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

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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 noncarious 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

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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, and type of the definitive luting agent.[1,2] The main objective of restorative procedures is to obtain an adaptation as close as possible between the restorative material and the tooth structure to avoid the presence of gaps and consequent microleakage.[3] Resin-based dental luting cements can infiltrate into the dentinal tubules and exposed collagen network to promote a micromechanical interlock.[4,5] Also, because of the tooth-colored appearance, minimal solubility, biocompatibility, and strengthening effects to the remaining dental structure provided by resin luting materials, their use has been increasing over the past decade.[6]

Despite the positive aspects of resin luting cements, the main disadvantage of the adhesive cementation technique is the number of steps involved in the luting protocol. For longer than 20 years, conventional resin cements have been used in conjunction with dentin bonding agents; this has resulted in a multistep application technique that is considered time-consuming and technique-sensitive.[7] Also, the discrepancy between the depth of acid etching and resin infiltration can lead to postoperative sensitivity and hydrolytic degradation because of the large area of collagen fibrils exposed but not encapsulated by the bonding resin.[8] A resin cement that combines pretreatment of dental tissues and resin infiltration in a single application would be advantageous because it may overcome some of the limitations associated with a multistep technique.[3] The introduction of self-adhesive resin cements was a major advance in dental adhesive cementation early in the decade, because they do not require additional steps of etching, priming, or bonding; instead, their application is accomplished through a single clinical step, which allows the clinician to use a cementation protocol very similar to that used with conventional zinc-phosphate and polycarboxylate cements. Self-adhesive cements are based on multifunctional phosphoric acid methacrylates, which demineralize and infiltrate the tooth structure, resulting in micromechanical retention.[6]

Regardless of the luting protocol of resin cements, the bonding technique can be adversely affected by the presence of remnants of provisional restorative materials. Removal of provisional cement debris is desirable to promote contact between the dentin and 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 superficially with dentin, leading to partial demineralization of the smear layer, which would result in a weak bonding mechanism.[18] 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/cm^2 output). Polymerization was completed in an oven (Dentacolor XS, Kulzer, Irvine, CA) at 120°C for seven minutes. RBC surfaces were sandblasted with 50 μm Al_2O_3 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/cm^2 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

Table 1: *Experimental Groups With Cleansing Treatments*

Group	Dentin-Cleansing Technique	Treatment
G1	Hand instrument (control)	Excavator was used for 10 seconds; dentin was rinsed and excess water removed with absorbent paper[a]
G2	0.12% chlorhexidine digluconate	Scrubbed over dentin for 10 seconds using a disposable applicator and gently dried with absorbent paper[a]
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[a]
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[a]
G5	Aluminum oxide, 50 μm Al_2O_3	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[a]

[a] *To avoid dehydration of the dentin.*

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 2: *Product Names, Batch Numbers, and Manufacturers*

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

Table 3: *Mean of Bond Strength Values (MPa) of Different Dentin Surface Treatments*			
Group	**Dentin-Cleansing Technique**	**No. of Specimens**	**Mean Shear Bond Strength, MPa* (mean ± SD)****
G1	Hand instrument (control)	13	7.74 ± 1.72[ab]
G2	0.12% chlorhexidine digluconate	13	6.37 ± 1.47[a]
G3	40% polyacrylic acid	13	9.14 ± 2.11[b]
G4	Pumice flour + water	13	7.33 ± 2.85[ab]
G5	Aluminum oxide, 50 μm Al_2O_3	13	11.3 ± 1.70[c]

** 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.*

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-cleaning 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.[19] Furthermore, the shear bond test is considered a reliable and convenient method of accessing the bond strength of luting materials.[20]

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

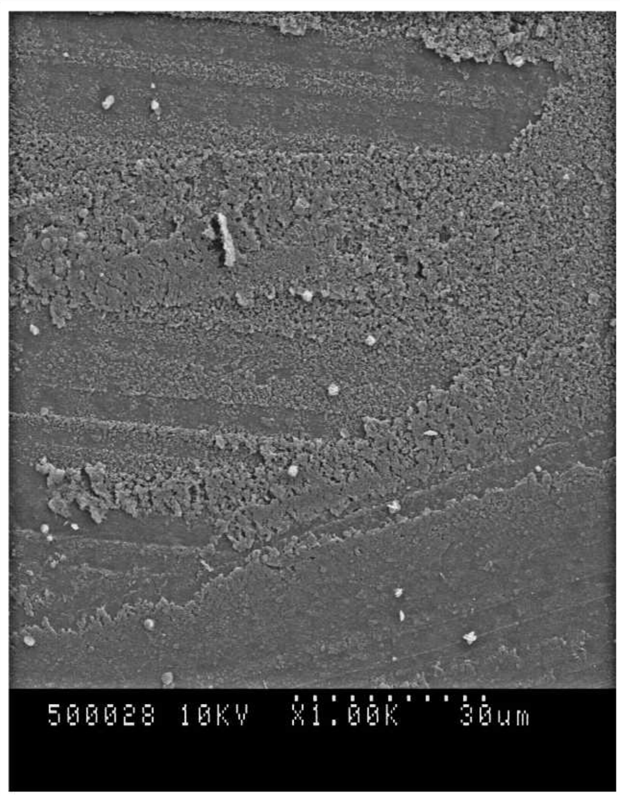

Figure 1. *SEM micrograph of dentin surface after cleansing treatment: G1—excavator (control).*

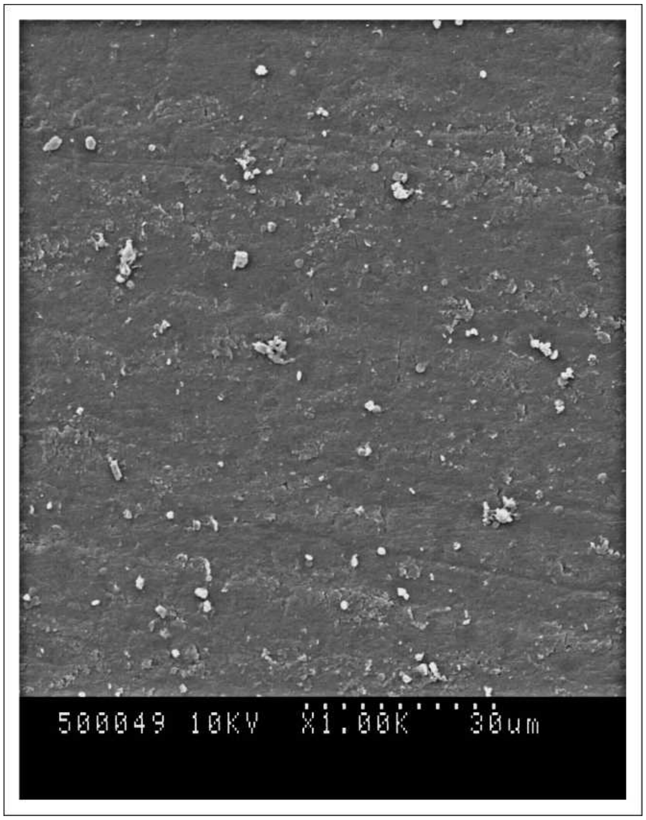

Figure 2. *SEM micrograph of dentin surface after cleansing treatment: G2—0.12% chlorhexidine digluconate.*

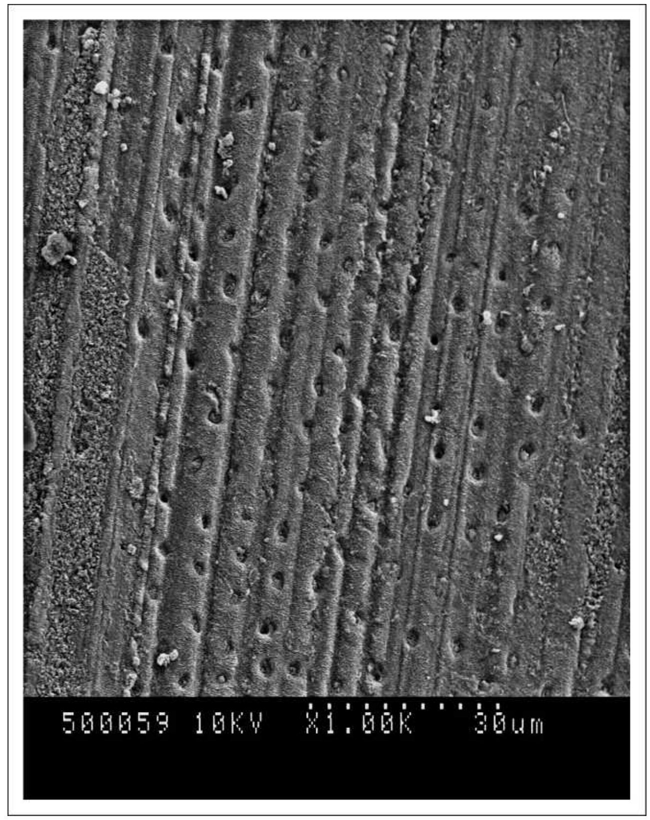

Figure 3. *SEM micrograph of dentin surface after cleansing treatment: G3—40% polyacrylic acid.*

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 μm 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 μm 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

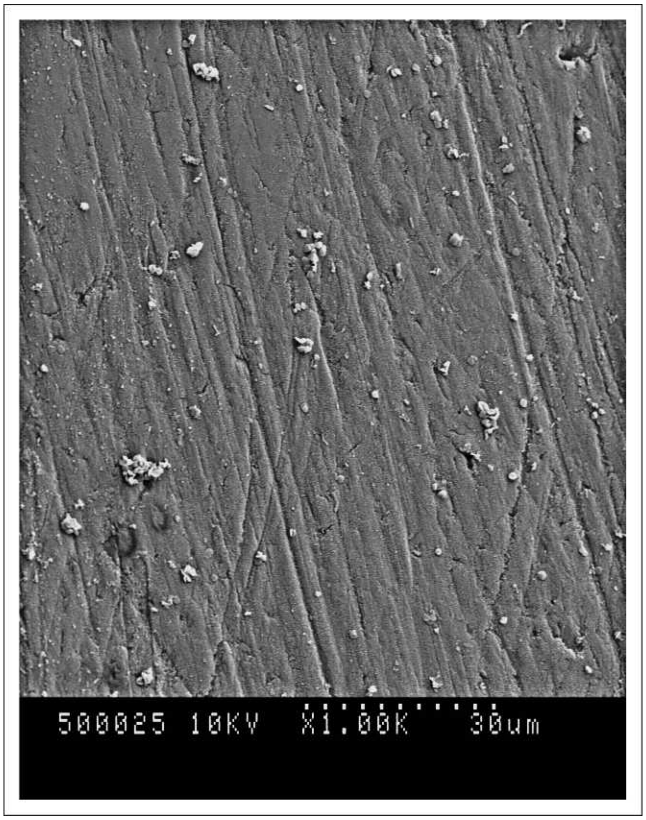

Figure 4. *SEM micrograph of dentin surface after cleansing treatment: G4—pumice slurry.*

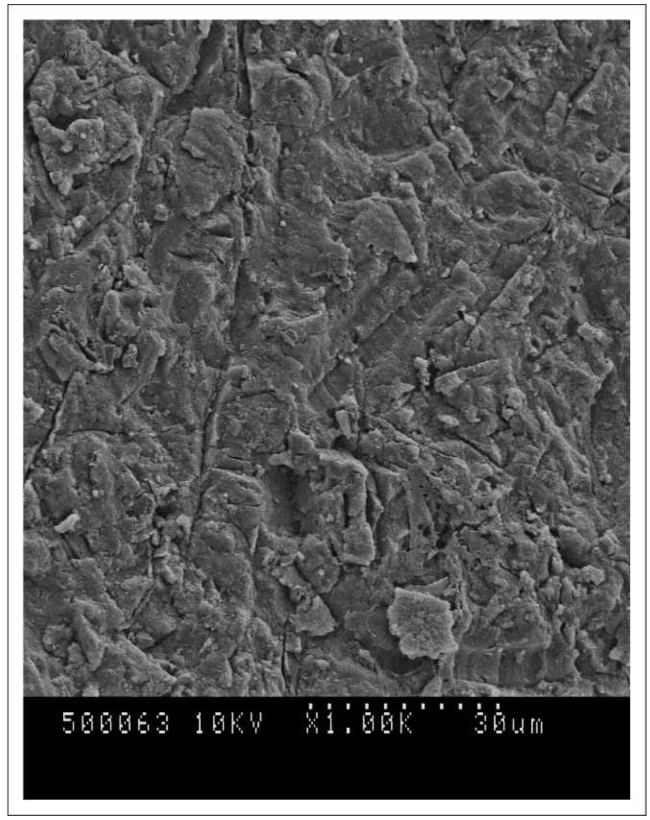

Figure 5. *SEM micrograph of dentin surface after cleansing treatment: G5—sandblasting with alumina oxide 50 μm.*

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[3,23] on dentin; others have reported otherwise.[20,24] 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.[25] Sealed dentinal tubule openings can prevent the impregnation of resin and can reduce effective bonding.

In another study, Saraç and others[6] 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.[26] 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.[27] 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[6,28]; others observed no difference in bonding strength values.[29,30]

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[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,[6] 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 data from a recent study[31] demonstrating that the use of chlorhexidine solution reduced the bond strength of self-etch resin cement systems, because this substance is not effective in removing the smear layer. Also, because of its affinity for phosphate groups (cationic properties), the bonding of chlorhexidine to these loose apatite remnants within the smear layer could have interfered with the functions of acidic monomers of self-adhesive resin cements. In the same study, no detrimental effect on bonding efficacy was observed when chlorhexidine was applied to acid-etched dentin, followed by application of total-etch adhesive systems.

According to the information available on self-adhesive cement, the presence of water, phosphoric acid, and methacrylate monomer 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 acid that occurs as polymerization progresses[21]; however, insufficient demineralization and limited resin infiltration have been observed.[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.[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.[3,14]

Although polyacrylic acid increases surface roughness and exposes dentinal tubules, limited diffusion of the viscus-filled self-adhesive resin cement was observed in a recent study.[14] A previous study[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.[14]

This *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 *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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