The Effects of Tooth Preparation Cleansing Protocols on the Bond Strength of Self-adhesive Resin Luting Cement to Contaminated Dentin

Y Chaiyabutr • JC Kois

Clinical Relevance
Tooth preparation cleaning protocol surface management with lower pressure aluminous particle abrasion provides a significant improvement in bond strength to dentin.

SUMMARY
This in vitro study evaluated the bond strength of a self-adhesive luting cement after using four different techniques to remove surface contamination on dentin. Extracted human molars were flattened to expose the dentin surface and prepared for full crown preparation. Acrylic temporary crowns were fabricated and placed using temporary cement. The specimens were stored at room temperature with 100% relative humidity for seven days. Following removal of the temporary crowns, the specimens were randomly divided into four groups, and excess provisional cement was removed with 1) a hand instrument (excavator), 2) prophylaxis with a mixture of flour pumice and water 3) aluminous oxide abrasion with a particle size of 27 µm at 40 psi and 4) aluminous oxide abrasion with a particle size of 50 µm at 40 psi. The microstructure morphology of the tooth surface was evaluated and residual materials were detected using SEM and EDS analysis of randomly selected specimens. The ceramics were treated with 9.5% hydrofluoric acid–etch and silanized to the prepared dentin prior to cementing with self-adhesive resin cement (RelyX Unicem, 3M ESPE). The shear bond strength was determined at a crosshead speed of 0.5 mm/minute. The results were analyzed with one-way ANOVA, followed by Tukey’s test. Particle abrasion treatment of dentin with an aluminous oxide particle provided the highest values of bond strength, while hand instrument excavation was the lowest (p<0.05). Aluminous oxide particle size did not significantly influence the bond strength at 40 psi. The use of low pres-
sure and small particle abrasion treated dentin as a mechanical cleansing protocol prior to definitive cementation increased the bond strength of self-adhesive resin-luting cement to dentin following eugenol-containing temporary cement.

INTRODUCTION

Factors, such as tooth preparation design, type of definitive luting cement and tooth surface management, influence the retention and longevity of definitive restorations.\textsuperscript{1,2} During the fabrication of indirect restorations, provisional restorations are placed with temporary luting cement in order to avoid sensitivity, infection and tooth movement. The temporary luting cement is required to be removed from the dentin surface prior to definitive cementation. Contradictory findings have been reported with regard to bond strength to dentin after placement and removal of temporary luting cement. Eugenol-containing materials are commonly used as temporary luting cements, because of their bacteriostatic effect, low cost, ease of removal and good sealing ability.\textsuperscript{3} Some studies found that eugenol-containing cements have adverse effects on the bond strength of the definitive restoration. These effects include changes in wettability, reactivity of the dentin and interaction with the polymerization of resin-based materials.\textsuperscript{4-7} While other studies have not observed any detrimental effect on bond strength when eugenol-containing cement is used,\textsuperscript{8-10} it was suggested that the negative affect may not be caused by eugenol, but by the presence of residual temporary luting cement. Simple mechanical cleansing methods may partially remove oily, greasy additives in the cement, as evidenced by remnants of the temporary luting cement still being observed microscopically on the dentin surface after cleaning, while the surface appeared macroscopically clean.\textsuperscript{11-13}

Regardless of the definitive luting cement, whether conventional or resin-based cement is used, an effective tooth preparation cleansing protocol seems to be a desirable procedure to avoid any interference along the interface between the dentin surface and luting cement. Various attempts by tooth preparation cleansing protocols have been proposed, including a chemical cleansing agent and a mechanical cleansing protocol. Different chemical cleaning agents, such as chlorhexidine gluconate and agents containing ethanol, ethyl-acetate and acetone, have been used. The disadvantage of these chemical agents is that their effectiveness occurs only on the superficial layer of the dentin and an additional mechanical cleansing protocol may be necessary. A mechanical cleansing protocol generally includes rotary instrumentation with pumice, an air polisher or micro-particle abrasion. Several investigations involving rotary instrumentation with pumice showed that this technique is more effective in removing residual temporary cement when compared to a chemical cleansing agent (0.12% chlorhexidine gluconate) and an explorer/air-water technique, alone.\textsuperscript{12,13} However, other studies reported contradictory results, indicating that pumice may not be very effective in every situation.\textsuperscript{14-16} The micro-particle abrasion technique has been originally described as a method of tooth structure pretreatment. This technique was developed by RB Black as an alternative treatment to minimize the vibration, pressure and heat associated with rotary cutting instruments.\textsuperscript{17} Kinetic energy, generated by a high-velocity stream of aluminum oxide particle, is utilized to prepare dentin and enamel, while having little impact on softer materials, such as gingival tissue. The system, however, was not well accepted at that time, since the tooth preparation for gold and amalgam required supplemental mechanical retention.\textsuperscript{18}

Along with the development of dentin bonding agents, tooth preparation for bonded restorations has taken on a more conservative form, and micro-particle abrasion technology is again used as an alternative for surface preparation in addition to high-speed mechanical instrumentation.\textsuperscript{19} Particle abrasion creates rough, irregular surfaces that increase the bonding surface area, which was reported to increase the bond strength of restorations to enamel and dentin.\textsuperscript{20} However, using a mechanical cleaning protocol prior to definitive cementation has limited documentation. It has been postulated that using alumina oxide particle abrasion may affect the bond strength of restorations using a self-adhesive resin-luting cement as the definitive luting cement, because any alumina particle embedded in the dentin would inhibit the self-etching effect and any chemical reaction between the cement and an alumina oxide particle may have a negative affect on bond strength.

Therefore, this study 1) evaluated the \textit{in vitro} bond strength of a self-adhesive luting cement by using four different techniques to remove surface contamination on dentin and 2) investigated the effect of alumina oxide particle abrasion used as a mechanical cleaning protocol on the bond strength of ceramic luted with self-adhesive resin-luting cement. The null hypothesis was that there is no difference in bond strength in any technique of the tooth preparation cleansing protocol.

METHODS AND MATERIALS

Extracted intact human third molars were selected based on the criteria that there was no evidence of caries and no restorations with any cracks or fractures in the crown. Overall, the tooth size was within a 10% standard deviation. From the time of extraction, these
teeth were kept hydrated in distilled water at room temperature and, during the preparation, each tooth was wrapped in water-moistened gauze. Each tooth was mounted in a copper cylinder with the buccal-cementoenamel junction 3 mm above the top of the copper cylinder. Each tooth was attached with wax to a dental surveyor rod, with the long axis of the tooth being parallel to the surveyor rod. These teeth were then lowered into the copper cylinder and positioned in its center. Premixed autopolymerizing resin (GC Pattern Resin, GC America, Scottsdale, AZ, USA) was injected into the cylinder until the cylinder was full. After the resin had set, the teeth were flattened to expose the dentin surface. They were then prepared as full crown with a chamfered margin, using a high-speeding handpiece and diamond bur (KS-1; Brasseller USA, Savannah, GA, USA). The axial surface of the prepared teeth was aimed parallel with the long axis of the teeth.

A provisional crown was made using acrylic resin (Temporary Crown and Bridge Resin; Dentsply Caulk Inc, Milford, DE, USA) and provisional cementation was performed to simulate the clinical condition. The provisional cement (Table 1), with eugenol (Temp Bond; Kerr Co, Orange, CA, USA), was placed in the acrylic provisional crown. The excess provisional luting cement was removed and the specimens were stored at room temperature and 100% relative humidity for seven days prior to bonding with ceramic.

After seven days, the provisional crowns were removed, along with the remaining cement particles, and the teeth were then randomly divided into four groups and the dentin surfaces were cleaned with 1) a hand instrument (excavator) alone (n=8); 2) a hand instrument (excavator), followed by prophy with a mixture of flour of pumice and water (n=8); 3) a hand instrument (excavator), followed by aluminous oxide abrasion with a particle size of 27 µm at 40 psi at a distance of 2 mm (PrepStart, Danville Engineering, CA, USA) (n=8), rinsed and dried gently or 4) a hand instrument (excavator) followed by aluminous oxide abrasion with a particle size of 50 µm at 40 psi at a distance of 2 mm (PrepStart) (n=8), rinsed and dried gently.

Ceramic powder (Reflex; Wieland Dental Systems Inc, Milford, CT, USA) was mixed using a standard water-powder ratio and vibrated into silicone molds to form the ceramic bar. The ceramics were fired in a furnace (Dekema Austromat 3001; Frankfurt, Germany) at 905°C with a ramp rate of 75 seconds and the holding time of 120 seconds. The ceramic bars were glazed and polished with 1 µm diamond paste (Ecomet 3; Buehler, Lake Bluff, IL, USA) to create the flat surface before they were cut to size (4 x 4 x 2 mm). The ceramics were treated with 9.5% hydrofluoric acid for 180 seconds, then silanized for 60 seconds and air-dried (Table 1). The ceramics were bonded to the prepared teeth using the self-adhesive resin cement (RelyX Unicem Aplicap; 3M ESPE, St Paul, MN, USA). The apical capsule of RelyX Unicem was inserted into the apicap activator. The level arm was pushed down to release the acid and held for five seconds; the capsule was immediately placed in the mixing machine and mixed for 15 seconds. Note that this mixing protocol has been studied and modified to yield the best mixing results. The mixed cement was directly applied to both the prepared teeth and ceramic. The ceramic was seated on the flattened dentin surface with finger pressure, and the excess cement was removed with an explorer after initial setting. The specimens were left without light activation to simulate the clinical situation for metal substrate crowns. The bonded specimens were kept in a dry condition for approximately 30 minutes (starting when the cements were mixed, the ceramics were bonded to the teeth, the cement excess was removed from the initial set until the cements were completely set) to ensure that the ceramic specimens were successfully bonded to the teeth, then stored at room temperature.

### Table 1: Overview of Materials and Application Mode Under Investigation

<table>
<thead>
<tr>
<th>Materials and Application</th>
<th>Composition</th>
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<tbody>
<tr>
<td><strong>Temporary Luting Agent</strong></td>
<td><strong>Temp Bond (Kerr Co, Lot #6-1018)</strong></td>
</tr>
<tr>
<td><strong>Self-adhesive Luting Agent</strong></td>
<td><strong>RelyX Unicem (3M ESPE, Lot #227706): 15 seconds for mixing time, 5 seconds for liquid activate time</strong></td>
</tr>
<tr>
<td><strong>Materials used for ceramic surface treatment</strong></td>
<td><strong>Powder: Silanized glass powder (85-95%wt), Silane Treated Silica (5-10 %wt), Calcium hydroxide (1-5%wt), Sustituted Pyrimidine (1-5%wt), Sodium Persulfate (&lt;1%wt)</strong></td>
</tr>
<tr>
<td><strong>RelyX Ceramic Primer (3M ESPE, Lot #2721): applied for 60 seconds and dry</strong></td>
<td><strong>Liquid: Methacrylated phosphoric esters (40-50%wt), Triethylene Glycol Dimethacrylate (25-35%wt), Substituted Dimethacrylate (22-34 %wt)</strong></td>
</tr>
<tr>
<td><strong>Ultradent Porcelain Etch Gel (Ultradent, Inc, Lot #X063): applied for 180 seconds</strong></td>
<td><strong>9.5% Hydrofluoric acid</strong></td>
</tr>
<tr>
<td><strong>Ethyl alcohol (70-80%wt), Water (20-30%wt)</strong></td>
<td><strong>Methacryloxypropyltrimethoxysilane (&lt;2%wt)</strong></td>
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Each specimen was mounted onto a metal holder in the universal testing machine (Instron model 5585H, Instron Corp, Canton, MA, USA), and the load was applied with a flatten-rod (custom made) 2 mm in diameter, corresponding to the size of the ceramic thickness. Each specimen was tightened and stabilized to ensure that the edge of the shearing rod was positioned as close to the ceramic-tooth interface as possible. A shear load was applied at a crosshead speed of 0.5 mm/minute until failure. The ultimate load to failure was recorded in Newton (N). The average bond strength (MPa) was calculated by dividing the maximum ultimate load to failure (N) by the bonded cross-sectional area (mm²). The mean and standard deviations were recorded. The fractured surface was examined under an optical microscope for the mode of failure. The failed specimens were classified into five types based on mode of failure: Type I—Cement principally >3/4 on the prepared tooth (Adhesive); Type II—Cement totally on the ceramic (Adhesive); Type III—Cement principally >3/4 on ceramic (Adhesive); Type IV—Tooth fracture (Cohesive); Type V—Ceramic fracture (Cohesive). The data was subjected to one-way ANOVA, followed by Tukey’s multiple range tests to define significant differences at a confidence level of 95%. This determined whether significant differences existed in bond strength between the testing groups. Randomly selected specimens were scanned by Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDS) to evaluate the microstructure morphology of the tooth surface with different mechanical cleansing protocols.

### RESULTS

Significant differences were found in bond strength, resulting from the different tooth preparation cleansing protocols (Table 2). Particle abrasion treatment of dentin with an aluminous oxide particle at a low pressure of 40 psi provided the highest values of bond strength using self-adhesive luting cement, while a hand instrument alone was the lowest ($p<0.05$). When using aluminous oxide particle abrasion, particle size did not significantly influence bond strength ($p>0.05$).

The mode of failure from the hand instrument alone and the pumice polish were adhesive failure at the dentin interface, while cohesive failure was found in the 27 µm particle abrasion group. The morphologic characteristics of the dentin surface following exposure to different mechanical cleaning protocols are shown in the SEM micrographs (Figures 1-4). The dentin surface after using a hand instrument excavator alone had remnants of some additive temporary cement remaining on the surface. Irregular rough surfaces were detected in those particle abrasion groups with both a particle size of 27 µm or 50 µm. There was some artifact crack formation found on the surface. These cracks were created during desiccation for scanning electron microscope evaluation. Energy dispersive analysis revealed the major component elements of carbon (C), oxygen (O), sodium (Na), magnesium (Mg), phosphorous (P) and cal-

### Table 2: Mean of Bond Strength Values (MPa), the Respective Standard Deviations (± SD) of Different Tooth Surface Treatment and Percentage of Failure Mode

<table>
<thead>
<tr>
<th>Mechanical Cleansing Protocol</th>
<th>Bond Strength</th>
<th>Adhesive Failure</th>
<th>Cohesive Failure</th>
</tr>
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<tbody>
<tr>
<td></td>
<td></td>
<td>Type I</td>
<td>Type II</td>
</tr>
<tr>
<td>Hand Instrument</td>
<td>3.861 ± 1.152</td>
<td>C</td>
<td>-</td>
</tr>
<tr>
<td>Pumice</td>
<td>5.042 ± 1.430</td>
<td>B</td>
<td>-</td>
</tr>
<tr>
<td>27 µm Al₂O₃</td>
<td>8.615 ± 4.105</td>
<td>A</td>
<td>-</td>
</tr>
<tr>
<td>50 µm Al₂O₃</td>
<td>8.476 ± 3.343</td>
<td>A</td>
<td>-</td>
</tr>
</tbody>
</table>

Different letters indicate statistically different means:
- Type I: Cement principally >3/4 on prepared tooth
- Type II: Cement on ceramic
- Type III: Cement principally >3/4 on ceramic
- Type IV: Tooth fracture
- Type V: Ceramic fracture

Figure 1. SEM micrograph of the dentin surface of tooth preparation after mechanical cleansing with a hand instrument. Notice that the remaining additive particles of temporary cement were detected on the surface. (Original magnification 500x).
Cesium (Cs) in all the testing groups (Figure 5). The average element measurement composition of the aluminum (Al) content of 0.67% by weight was found in specimens treated with particle abrasion (Figure 6).

**DISCUSSION**

Eugenol-containing temporary cement was used in this study, since it is presumably a common material used in dental practice. It has been reported that eugenol diffusion from eugenol-containing cement appears to depend more on the role of the hydrolysis of eugenol from eugenol-containing cement than on dentin permeability. The diffusion rate of eugenol released from the cement increased to a peak after one day, which is about 0.3 ηmol per minute, then decreased slowly to 0.08 ηmol per minute after 14 days. Therefore, a period of seven days was considered mandatory to allow the eugenol to diffuse into the dentin and eventually possibly have an affect on bond strength. In addition, the seven-day period is reasonable for temporary restorations placed in clinical situations. After seven days following temporary cementation, the provisional restorations were removed and four different mechanical cleansing protocols were utilized. The surfaces were then vigorously rinsed with water spray after the cleansing protocol was done. It was noticed that major particle remnants were removed in the groups with pumice and aluminous particle abrasion, as evidenced in the SEM micrograph and EDS analysis. However, some remaining particles of additive temporary cements were found microscopically in the group using the hand instrument alone (Figure 7). Any remaining particles could interfere with the chemical bond and micromechanical retention of self-adhesive cement, resulting in lower bond strength. These results warrant rejection of the null hypothesis that there is no difference in bond strength among the different procedures, where particle abrasion generally was significantly higher than the bond strength from pumice and hand-instrumentation only. This finding is consistent with previous results, that the increased irregular, rough surface improved bond strength due to the increasing bonding surface area. The chemical composition of the dentin surface, which, together with surface roughness and capillary action, may be the decisive factor for dentin surface-free energy and, consequently, diffusion of the self-adhesive luting cement into the dentin surface. The increase in bond strength in the particle abrasion group could also be explained by the mode of failure. Cohesive failures were found in the 27 µm particle abrasion group, while hand instrumentation alone and pumice had more adhesive failures at the dentin interface. The specimens with more adhesive failures at the dentin interface also provided lower bond strengths, which indicat-
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ed weaker resin-dentin bonds. The result was also consistent with other reports that showed no significant difference in bond strength among the different particle sizes of aluminous oxide. However, the mode of failure was not similarly affected by the particle sizes. The tooth surfaces that were treated with 27 µm particle abrasion increased the cohesive failures in dentin. The smaller particles would have created a more retentive microscopic pattern, resulting in a stronger bond interface.

The type of dental substrate could influence bond strength, since the dentin surface may vary from tooth to tooth, as the dentin tubule diameter varies with age and also changes in size from the surface to the pulp chamber, creating a variation in dentin bond strength within the same tooth, depending on the bonding site. To reduce these variables, the extracted teeth used in this study were limited to patients ranging in age from 15 to 25 years having teeth with restorations extracted within six months.

Self-adhesive luting cement (RelyX Unicem) was used in this study as an example of the definitive cement for cohesively retained restorations. Restorations are mainly classified into two categories: cohesively retained restorations and adhesively retained restorations. Cohesively retained restorations require a preparation design that exhibits resistance and retention form. To improve the longevity of cohesively retained restorations, selecting the proper definitive cement that provides stress distribution, great dentin sealing ability, less post-treatment sensitivity and predictability in handling, is crucial. The use of self-adhesive resin cement has increased in practice due to its advantages, including fewer steps in the bonding protocol and the fact that it is less technique sensitive due to a lack of pretreatment of the tooth structure. According to the information available on this self-adhesive cement, theoretically, with the presence of water, phosphoric acid methacylate monomer in cement will demineralize the smear layer and the underlying dentin, while simultaneously infiltrating the porous dentin surface as a result of its hydrophilic properties and the neutralization of acidic reaction that occurs as polymerization progresses (ESPE [information from the manufacturer]). It has been reported that the bond strength of this self-adhesive resin cement is comparable to the bond strength of dual-polymerization resin cement and is higher in bond strength than traditional zinc phosphate luting cement, glass ionomer and resin-modified glass-ionomer cement. The seal of self-adhesive resin-based cement is also comparable to cements that employ adhesives for sealing dentin.

In the current study, it was confirmed that using low-pressure alumina particle abrasion as a mechanical cleansing protocol to provide dentin surface treatment

Figure 5: Energy Dispersive Analysis (EDS) of the tooth surface and content of element after mechanical cleansing with pumice and rinsed with water.

Figure 6: Energy Dispersive Analysis (EDS) of the tooth surface and content of element after mechanical cleansing with Al₂O₃ particle abrasion.

Figure 7. Specimens of surface treatment mechanical cleansing with pumice (left) and mechanical cleansing with Al₂O₃ particle abrasion (right).
prior to definitive cementation is beneficial and did not have an adverse effect on the bond strength of self-adhesive resin-luting cement. This study utilized 40 psi pressure, a nozzle tip with a diameter of 0.015 mm and a 2 mm distance from the tooth surface. Changing various parameters, such as nozzle tip diameter, nozzle tip distance and higher air pressure, could affect the results of bonding.20

CONCLUSIONS

Within the limitations of this study, using low-pressure and small-particle abrasion-treated dentin as a mechanical cleansing protocol prior to definitive cementation improved the bond strength of self-adhesive resin-luting cement to dentin following the use of eugenol-containing temporary cement.

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References


