Effect of Dentin-cleaning Techniques on the Shear Bond Strength of Self-adhesive Resin Luting Cement to Dentin

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GC Santos Jr

Clinical Relevance

This *in vitro* study showed that sandblasting dentin with aluminum oxide is the most effective method to enhance bonding of an indirect restoration to the tooth surface when a self-adhesive resin luting cement is used.

SUMMARY

Objective: This *in vitro* study evaluated the influence of different cleansing techniques on the bond strength of self-adhesive cement to dentin.

Methods and Materials: A total of 33 noncari-ous human molars were sectioned mesiodistally and embedded in chemically cured resin with the buccal or lingual surfaces facing upward. Superficial dentin was exposed and resin disk provisional restorations were cemented to the dentin surfaces with noneugenol provisional cement and were stored in distilled water at 37°C. After seven days, the provisional restorations were removed and 13 specimens were randomly assigned to each of the five groups (n=13), according to the following cleansing treatments: G1—excavator (control); G2—0.12% chlorhexidine digluconate; G3—40% polyacrylic acid; G4—mixture of flour pumice and water; and G5—sandblasting with 50 μm aluminum oxide particles at a pressure of 87 psi. Resin composite disks (Filtek Supreme Plus, 3M ESPE Dental Products, St Paul, MN, USA) 4.7 (±0.1) mm in diameter and 3.0 (±0.5) mm in height were cemented with self-adhesive cement (RelyX Unicem, 3M...
ESPE), photocured, and stored in distilled water at 37°C for 24 hours. Shear bond strength testing was conducted using a universal test machine at a crosshead speed of 0.5 mm/min until failure.

Results: Data were analyzed using analysis of variance (ANOVA) and the Tukey-B rank order test. Sandblasting with aluminum oxide (11.32 ± 1.70 MPa) produced significantly higher shear bond strength values compared with any other treatment groups (p<0.05). No significant differences were found between G1-control (7.74 ± 1.72 MPa), G2-chlorhexidine (6.37 ± 1.47 MPa), and G4-pumice (7.33 ± 2.85 MPa) (p<0.05).

INTRODUCTION

The performance and longevity of indirect restorations can be affected by several factors, such as preparation design/coarseness, provisional luting agent, cleansing protocol, fit of the definitive restoration, design/coarseness, provisional luting preparations can be affected by several factors, such as the performance and longevity of indirect restorations, the main disadvantage of the adhesive system, which will result in higher bond strengths.1,2,9,10 Some studies have shown that mechanical removal of provisional cement by excavators is not adequate in that cement remnants can still be observed microscopically on dentin surfaces that appear to be macroscopically clean.10,11

Furthermore, the presence of a smear layer is a factor that can compromise the clinical bonding effectiveness of a final restoration.12 For self-adhesive cements, no pretreatment is recommended to remove the smear layer. However, several studies have proposed the use of different agents to enhance the interaction between resin cement and dental tissues, thereby increasing the bond strength.4,13,14 The related literature reports a great number of cleaning agents for the dentinal surface, some of which are based on mechanical methods; others use chemical agents. The most common techniques for mechanical cleansing include the use of rotary instrumentation with pumice and sandblasting with aluminum oxide particles; chemical cleansing techniques include the use of chlorhexidine digluconate, sodium hypochlorite, hydrogen peroxide, and polyacrylic acid. Application of different cleaning treatments to the dentin surface has shown different results on the smear layer and on the removal of provisional cement. Their effects range from simple removal of contaminants such as blood and debris to total or partial removal of the smear layer, promoting demineralization that can facilitate interaction between the resin and the collagen network on the dentin surface.2,3,6

Although some studies have reported that self-adhesive cements promote adequate bond strength to dentin,4,13,15 others have verified lower bond strength values when compared with conventional resin cements.5,16,17 These studies have observed that self-adhesive cements might interact superfi-
cially with dentin, leading to partial demineralization of the smear layer, which would result in a weak bonding mechanism. Because bond strength is considered a relevant factor in the longevity of indirect restorations, this study aims to verify the influence of different cleansing techniques on bond strength values of self-adhesive cement. It was hypothesized that shear bond strength values would differ significantly among the different cleansing techniques.

MATERIALS AND METHODS

Tooth Preparation

Thirty-three freshly extracted human molars free of cracks, caries, or restorations were selected for this study. After they were cleaned for calculus deposits and soft tissues, teeth were stored in 0.1% thymol solution for a maximum of 6 months until use. Before bonding experiments were begun, the teeth were retrieved from the disinfectant solution and were stored in distilled water, with daily changes of the latter for two weeks to remove the disinfectant. Approval to use human teeth was obtained from the Research Ethics Committee at the University of Western Ontario.

The teeth were sectioned mesiodistally at the central groove and the roots removed using a slow-speed saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. Sectioned teeth were embedded in chemically cured acrylic resin (Dentsply Caulk Orthodontic Resin, Milford, DE, USA) in cylindrical rings, with the buccal or lingual surface facing upward. Each tooth had its proximal enamel and superficial dentin removed with the use of a series of SiC-papers on a polisher (Polimet, Buehler Ltd, Lake Bluff, IL, USA), under water cooling, ending with 600 grit to obtain a flat dentin surface 1.5 to 2.0 mm from the pulp.

Provisional Composite Specimen Preparation

Sixty-five disk specimens were prepared by loading a composite temporization material (Protemp Plus, 3M ESPE Dental Products, St Paul, MN, USA) into translucent polyethylene cylindrical molds with an inner diameter of 3 mm and a height of 4 mm to fabricate the provisional restorations. These restorations were cemented to the center of dentin surfaces with non-eugenol-containing provisional cement (Temp Bond NE, Kerr Corp, Orange, CA, USA) and were held in place with a 500 g load for one minute. Teeth with provisional restorations were stored in distilled water at 37°C for seven days, after which time the provisional restorations and the cement were mechanically removed with a carving instrument until the dentin surface appeared macroscopically clean.

Resin-Based Composite (RBC) Specimen Preparation

A visible light–activated RBC (Filtek Supreme Plus, 3M ESPE), shade A3, was used to prepare disk specimens by loading the composite resin into a translucent polyethylene cylindrical mold with an inner diameter of 3 mm and a height of 4 mm. The RBC was placed in the mold in increments of about 2 mm. Each layer was individually light-cured for 40 seconds (Dentsply QHL75, with 600 mW/cm² output). Polymerization was completed in an oven (DentaColor XS, Kulzer, Irvine, CA) at 120°C for seven minutes. RBC surfaces were sandblasted with 50 lAl₂O₃ for 10 seconds, using an intraoral air abrasion device at a pressure of 87 psi (Optiblast, Buffalo Dental Mfg Inc, New York, NY, USA). All specimens were cleaned with 35% phosphoric acid gel for 5 seconds (Scotchbond Etchand, 3M ESPE) and were washed and dried.

Group Classification and Bonding Procedure

Specimens were randomly assigned to five groups of 13 specimens each, according to the cleansing protocols listed in Table 1. The materials employed are summarized in Table 2. The self-adhesive resin luting cement (Rely-X Unicem Clicker, 3M ESPE) was mixed for 20 seconds, according to the manufacturer’s instructions; it was then applied over the restoration and placed in the center of the tooth surface. The cement was initially light-cured for 10 seconds (Dentsply QHL75, with a 600 mW/cm² output) to allow removal of cement excess at the dentin restoration margin; then light-curing was performed at the lateral surfaces of the bonded RBC (20 seconds on each of the four surfaces, totaling 90 seconds of light activation). During the cementation procedure, a load of 500 g was used over the indirect restoration to standardize the pressure during cementation. Before shear bond strength measurement, the specimens were stored at 37°C in distilled water for 24 hours.

Shear Bond Strength Test

Each specimen was mounted in a Bencor Multi-T device to keep it stable and parallel to the base when positioned in a universal testing machine (Instron, Model 5585H, Instron Corp, Canton, MA, USA), and loading was applied with a flattened rod at a...
crosshead speed of 0.5 mm/min. Each specimen was tightened and stabilized to ensure that the edge of the shearing rod was positioned as close to the restoration-tooth interface as possible. Shear bond strength in megapascals (MPa) was calculated from the maximum stress at failure divided by the specimen surface area. Means and standard deviations were recorded for each group tested. One-way analysis of variance (ANOVA) was performed to assess the significance of differences in interfacial strength among the five treatments. Post hoc multiple comparisons were achieved using the Tukey test. *P*-values lower than 0.05 were considered to be statistically significant in all tests.

Table 1: Experimental Groups With Cleansing Treatments

<table>
<thead>
<tr>
<th>Group</th>
<th>Dentin-Cleansing Technique</th>
<th>Treatment</th>
</tr>
</thead>
</table>
| G1    | Hand instrument (control)   | Excavator was used for 10 seconds; dentin was rinsed and excess water removed with absorbent paper<br>
| G2    | 0.12% chlorhexidine digluconate | Scrubbed over dentin for 10 seconds using a disposable applicator and gently dried with absorbent paper<br>
| G3    | 40% polyacrylic acid        | Scrubbed over dentin for 10 seconds using an applicator. Then rinsed off for 10 seconds and excess water removed with absorbent paper<br>
| G4    | Pumice flour + water        | Pumice slurry was used with a prophy cup in a slow-speed rotary instrument for 10 seconds and rinsed for 10 seconds; excess water was removed with absorbent paper<br>
| G5    | Aluminum oxide, 50 μm Al₂O₃ | Dentin was sandblasted for 10 seconds at 87 psi at a distance of 2 cm. Then rinsed for 10 seconds and excess water removed with absorbent paper<br>

* To avoid dehydration of the dentin.

Table 2: Product Names, Batch Numbers, and Manufacturers

<table>
<thead>
<tr>
<th>Material</th>
<th>Product Name</th>
<th>Batch No.</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Provisional cement</td>
<td>Rely X Temp NE</td>
<td>355430</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td>Provisional restorative material</td>
<td>Protemp Plus</td>
<td>348072</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td>Luting cement</td>
<td>Rely X Unicem, Self-Adhesive Universal Cement</td>
<td>356321</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td>Restorative material</td>
<td>Filtek Supreme Plus</td>
<td>8RB</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td>Dentin-cleansing agents</td>
<td>Chlorhexidine digluconate</td>
<td>358558</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td></td>
<td>Durelon (polyacrylic acid)</td>
<td>358558</td>
<td>3M/ESPE, St Paul, MN, USA</td>
</tr>
<tr>
<td></td>
<td>Pumice flour</td>
<td>M01771</td>
<td>Quadra Chemicals Ltd, Vaudreuil-Dorion, QC, CA</td>
</tr>
<tr>
<td></td>
<td>Aluminum oxide</td>
<td></td>
<td>Opiblast, Buffalo Dental Mfg Inc, New York, NY, USA</td>
</tr>
</tbody>
</table>

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RESULTS

Mean shear bond strengths and standard deviations for each treatment group are presented in Table 3 and Figure 1. Data analysis with ANOVA revealed that specimens sandblasted with aluminum oxide produced the highest mean shear bond strength values (11.3 ± 1.70), and those cleaned with 0.12% chlorhexidine digluconate resulted in the lowest shear bond strength values (6.37 ± 1.47). The mean shear bond strength of the aluminum oxide group was significantly higher than that of any other treatment group (p<0.05). Results also showed that the mean shear bond strength of specimens cleaned with chlorhexidine digluconate was significantly lower than those of the polyacrylic acid and aluminum oxide groups (p<0.05). When all dentin-cleansing agents were compared, no significant differences were found between the control (excavator), chlorhexidine digluconate, and pumice groups (p>0.05), or between the chlorhexidine, pumice, and polyacrylic acid groups (p>0.05).

DISCUSSION

The use of dentin-cleaning techniques to avoid any contaminants along the dentin-cement interface to improve the bond strength of self-adhesive luting systems to the dentin surface seems to be a desirable procedure. In the present study, chemical and mechanical cleansing protocols were tested using shear forces, because shear stress tends to provide a better representation of the forces capable of displacing crowns in the oral environment when compared with tensile stress. It was hypothesized that different dentin-cleansing techniques would yield statistically significant differences in shear bond strength values of a self-adhesive resin cement to dentin. Results of this

Table 3: Mean of Bond Strength Values (MPa) of Different Dentin Surface Treatments

<table>
<thead>
<tr>
<th>Group</th>
<th>Dentin-Cleansing Technique</th>
<th>No. of Specimens</th>
<th>Mean Shear Bond Strength, MPa* (mean ± SD)**</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>Hand instrument (control)</td>
<td>13</td>
<td>7.74 ± 1.72ab</td>
</tr>
<tr>
<td>G2</td>
<td>0.12% chlorhexidine digluconate</td>
<td>13</td>
<td>6.37 ± 1.47b</td>
</tr>
<tr>
<td>G3</td>
<td>40% polyacrylic acid</td>
<td>13</td>
<td>9.14 ± 2.11b</td>
</tr>
<tr>
<td>G4</td>
<td>Pumice flour + water</td>
<td>13</td>
<td>7.33 ± 2.85ab</td>
</tr>
<tr>
<td>G5</td>
<td>Aluminum oxide, 50 μm Al₂O₃</td>
<td>13</td>
<td>11.3 ± 1.70c</td>
</tr>
</tbody>
</table>

* Mean MPa with the same letters indicate groups that were not statistically different (p>0.05).
** Data analysis was done using ANOVA and the Tukey-B rank order test.

Figure 1. SEM micrograph of dentin surface after cleansing treatment: G1—excavator (control).
study partially support the hypothesis, in that a significant difference in shear bond strength was observed between group 5 (aluminum oxide) and all other groups. The use of aluminum oxide particles significantly improved the bond strength of a self-adhesive cement to dentin \((p<0.05)\). Particle abrasion using aluminum oxide particles is a dentin cleansing technique that has only recently regained attention in operative dentistry. It is a relatively old technique that is widely used by prosthodontists and dental technicians to increase surface roughness and enhance adhesion.\(^{10}\) Specimens abraded with aluminum oxide showed the highest shear bond strength values compared with any other cleansing technique. This result is consistent with a previous study\(^{21}\) showing significantly higher bond strength when aluminum oxide particles were used for dentin surface treatment before indirect restorations were cemented with a self-adhesive resin luting cement, compared with pumice and hand instruments. In their study, tooth surfaces were treated with 50 and 27 \(\mu m\) aluminum oxide particles. Investigators observed that although the smaller particles promoted a more retentive pattern, particle size did not significantly influence bond strength. In the present study, 50 \(\mu m\) aluminum oxide particles were used and presented significantly higher bond strength compared with all other groups. Representative areas of the dentin surfaces treated with different cleaning procedures are shown in Figures 1-5.

The high shear bond strength observed in this group can be attributed to the fact that particle abrasion using aluminum oxide creates rough, irregular surfaces that increase the bonding surface area, as visualized on scanning electron micrographs. This, in turn, has been reported to increase the bond strength of restorations to both enamel and dentin.\(^{22}\) It is important to mention that the use of rubber dam isolation and of a high-volume evacuation system is recommended, to avoid inhalation of alumina particles during clinical procedures.

In contrast to aluminum oxide, dentin treatment with slurry of pumice resulted in significantly lower shear bond strengths and no significant differences compared with other tested groups. The use of pumice flour to remove plaque and/or surface debris is a well-known procedure that has been used in dentistry for many years. It has been used as an abrasive or polishing agent to clean teeth and
remove plaque and debris from the area to be bonded. Investigators have attained variable results regarding the use of pumice to improve bond strength. Some support the use of pumice for cleaning provisional cement on dentin; others have reported otherwise. The latter study showed that pumice is not effective as a dentin-cleansing agent, as indicated by the low shear bond strength values achieved. A previous study similarly observed that use of pumice on a flat tooth surface produces a surface covered by pumice residues condensed by the rubber cup, which negatively interferes with adhesion. Sealed dentinal tubule openings can prevent the impregnation of resin and can reduce effective bonding.

In another study, Saraç and others observed by SEM that when a rotary instrument was used to apply cleaning agents to dentin, dentinal tubules were plugged with provisional cement by the force of rotation, reducing the surface area for micromechanical interlocking. In the present study, pumice slurry was applied using a prophy cup in a rotary instrument. The rotary instrument could have smeared the remnants of provisional cement on the dentin surface, thereby yielding low shear bond strength values for pumice as compared with aluminum oxide.

Chlorhexidine is a chemical agent that has been indicated for tooth surface cleaning because of its antibacterial action, and also because of its effect as a matrix metalloproteinase (MMP) inhibitor. Recently, chlorhexidine has been shown to provide structural integrity to hybrid layers through its inhibitory effect on endogenous collagenolytic activity in dentin, preventing collagen degradation and disintegration of the bonding interface over time. However, besides the potential advantages of using this substance for dentin pretreatment, previous studies have reported conflicting results, such as reduced bond strength, with its use, and others observed no difference in bonding strength values.

In the present study, no statistically significant differences were found between the control (excavator), chlorhexidine digluconate, and pumice groups (p>0.05). The similarity in effects of chlorhexidine digluconate and a hand instrument on dentin cleaning has been documented in other studies.
Grasso and others\textsuperscript{2} reported no statistical difference when 0.12\% chlorhexidine digluconate, an explorer, and air-water spray were used to remove provisional cement from the dentin surface. In another study by Saraç and others,\textsuperscript{9} no significant difference was observed between shear bond strengths of specimens cleaned with a hand instrument and a cavity cleanser (2\% chlorhexidine digluconate).

The low shear bond strength values obtained with the use of chlorhexidine solution indicate that this cleaning agent is not effective when used to clean provisional cement off dentin before self-adhesive cement is applied. This finding is in agreement with provisional cement off dentin before self-adhesive cleaning agent is not effective when used to clean the use of chlorhexidine solution indicate that this cleanser (2\% chlorhexidine digluconate).

Cleaned with a hand instrument and a cavity observed between shear bond strengths of specimens liquid) was used to clean a normal dentin surface, reported that when 40\% polyacrylic acid (Durelon acid was used to verify whether this weak acid would progress\textsuperscript{21}; however, insufficient demineralization and limited resin infiltration have been observed.\textsuperscript{5,14,18,31} In the present study, 40\% polyacrylic acid was used to verify whether this weak acid would provide further demineralization to enhance bonding. The beneficial effect of polyacrylic acid on bond strength to dentin lies in the removal of the smear layer and the shallow demineralization of the tooth tissue.\textsuperscript{32} Although results of this study show that the polyacrylic acid group was superior to the chlorhexidine digluconate group, the use of polyacrylic acid did not improve shear bond strength compared with the control group. This finding is consistent with data from other studies.\textsuperscript{5,14}

Although polyacrylic acid increases surface roughness and exposes dentinal tubules, limited diffusion of the viscous-filled self-adhesive resin cement was observed in a recent study.\textsuperscript{14} A previous study\textsuperscript{3} reported that when 40\% polyacrylic acid (Durelon liquid) was used to clean a normal dentin surface, dentinal tubule openings were clearly visible and the smear layer of dentin was significantly reduced. However, in that study, it was shown that when this acid was used to clean provisional cement, the effect was significantly decreased. The explanation relies on the presence of a provisional cement layer that reduces the action of Durelon liquid. This finding also helps to clarify the poor effects of polyacrylic acid on bond strength, as observed in the present study. In addition, the absence of a low-viscosity agent to facilitate diffusion into the demineralized dentin may contribute to the lower bond strength.\textsuperscript{14}

This \textit{in vitro} study used a predetermined protocol with each cleaning agent; this was a limitation of the study. Several factors can alter the effectiveness of dentin-cleaning agents used in this study. Some of these include time of application, application protocol, and concentration of solutions. Varying previously mentioned conditions can alter the performance of these cleaning agents. This is an area that needs further research to specify the conditions under which each dentin-cleaning agent will have an optimal effect on the provisional cement and the smear layer. Results may more closely reflect the reality of clinical practice.

**CONCLUSIONS**

Within the limitations of this \textit{in vitro} study, the highest mean shear bond strength values of a self-adhesive resin luting cement were achieved when dentin was sandblasted with aluminum oxide.

On the contrary, the use of chlorhexidine digluconate as a dentin-cleaning agent resulted in the lowest shear bond strength values with no significant difference from the control group (excavator).

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**REFERENCES**


