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Laboratory Research

The Effect of Tooth-preparation Cleansing Protocol on the Bond Strength of Self-adhesive Resin Cement to Dentin Contaminated with a Hemostatic Agent

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Clinical Relevance

After contamination with hemostatic agents, tooth-preparation cleansing protocols using either particle abrasion with low-pressure aluminum oxide particles or phosphoric acid-etching restored bond strengths to pre-contamination levels for a self-adhesive resin cement.

ABSTRACT

This study evaluated the effect of tooth-preparation cleansing protocols on the bond strength of a self-adhesive resin cement to dentin contaminated with two different types of hemostatic agents. The occlusal surface of extracted third molars was flattened to expose the dentin surface and prepared for a full crown. Acrylic temporary crowns were fabricated and placed using temporary cement. The specimens were stored at 100% relative humidity for seven days. Following removal of the temporary crowns, the specimens were surface debrided using aluminum oxide abrasion with a particle size of 27 µm at 40 psi. The specimens were randomly assigned to three groups, according to the hemostatic agents: Group I–an agent containing aluminum chloride was applied to the tooth surface; Group II–an agent containing ferric sulfate was applied to the tooth surface and Group III–uncontaminated (control). The contaminated specimens were then further subdivided into three subgroups (A–C; n=12): Group A–tooth surface cleansing with water spray; Group B–tooth surface cleansing with phosphoric acid etch and Group C–tooth

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DOI: 10.2341/09-308-LR1

surface cleansing with aluminum oxide abrasion with a particle size of 27 µm at 40 psi. Ceramic blocks were treated with a 9.5% hydrofluoric acid-etch and silanized prior to being cemented with self-adhesive resin luting agent (RelyX Unicem) to the prepared dentin. The shear bond strength was determined at a crosshead speed of 0.5 mm/minute. The data were analyzed with two-way ANOVA, followed by the Duncan multiple range test, to determine any significant differences between the testing groups. The microstructure morphology of the tooth surface was evaluated using SEM analysis. The results revealed that there was a significant difference between the bond strength of the control and the contaminated testing groups ($p<0.05$). A tooth preparation cleansing protocol using particle abrasion with low-pressure aluminum oxide particles provided a significant improvement in bond strength to contaminated dentin, while rinsing with water spray resulted in the lowest mean bond strength of the self-adhesive resin cement to dentin ($p<0.05$).

INTRODUCTION

The longevity of restorations is influenced by the tooth preparation design, type of luting agents and tooth surface management. In general, during the definitive cementation procedure, the prepared tooth should be free of contaminants in order to achieve a lasting bond between the luting agent and tooth structure.[1] However, appropriate contamination control might not always occur, especially near or along the gingival margin. Blood and gingival crevicular fluid due to gingival inflammation may be present on the prepared tooth surface.

The most common procedures used to control bleeding and decrease the flow of gingival fluid involve the use of a topical hemostatic agent. This procedure is important for avoiding any blood contamination along the interface between the prepared tooth surface and the luting agent. A hemostatic agent containing an aqueous solution of aluminum chloride ($AlCl_3$) is frequently used in restorative dentistry. Aluminum compounds with a concentration range of 20% to 25% have the ability to precipitate protein, constrict blood vessels and extract fluid from tissue.[2] Ferric salts, such as ferric subsulfate, ferric sulfate and ferric chloride, have also been used as hemostatics and astringents. One such hemostatic agent consists of a 13% to 15% solution of ferric sulfate ($Fe_2(SO_4)_3$) in glycol and water. It has been reported that this solution is effective in coagulating fresh blood in the gingival area without adverse side effects or staining of the teeth.[3] Most hemostatic agents are soluble in water,[4] therefore, vigorous rinsing with water spray has been recommended prior to any bonding procedure.

However, several studies indicate that hemostatic agents can remain on the tooth surface and cause a significant decrease in bond strength, especially with a self-etch adhesive system.[5-6] It is believed that contaminants obstruct the flow of resin monomer into the dentinal tubules, as small contaminant particles penetrate the dentinal tubules and ultimately affect the development of the hybrid layer.[7] In addition, previous studies have demonstrated that hemostatic agents are highly acidic, with pH values varying from 0.7-3.0.[5,8-9] The acidity of these solutions may contribute to a change in the dentin surface.[10]

Different tooth preparation cleansing methods have been proposed for removing debris or remnants from the dentin surface, including chemical cleansing agents and a mechanical cleansing protocol. Acid etching, which is used to demineralize the dentin,[11] has been reported to dissolve microscopic remnants from the dentin surface.[12] A previous study demonstrated that saliva, blood and handpiece lubricant can be decontaminated by re-etching with phosphoric acid. Without an additional mechanical cleansing protocol, the bond strengths of this re-etched surface were similar to uncontaminated enamel and dentin.[13]

Recently, the use of a low-pressure aluminum oxide particle abrasion has been reported as an effective mechanical cleansing surface treatment in removing residual provisional luting agents.[14] The microparticle abrasion technique has been described as an alternative method of tooth structure pretreatment that minimizes the vibration, pressure and heat associated with rotary cutting instruments. Kinetic energy, generated by a high-velocity stream of aluminum oxide particles, is used to prepare dentin and enamel, while having only a slight impact on softer materials, such as gingival tissue.[15] Particle abrasion creates rough and irregular surfaces that increase the bond strength of restorations to enamel and dentin.[16] However, using this technique to decontaminate hemostatic agents has limited documentation.

The current study evaluated the effect of tooth preparation cleansing protocols on the bond strength of self-adhesive resin cement to dentin contaminated with two different types of hemostatic agents (containing either aluminum chloride or ferric sulfate). The null hypothesis was that there is no difference in bond strength based on different tooth preparation cleansing protocols used with different types of hemostatic agents.

METHODS AND MATERIALS

Extracted intact human third molars were selected based on the inclusion criteria that there was no evidence of caries, no restorations and a lack of any cracks or fractures in the crown. The bucco-lingual dimensions of the teeth were measured using a digimatic caliper that is accurate to within 0.001 of an inch (Mitutoyo series 551; Mitutoyo USA, Aurora, IL, USA). Three measurements were made at the greatest bucco-lingual and mesial-distal widths of the specimens, with the average being determined in each dimension. 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. During preparation, each tooth was wrapped in water-moistened gauze. Individually, each tooth was mounted in a copper cylinder with the buccal cementoenamel junction 3 mm above the top of the copper cylinder. These teeth were each attached with wax to a dental surveyor rod with the long axis of the tooth parallel to the surveyor rod. They were then lowered into the copper cylinder and positioned at its center. Premixed autopolymerizing resin (GC Pattern Resin, GC America, Scottsdale, AZ, USA) was injected into the cylinder until the cylinder was completely full. After the resin had set, the occlusal surfaces of the teeth were flattened to expose the dentin surface. The axial surfaces of the teeth were flattened parallel to their long axes. The teeth were then prepared for full crowns with chamfer margins using a high-speed handpiece and diamond bur (KS-1, Brasseler USA, Savannah, GA, USA). The crown preparations were done to simulate the treatment of fixed partial dentures (single-unit or multiple-unit restorations). Temporary luting cement (Temp Bond, Kerr Co, Orange, CA, USA) was placed in provisional crowns (Temporary Crown and Bridge Resin, Dentsply Caulk Inc, Milford, DE, USA) and the crowns were seated on the prepared teeth. The excess provisional luting cement was removed and the specimens were stored in 100% relative humidity at room temperature. The specimens were kept for seven days prior to bonding with ceramic, as this period is reasonable for a provisional crown placed in a clinical situation. After seven days, the provisional crowns were removed, along with the remaining cement particles. Subsequently, the surfaces of the teeth were debrided using aluminous oxide abrasion with a particle size of 27 µm at 40 psi from a distance of 2 mm (PrepStart, Danville Material, San Ramon, CA, USA).[14] The teeth were then rinsed and gently dried.

The specimens were randomly divided into three groups: Group I–prepared for an application of hemostatic agent containing aluminum chloride (Viscostat Clear, Ultradent Inc, South Jordan, UT, USA) (Table 1); Group II–prepared for an application of hemostatic agent containing ferric sulfate (Ferric Subsulfate Dental Gel, Beall's Pharmacy Inc, Puyallup, WA, USA) and Group III–no contamination (control) (Figure 1). For Groups I and II, blood was applied to the prepared tooth surface. Blood was collected from a single individual (needle-prick of an alcohol-wiped forefinger) and collected at the time of experiment. It has been shown that freshly drawn capillary blood is more suitable in laboratory experiments involving blood contamination than heparinized blood.[17] Hemostatic agents were then applied to the blood-contaminated tooth surface for five minutes.[9-10] The contaminated specimens were then further subdivided into three subgroups (A-C; n=12) for a tooth surface cleansing treatment: Group A–a vigorous water spray was used to rinse off the hemostatic agent contamination for 20 seconds, followed by lightly drying with air free of water and oil for 20 seconds. The dentin surfaces remained visibly moist; Group B–the dentin surface was etched for 15 seconds with 37% phosphoric acid (Scotchbond Etchant gel, 3M ESPE, St Paul, MN, USA) and rinsed for 10 seconds. Excess water was blot-dried from the surface to achieve moist dentin and Group C–the dentin surfaces were mechanically debrided using aluminous oxide abrasion with a particle size of 27 µm at 40 psi at a distance of 2 mm (PrepStart, Danville Material, San Ramon, CA, USA) and thoroughly rinsed using water spray. Excess water was blot-dried from the surface to achieve moist dentin.

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 ceramic bars. The ceramics were fired in a furnace (Dekema Austromat 3001, Frankfurt, Germany) at 905°C with a ramp rate of 75 seconds and a holding time of 120 seconds. The ceramic bars were glazed and polished with 1 µm diamond paste (Ecomet 3, Buehler Ltd, Lake Bluff, IL, USA) to create a flat surface. The ceramic bars were cut into small blocks (L: 4.0 ± 0.2 x W: 4.0 ± 0.1 x H: 2 ± 0.1 mm). This was performed using diamond disks (Brasseler USA, Savannah, GA,

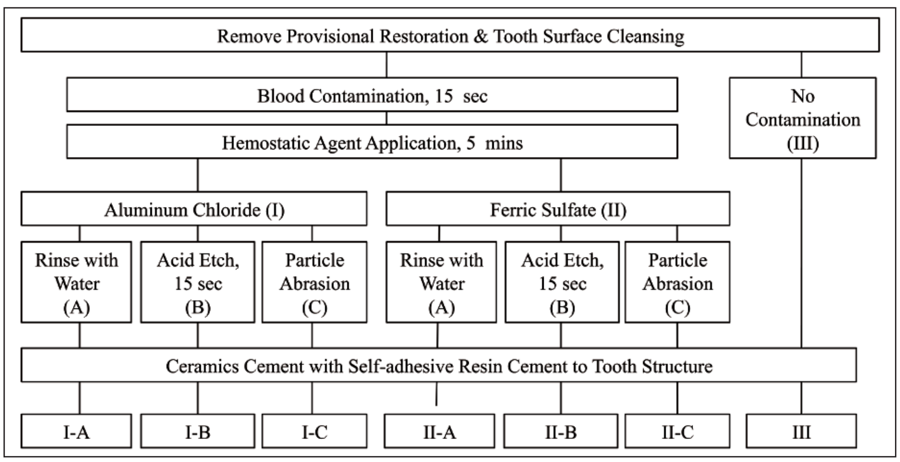

Figure 1. *Schematic diagram representing the experimental procedure and testing groups in this current study.*

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

Materials and Application		Composition
Hemostatic agents	ViscoStat Clear (Ultradent Products, Inc, Lot #B2M5Y): applied for five minutes	25% Aluminum chloride
	Ferric Subsulfate Dental Gel (Beall's Compounding Pharmacy, Lot #0610512009): applied for five minutes	13% Ferric Sulfate, flavored sweetened oral hemostatic
Materials used for provisional restoration	Temporary Crown and Bridge Resin (Dentsply Caulk Inc, Lot #050720): placed on prepared tooth for seven days	Methyl Methacrylate Monomers, polymerizable dimethacrylate resin, stablizers
	Temp Bond (Kerr Co, Lot #6-1018): placed on prepared tooth for seven days	Accelerator: 4-Allyl-2-Methoxyphenol (Eugenol, Oil of Cloves) (28-32%wt) Base: Mineral oil, zinc oxide, cornstarch
Materials used for tooth surface cleansing	Scotchbond Etchant Gel (3M ESPE, Lot #8LL): applied for 15 seconds	37% Phosphoric acid
	PrepStart Kit (Danville Material, Lot #200259-90): applied at 40 psi at a 2 mm distance	Handpiece with nozzle tip of 0.015 inch, an aluminous oxide powder of particle size of 27 µm
Materials used for ceramic surface treatment	Reflex Ceramic (Wieland Dental Systems Inc, Lot #83001510	Feldspathic ceramic, leucite (17-25% mass), Colorants, Opacifers, High-melting glass particles
	Ultradent Porcelain Etch Gel (Ultradent Products, Inc, Lot #X063): applied for 180 seconds	9.5% Hydrofluoric acid
	RelyX Ceramic Primer (3M ESPE, Lot #5WU): applied for 60 seconds and air-dried	Ethyl alcohol (70-80%wt), water (20-30%wt), methacry loxypropyltrimethoxysilane (<2%wt)
Self-adhesive luting agent	RelyX Unicem Aplicap (3M ESPE, Lot #310334): mixed under Rotomix mixing machine and applied to both prepared tooth and ceramic	Powder: Silanized glass powder (85-95%wt), silane treated silica (5-10%wt), Calcium hydroxide (1-5%wt), substituted pyrimidine (1-5%wt), sodium persulfate (<1%wt) Liquid: Methacrylated phosphoric esters(40-50%wt), triethylene glycol dimethacylate (25-35%wt), substituted dimethacrylate (22-34%wt)

USA) to avoid causing any damage on the ceramic surface that already had been glazed and prepared for bonding to dentin. The ceramics were treated with 9.5% hydrofluoric acid (Ultradent Porcelain Etch Gel, Ultradent Products, Inc) according to the manufacturer's protocol. A silane solution (RelyX Ceramic Primer, 3M ESPE) was applied on the ceramic surface for 60 seconds, then gently air-dried. The ceramics were then bonded to the dentin surface of the prepared teeth using self-adhesive resin cement (RelyX Unicem Aplicap, 3M ESPE). An Aplicap capsule of RelyX Unicem was inserted into the Aplicap activator. The lever arm was pushed down to release the acid, and the capsule was immediately placed in the mixing machine (Rotomix, 3M ESPE) according to the manufacturer's protocol. The mixed cement was directly applied to both the prepared tooth and ceramic. The ceramic was seated on the flattened axial dentin surface with finger pressure and the excess cement removed with an explorer after the initial setting. Most of the self-adhesive resin cements have been developed as dual-cured adhesive-luting cements for metal-ceramic or all-ceramic indirect restorations. Although light activation through the translucent restorative materials is recommended, a slightly different situation exists for cementation of metal-ceramic restoration or thick layers of all-ceramic restorations, where light access through restorations is limited and self-cure (chemical reaction) is required. In the current study, the specimens were not light-activated to simulate the clinical situation for these types of restoration. The bonded specimens were then stored at room temperature in 100% relative humidity for 48 hours prior to shear bond strength testing. It has been demonstrated that, at room temperature (20°C), the mechanical properties of dental restorative resin are similar to those at body temperature (37°C).[18-19]

Each specimen was mounted onto a metal holder in the universal testing machine (Instron model 5585H, Instron Corp, Canton, MA, USA) equipped with a 1-kN

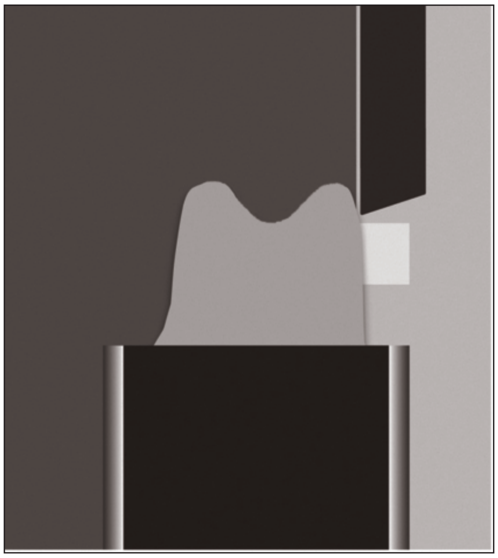

Figure 2. *Schematic diagram representing the test assembly for determining shear bond strength.*

load cell at a crosshead speed of 0.5 mm/minute. All the specimens were tightened and stabilized to ensure that the edge of the shearing rod was positioned as close to the ceramic-tooth interface as possible (Figure 2). A shear load was applied until failure occurred. The ultimate load to failure was recorded in newtons (N). The average bond strength (MPa) was calculated by dividing the maximum ultimate load to failure (N) by the bonded cross-sectional area (mm^2). The means and standard deviations were recorded. The fractured surfaces were then examined under an optical microscope (Wolfe Microscope, Carolina, Burlington, NC, USA) to obtain the mode of failure. The failed specimens were classified into five types based on the mode of failure: Type I–adhesive failure at the tooth/cement interface, mostly cement (more than 75%) remains on the prepared tooth; Type II–adhesive failure at the tooth/cement interface, all the cement remains on the ceramic; Type III–adhesive failure at the tooth/cement interface, mostly cement (more than 75%) remains on the ceramic; Type IV–cohesive failure, tooth fracture; Type V–cohesive failure, ceramic fracture. The data were subjected to two-way analysis of variance (ANOVA), followed by the Duncan multiple range tests to define significant differences at a confidence level of 95%. This determined whether significant differences existed in the bond strength among the testing groups. Randomly selected specimens were scanned by a Scanning Electron Microscope (SEM) to evaluate the microstructure morphology of the tooth surface.

RESULTS

The means of the bond strength are presented in Table 2. Two-way analysis of variance revealed a significant difference among the mean bond strengths of the groups (Table 3). A significant effect of tooth prepara-

Table 2: *Means of the Bond Strength Values (MPa), Respective Standard Deviation (± SD) of the Different Hemostatic Agents, Different Tooth Surface Treatments and the Percentage of Failure Mode*

Group	Bond Strength (MPa	Percentage of Failure Mode				
		Adhesive Failures			Cohesive Failures	
		Type I	Type II	Type III	Type IV	Type V
I-A	6.49 ± 1.42[b]	-	100.0	-	-	-
I-B	8.60 ± 2.82[a]	-	41.7	58.3	-	-
I-C	9.39 ± 2.29[a]	-	66.7	25.0	-	8.3
II-A	7.27 ± 2.92[b]	-	91.7	8.3	-	-
II-B	9.04 ± 2.88[a]	-	75.0	25.0	-	
II-C	9.70 ± 2.39[a]	-	50.0	33.3	-	16.7
III	10.96 ± 2.65[a]	8.3	58.4	25.0	-	8.3
	**Different letters indicate statistically different means.*	*Type I–adhesive failure at the tooth/cement interface, cement mostly (more than 75%) remains on prepared tooth; Type II–adhesive failure at the tooth/cement interface, all cement remains on the ceramic; Type III–adhesive failure at the tooth/cement interface, cement mostly (more than 75%) remains on the ceramic; Type IV–cohesive failure, tooth fracture; Type V–cohesive failure, ceramic fracture.*				

Table 3: *Two-way Analysis of Variance*

Source	Type III Sum of Squares	df	Mean Square	F	Sig
Corrected Model	97.237	5	19.447	3.098	.014
Intercept	5096.966	1	5096.966	811.935	.000
Tooth surface cleansing protocols (A)	91.753	2	45.877	7.308	.001
Type of hemostatic agent (B)	4.738	1	4.738	0.755	.388
A * B	0.746	2	0.373	0.059	.942
Error	414.319	66	6.278		
Total	5608.522	72			
Corrected Total	511.556	71			

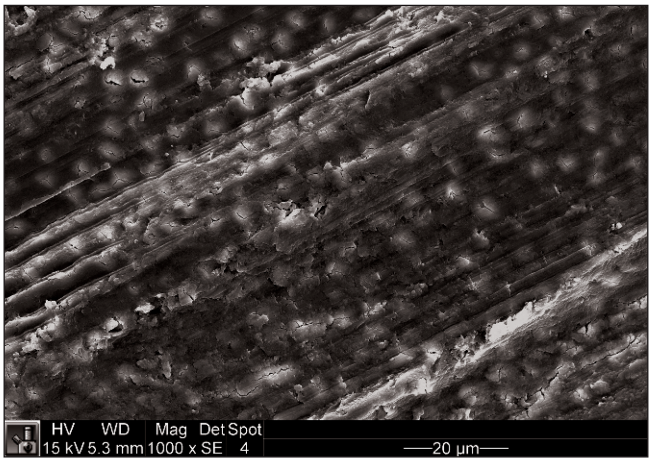

Figure 3. *SEM micrograph of the dentin surface of the tooth preparation contaminated with aluminum chloride after being rinsed with water spray. Note: Partial smear layer removal and etching effect occurs with mainly dentin tubules remaining occluded. (Original magnification 1000x)*

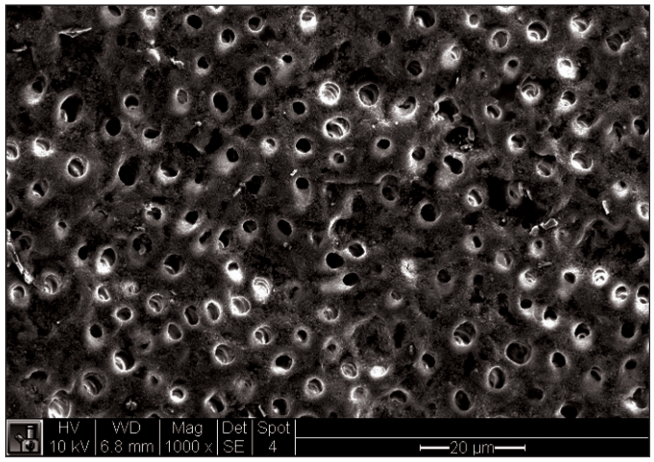

Figure 4. *SEM micrograph of the dentin surface of the tooth preparation contaminated with ferric sulfate after being rinsed with water spray. Note: The dentinal tubules exhibited a more pronounced etching effect with the dentin tubules remaining partially occluded. (Original magnification 1000x)*

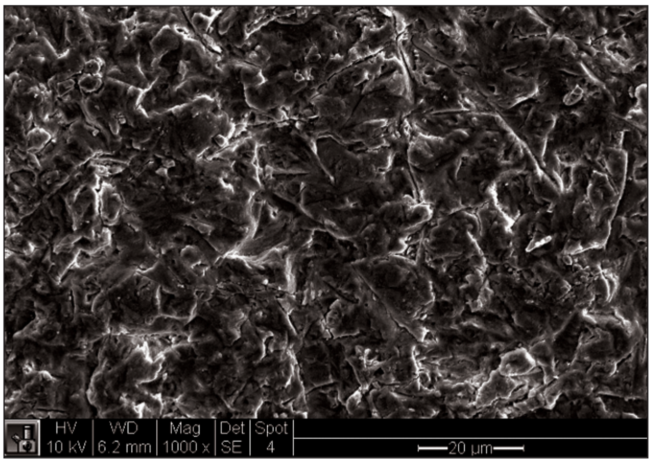

Figure 5. *SEM micrograph of the dentin surface of the tooth preparation after mechanical cleansing with 27 μm Al_2O_3 particle abrasion. (Original magnification 1000x)*

tion cleansing protocols was found; however, there was no significant effect of the different types of hemostatic agents. The interaction between the tooth preparation cleansing protocols and the types of hemostatic agents was not significant. The highest mean bond strength was obtained from uncontaminated dentin (control group). The mean bond strengths of the acid-etch groups and the particle abrasion groups were not significantly different from that of the control group. The group using water rinsing alone provided the lowest mean bond strength for both types of hemostatic agents. This same group exhibited the greatest number of adhesive failures at the dentin interface, while mixed failures were found in groups of phosphoric acid etch and particle abrasion in both types of hemostatic agents.

The morphologic characteristics of the dentin surface following exposure to the hemostatic agent are shown in SEM micrographs. Each hemostatic agent has a distinct effect on the dentin surfaces. For the aluminum chloride application groups, dentinal tubules were mainly occluded with partial smear layer removal (Figure 3). In the ferric sulfide groups, the dentinal tubules exhibited a more pronounced etching effect (Figure 4). Irregular rough surfaces were detected after the particle abrasion surface treatment with the smear plugs occluding the tubule orifices (Figure 5). There was some artifact crack formation found on the surface. These cracks were created during desiccation by the Scanning Electron Microscope.

DISCUSSION

During bonding procedures, isolation of the working field is crucial, as any contamination of a prepared tooth structure might have a detrimental effect on the adhesion and retention of restorative materials, thus influencing the long-term success of restorations.[13,20] The current study investigated the effect of the tooth preparation cleansing protocol on the bond strength of a self-adhesive resin cement to dentin contaminated with two different types of hemostatic agents. The results warrant rejection of the null hypothesis that there is no difference among different procedures, because particle abrasion resulted in significantly higher bond strengths than did water rinsing alone.

It has been reported that the effect of the hemostatic agent varies greatly, depending on the type of bonding agent (etch & rinse or self-etch) and the surface condition of the adherent.[21] A previous study demonstrated that using a self-etch adhesive system decreased the bond strength if contaminated dentin was present. Most self-etch adhesive systems contain a self-etching primer that is a mild acid with a pH of approximately 2.[22] If the etching effect is impeded and the self-etching system cannot completely dissolve or eliminate the remnants of residual contaminants, a decrease in bond strength is observed.[5-7] In the current study, a similar result was found for a self-adhesive resin cement. The use of self-adhesive resin cements has increased in dentistry due to their advantages, including fewer steps in the bonding protocol and the fact that they present a less sensitive technique due to a lack of pretreatment of the tooth structure. According to the information available on the self-adhesive cement used in the current study, the presence of water and phosphoric acid methacrylate monomer in the cement should theoretically demineralize the smear layer and the underlying dentin. Simultaneously, the cement should infiltrate as a result of its hydrophilic properties and neutralization of the acidic reaction that occurs as polymerization progresses (personal communication, 3M ESPE). Any remaining particles from hemostatic agents could interfere with the chemical bond and micromechanical retention of the self-adhesive resin cement, resulting in lower bond strengths.

In the current study, the control group, which was not contaminated, had the highest bond strength, while the tooth preparation cleansing protocol using water rinsing demonstrated the lowest bond strength. The low bond strength of the group rinsed with water was indicated by its mode of failure. Adhesive failures at the dentin interface were primarily found in this group, which might indicate a weakness of the resin-dentin bonded interface. A previous study demonstrated that, when the dentin surface was treated with water rinsing only, a greater amount of aluminum remained on the dentin surface than when the surface was treated with phosphoric acid.[5] As a result, a decrease in bond strength might be the result of remnants of the hemostatic agent on the dentin surface.

The application of phosphoric acid for surface cleansing improved bond strength approximately 25% to 30% over water rinsing alone. This might be explained by the aggressive etching effect of phosphoric acid. With a pH of approximately 0.5,[23] it simultaneously removed components of the dentin surface and possibly eliminated the negative effects of the hemostatic agents.[24-25] Although the bond strength increased over water rinsing alone, it was demonstrated that the bond strength decreased 6% to 8% when compared to particle abrasion surface cleansing and 18% to 21% when compared to the uncontaminated dentin surface. The bonding mechanism of self-adhesive resin cement relies more on chemical bonding than on micromechanical retention.[26] To obtain efficient bonding, the mineral phase of the dentin should be extracted from the dentin substrate without damaging the collagen matrix, and this exposed collagen fibril network should be filled with adhesive resin cement.[27] After applying phosphoric acid, the dentin-etched surface may be too deep to be penetrated by the limited diffusion of a viscous self-adhesive resin cement.[28] Some areas of exposed and non-resin impregnated collagen might have been present at the dentin adhesive junction, and therefore, a decrease in bond strength was observed.

The particle abrasion group for each of the hemostatic agents had a mean bond strength not significantly different from that of the control group. Particle abrasion commonly involves the use of aluminum oxide powder carried in a fine stream of compressed air. As the particles collide with a solid target, in this case, the dentin surface, the kinetic energy of the particles is transferred, resulting in microscopic fractures of the target. The target substance must be hard and brittle enough to cause rapid deceleration of the particles, so that their kinetic energy can cause a destructive collision.[29] As a result, contaminants may be removed from the surface, avoiding the formation of a poor-quality bond.

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. As a result, dentin bond strengths may vary within the same tooth, depending on the bonding site.[30] To reduce those variable factors, the extracted teeth used in the current study were from patients whose ages ranged from 15 to 25 years and were restored within six months of extraction.

The hemostatic agents were allowed to remain on the dentin for five minutes to simulate a typical clinical situation. According to the manufacturers' information, complete removal of the smear layer plug can occur if the hemostatic agent remains in contact with the dentin surface for eight minutes. Previous studies demonstrated that a five-minute exposure to ferric sulfate-containing hemostatic agents resulted in a severe etching pattern on the dentin surface.[9-10] In the current study, this finding is consistent with previous results, as evidenced in the SEM micrographs. For hemostatic agents containing aluminum chloride, the degree of dentin surface change appears to be a function of time.[9] Therefore, the results of this study may be different if the dentin is exposed to aluminum chloride for periods other than five minutes.

There are limitations to the current study. These results only pertain to the materials used in this study, since only two hemostatic agents and one self-adhesive resin cement were tested. Additional *in vitro* studies are required to determine the effect of hemostatic agent contamination on different types of adhesive resin cements. The self-adhesive resin cement was allowed to self-cure (chemical reaction) to simulate a clinical situation for the cementation of metal-ceramic restorations or thick layers of all-ceramic restorations, where light access through the restoration may be limited. The effect of the mode of curing was not determined in the current study. The possible effect of this needs to be determined by future research. Hemostatic agent contamination negatively affects the bond strength of self-adhesive resin cement to dentin if not properly removed prior to cementation. In a clinical situation, thorough rinsing with water may be less effective than additional dentinal surface cleansing, such as particle abrasion with low pressure or etching with phosphoric acid. Alternate materials might be considered if the hemostatic agent contamination cannot be avoided.

CONCLUSIONS

After hemostatic agent contamination, the tooth preparation cleansing protocol should use particle abrasion with low-pressure aluminous oxide particle or phosphoric acid etching to restore bond strength to pre-contamination levels when using a self-adhesive resin cement.

Acknowledgements

The authors greatly appreciate the technical help provided by Mr William Kuykendall at the Department of Mechanical Engineering, University of Washington, the Nanotech User Facility at the University of Washington, a member of the National Nanotechnology Infrastructure Network (NNIN) supported by NSF for technical assistance in SEM analysis and Ms Rachel Peterson for manuscript preparation.

(Accepted 8 November 2010)

References

1. Fischer DE (1998) Tissue management needs for adhesive dentistry now and in the future *Dental Clinics of North America* **42(4)** 595-606.
2. Burrell KH & Glick M (2000) Hemostatics, astringents and gingival retraction cords In: Ciancio SG, ed *ADA Guide to Dental Therapeutics 2nd ed* Chicago American Dental Association 104-118.
3. Csempesz F, Vág J & Fazekas A (2003) *In vitro* kinetic study of absorbency of retraction cords *The Journal of Prosthetic Dentistry* **89(1)** 45-49.
4. Council on Dental Therapeutics of the American Dental Association (1984) Hemostatics and astringents In: *Accepted Dental Therapeutics 40th ed* Chicago American Dental Association 334-341.
5. Kuphasuk W, Harnirattisai C, Senawongse P & Tagami J (2007) Bond strengths of two adhesive systems to dentin contaminated with a hemostatic agent *Operative Dentistry* **32(4)** 399-405.
6. O'Keefe KL, Pinzon LM, Rivera B & Powers JM (2005) Bond strength of composite to astringent-contaminated dentin using self-etching adhesive *American Journal of Dentistry* **18(3)** 168-172.
7. Sung EC, Tai ET, Chen T & Caputo AA (2002) Effect of irrigation solutions on dentin bonding agents and restorative shear bond strength *The Journal of Prosthetic Dentistry* **87(5)** 628-632.
8. Woody RD, Miller A & Staffanou RS (1993) Review of the pH of hemostatic agents used in tissue displacement *The Journal of Prosthetic Dentistry* **70(2)** 191-192.
9. Land MF, Rosenstail SF & Sandrik JL (1994) Disturbance of the dentinal smear layer by acidic hemostatic agent *The Journal of Prosthetic Dentistry* **72(1)** 4-7.
10. Land MF, Couri CC & Johnston WM (1996) Smear layer instability caused by hemostatic agents *The Journal of Prosthetic Dentistry* **76(5)** 477-482.
11. Oliveira SS, Pugach MK, Hilton JF, Watanabe LG, Marshall SJ & Marshall GW Jr (2003) The influence of the dentin smear layer on adhesion: A self-etching primer vs a total-etch system *Dental Materials* **19(8)** 758–767.
12. Toledano M, Osorio R, Perdigão J, Rosales JI, Thompson JY & Cabrerizo-Vilchez MA (1999) Effect of acid etching and collagen removal on dentin wettability and roughness *Journal of Biomedical Material Research* **47(2)** 198-203.
13. Xie J, Powers JM & McGuckin RS (1993) *In vitro* bond strength of two adhesives to enamel and dentin under normal and contaminated conditions *Dental Materials* **9(5)** 295-299.
14. Chaiyabutr Y & Kois JC (2008) The effects of tooth preparation cleansing protocols on the bond strength of self-adhesive resin luting cement to contaminated dentin *Operative Dentistry* **33(5)** 556-563.
15. Black RB (1955) Application and revaluation of air abrasive technique *Journal of the American Dental Association* **50(4)** 408-414.
16. Mujdeci A & Gokay O (2004) The effect of airborne-particle abrasion on the shear bond strength of four restorative materials to enamel and dentin *The Journal of Prosthetic Dentistry* **92(3)** 477-482.
17. Dietrich T, Kraemer ML & Roulet JF (2002) Blood contamination and dentin bonding–effect of anticoagulant in laboratory studies *Dental Materials* **18(2)** 159-162.
18. Ferracane JL & Greener EH (1986) The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins *Journal of Biomedical Material Research* **20(1)** 121-131.
19. Walter R, Swift EJ Jr, Sheikh H & Ferracane JL (2009) Effects of temperature on composite resin shrinkage *Quintessence International* **40(10)** 843-847.
20. Dietrich T, Kraemer M, Lösche GM, Wernecke KD & Roulet JF (2000) Influence of dentin conditioning and contamination on the marginal integrity of sandwich Class II restorations *Operative Dentistry* **25(5)** 401-410.
21. Kimmes NS, Olson TL, Shaddy RS & Latta MA (2006) Effect of ViscoStat and ViscoStat Plus on composite shear bond strength in the presence and absence of blood *Journal of Adhesive Dentistry* **8(6)** 363-366.
22. Van Meerbeek B, Vargas M, Inoue S, Yoshida P, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **(Supplement 6)** 119-144

23. Perdigão J, Gomes G & Lopes MM (2006) Influence of conditioning time on enamel adhesion *Quintessence International* **37(1)** 35-41.

24. Rosales-Leal JI, Osorio R, Toledano M, Cabrerizo-Vílchez MA & Millstein PL (2003) Influence of eugenol contamination on the wetting of ground and etched dentin *Operative Dentistry* **28(6)** 695-699.

25. Saraç D, Bulucu B, Saraç YS & Kulunk S (2008) The effect of dentin-cleaning agents on resin cement bond strength to dentin *Journal of the American Dental Association* **139(6)** 751-758.

26. Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, Lambrechts P & Peumans M (2007) Bonding effectiveness of adhesive luting agents to enamel and dentin *Dental Materials* **23** 71–80.

27. Spencer P, Wang Y, Walker MP, Wieliczka DM & Swafford JR (2000) Interfacial chemistry of the dentin/adhesive bond *Journal of Dental Research* **79(7)** 1458-1463.

28. Mazzitelli C, Monticelli F, Toledano M, Ferrari M & Osorio R (2010) Dentin treatment effects on the bonding performance of self-adhesive resin cements *European Journal of Oral Sciences* **18(1)** 80-86.

29. Gray GB, Carey GP & Jagger DC (2006) An *in vitro* investigation of a comparison of bond strengths of composite to etched and air-abraded human enamel surfaces *Journal of Prosthodontics* **15(1)** 2-8.

30. Pashley DH, Pashley EL, Carvalho RM & Tay FR (2002) The effects of dentin permeability on restorative dentistry *Dental Clinics of North America* **46(2)** 211-245.