
<th>Proximal</th>
</tr>
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<tbody>
<tr>
<td>AB</td>
<td>17.9 (3.1)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>16.7 (3.7)&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Resin coating</td>
<td>25.7 (5.2)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>24.8 (6.7)&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Number of specimens = 11.

Four modes of fracture — A: partial adhesive failure, where remnants of resin cement remained on the dentin surface; B: cohesive failure within resin cement; C: complete and partial adhesive failure including cohesive failure of the resin coating-resin cement interface; D: partial adhesive failure at resin coating-resin cement interface where remnants of resin cement remained on the coating surface.

Same superscript letters among MTBS values represent no significant differences (p>0.05.).

![Fig. 2 Representative scanning electron micrographs of the fractured surface on the dentin side after microtensile bond strength test. (a) Non-coating group: Partial adhesive failure where remnants of resin cement (CE) remained on the dentin (D) surface. (b) Resin-coating group: Complete and partial adhesive failure including cohesive failure of the resin cement remaining at the resin coating (RC)-resin cement interface.](image_url)
bonding at the interface between indirect resin composite and dentin in both the resin-coated and non-coated specimens. However, in some non-coated specimens, some gaps were observed at the interface—probably due to poor bonding.

The hybrid layer was observed at the interface in every specimen. Thickness of the hybrid layer in resin-coated dentin was approximately 0.5 μm on both the occlusal (a) and proximal (b) sides. In the non-coating group, thickness of the hybrid layer was also 0.5 μm on both the occlusal (c) and proximal (d) sides.

DISCUSSION

When pitted against conventional cements, resin cements yield some advantages—such as micromechanical bonding to tooth structure, low solubility, and better wear resistance. Successful adhesion of the resin cement is very important for the longevity of indirect restorations. However, the bond strength of resin cements is still lower than that of adhesive systems for direct composites. With indirect restorations, contamination of the prepared dentin surfaces with temporary filling materials, blood, and saliva may also further deteriorate dentin bonding capacity.

Previous studies have shown that the bond strength of resin cements to dentin could be successfully improved by a resin coating technique using a combination of an adhesive system and a low-viscosity microfilled resin. This technique can also protect the pulp and reduce postoperative sensitivity in vital teeth, as well as reduce coronal leakage in endodontically treated teeth. To improve the bond strength of resin cements to dentin, careful and discerning selection of resin coating material and adhesive system is very important. On this account,
low-viscosity microfilled resin seems to be an expedient choice on two fronts. It can protect the underlying adhesive, and at the same time promote polymerization of the adhesive because of two-pronged effects: double-cure action of the adhesive as well as air inhibition on top of the adhesive, thereby resulting in increased bond strength. It has been shown that if the correct combination of adhesive and low-viscosity microfilled resin were selected, good bonding of the resin cement to dentin can be obtained for indirect restorations to the extent of being almost identical to the bond strength of direct composite restorations. A two-step self-etching primer bonding system, Clearfil SE Bond, and a low-viscosity microfilled resin, Protect Liner F, were used in this study. This combination was selected because it demonstrated the highest bond strength in a previous study.

It is difficult to evaluate regional bond strength in Class II cavities by conventional testing methods, chiefly because of the relatively large adhesive area needed. However, microtensile bond strength test enables us to evaluate regional bond strengths in the same tooth. Previous studies have demonstrated that microtensile bond strength is influenced by several clinical factors, such as bonding technique, cavity configuration factor (C-factor), remaining dentin thickness, dentin permeability, and also the age and depth of dentin. Contamination from saliva, blood, temporary filling materials, and impression materials also affects the adhesion of indirect restorations.

Smear layers produced with a regular diamond bur were found to be more acid-resistant than those produced by superfine diamond burs, thereby influencing the dentin bonding of a two-step self-etching primer bonding system. In light of this finding, cavities in the present study were finished with a superfine diamond bur.

From our results, it was shown that the bond strength of resin cement to dentin was influenced by the material, but not by region. Similarly, Zheng et al. reported that the bond strengths of different regions (pulp horn, center, and periphery) to dentin were influenced by the material and pulpal pressure, but not by region. In a study by Bouillaguet et al., they compared the regional (occlusal, axial, gingival) resin-dentin bond strengths to MOD cavities versus the same surfaces isolated by cutting away all but the test surface. They reported that the mean bond strengths in the cavity bonding group were significantly lower than those of the flat bonding surface group. However, there were no significant, consistent differences among the various regions within either group. On the other hand, it was also reported that regional differences were found in resin-dentin bond strengths, which were probably related more to operator variability than to material differences. Bond strength varies across a dentin surface, and the magnitude of variability can be influenced by the bonding technique.

The C-factor is the ratio of bonded surface area to unbonded or free surface area. Since the C-factor of a complex MO cavity is higher than that of a flat bonding surface, high polymerization stress may occur during cementation. The resin coating could have thus acted as a stress absorber and reduced the polymerization shrinkage stress at the interface.

Materials for temporization and impression taking influence the bond strength of resin cements to the surface of resin-coated cavities. Previous studies have demonstrated that a water-setting material, such as Cavit-G (3M ESPE, Seefeld, Germany), was an optimal material for temporization, while resin-based temporary filling materials reduced bond strength due to contamination of the coating surface. As for impression taking of resin-coated surfaces, hydrocolloid impression materials are recommended. When addition-cured silicon impression materials are used for resin-coated surfaces, the air-inhibited unpolymerized surface layer should be removed with an alcohol-soaked cotton pellet prior to impression taking.

Hybridization of the intertubular dentin is an essential mechanism of dentin bonding. In the case of directly placed resin composites, adhesion to dentin occurs by the formation of a hybrid layer—the same as with indirect restorations. As for the thickness of the hybrid layer with resin coating, it depends on the adhesive system used in combination with the resin cement. SE Primer contains an acidic monomer, MDP, which solubilizes the smear layer and demineralizes the underlying dentin, as a result of mild surface etching. ED Primer II also contains MDP, which plays the same role as in SE Primer. In the present study, thickness of the hybrid layer in both resin coating and non-coating groups was approximately 0.5 μm—which was due to mild surface etching. With these self-etching primer systems, smear plugs partially remain on the treated dentin as a result of incomplete formation of resin tags in the dentinal tubules.

This study showed that the resin coating technique evidently improved the microtensile bond strength of resin cement to the walls of Class II cavities. This achievement is essential for the prevention of microleakage and secondary caries. A gap-free interface between the restoration and cavity wall is undisputedly necessary for good clinical longevity of restorations. On this note, application of a resin coating on prepared cavities can reduce interfacial gap formation between composite inlays and the cavity surface. At this juncture, it should be highlighted that resin coating has been shown to
improve dentin bonding of resin cements, whereas no effect was observed on enamel bonding\(^2\). With the enamel surface, no significant statistical differences in bond strength were observed with or without the use of a low-viscosity microfilled resin. Further, the bonding performance of indirect restorations to enamel was clinically acceptable, which was almost identical to that achieved with direct resin composite restorations.

In view of the current, vigorous promotion of minimal intervention dentistry, the resin coating technique can indeed achieve this objective when an indirect restoration is indicated. However, complete and partial adhesive failure including cohesive failure of the resin cement at the resin coating-resin cement interface (category C) was the key fracture mode observed in the resin coating group. This finding suggested that the joint interface was the weak link in indirect composite restorations. In light of this finding, there is an imperative need for better, enhanced bond strength at the interface. Nonetheless, this needs can currently be met by the resin coating technique, whereby the resin coating material can protect the prepared dentin from chemical, physical, and biological insults$^{16}$.

**CONCLUSIONS**

Within the limitations of the present study, the following conclusions were drawn:

1. Application of a resin coating — using a combination of Clearfil SE Bond and Protect Liner F — to the dentin surface following cavity preparation significantly improved the microtensile bond strength of Panavia F to dentin in indirect Class II restorations.

2. No regional differences in microtensile bond strength between the occlusal and proximal dentin surfaces were observed.

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