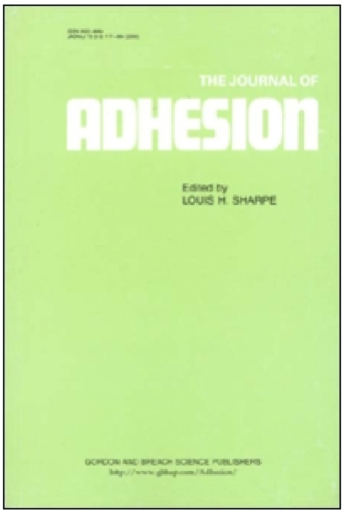

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Influence of Dentin Smear Layer Created by Chemo-Mechanical or Bur Excavation Methods on Adhesion of Self-Etching Primers and a Conventional Adhesive

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This study evaluated the influence of chemo-mechanical or bur excavation methods on bond strength of dentin bonding agents, micromorphology of the treated-dentin surfaces, and the bonded interfaces. Smear layer-free surfaces were used as control. The methods of cavity preparation (chemo-mechanical and rotary burs) were used under specific parameters and four commercial dentin bonding agents (three two-step self-etching primers and one "etch-and-rinse" adhesive system) were applied to treated surfaces, according to manufacturers' instructions. Composite blocks were built on bonded surfaces and restored teeth were vertically, serially sectioned to obtain bonded slices for interfacial micromorphologic analysis or to produce beam specimens for micro-tensile bond testing. A clear difference of the preparation of dentin surfaces and formation of hybrid layer and resin tags are noted. The use of burs or chemo-mechanical methods did not affect the bond strength for etch-and-rinse system and for a self-etching primer with a very low pH (0.5). However, dentin surface preparations decreased the bond strength for the milder versions (pH of around 2) of self-etching adhesive systems. The manner of dentin preparation prior to bonding procedures can influence the effectiveness of some self-etching primers, which dissolve the smear layer and dentin surface only partially.

Keywords: Adhesive system; Carisolv; Dental bur; Dentin; Smear layer

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INTRODUCTION

The tooth structure is formed by two hard tissues, enamel and dentin. Enamel covers the anatomical crown of the tooth and presents high mineral content. Whereas enamel is 92% inorganic hydroxyapatite by volume, dentin is only 45% inorganic. Dentinal hydroxyapatite is randomly arranged in an organic matrix that consists primarily of collagen. Dentin substrate is an intrinsically, wet organic tissue penetrated by fluid-filled channels or tubules, which results in a complex histological structure. Regional variations in dentinal structure and composition are reflected in the physiological characteristics at different locations within a tooth [1].

The new scientific information about the etiology and diagnosis of dental carious lesions has changed the treatments and the restorative procedures in dentistry. Contemporary adhesive composite restorations are used to replace carious dental tissues, fractured teeth, missing enamel in cervical non-carious lesions, and to change the shape and shade of teeth [2]. Before composite resin placement, an adhesive system is applied to enamel and dentin to serve as bonding agent and to seal the exposed surface. The basic mechanism of bonding to enamel and dentin is essentially an exchange process involving replacement of mineral removed from the hard tissue by adhesive resin monomers, which, upon setting, become micro-mechanically interlocked in the created micro-porosities [3]. The adhesion of light-cured composite restorative materials to enamel is a reliable technique; however, the bonding to dentin has proved to be more difficult and less predictable [2]. Two adhesion strategies are currently in use with adhesive systems. "Etch-and-rinse" adhesives involve a separate etch-and-rinse phase, while, self-etching do not require rinsing and serve simultaneously as conditioner/primer [3].

Caries-infected dentin must be removed and healthy or affected dentin is exposed for adhesive bonding [4]. Studies have shown that the manner of preparation of dentin surfaces prior to bonding procedures may affect the effectiveness of adhesive systems [5–7]. Caries removal and cavity preparations are usually performed with rotary carbide and diamond burs. An alternative caries removal method consists of very high pH gel application and the removal of the softened, infected, and demineralized dentin with hand excavators. This chemomechanical method can reduce patient discomfort caused by air-turbine noise and drilling vibration, beyond reducing the excessive loss of sound dental tissue, during cavity preparation [8,9].

The "smear layer" is formed during mechanical cavity preparation and represents an amorphous layer of organic (cut collagen fibrils) and inorganic (hydroxyapatite crystallites) debris deposited on the dentin. Differences have been related in smear layers prepared with burs or hand instruments, as well as in the morphology of treated dentin [10–12] and these differences would appear to affect the bond strength of dentin bonding agents [13–15]. As the self-etching primer bonding systems are applied directly to unetched smear layer-covered dentin, the performance of self-etching systems could be affected probably due to the difficulty of adhesive acidic monomers infiltrating through the smear layer into underlying dentin. Conversely, once removed by acid etching, the smear layer has no influence on dentin adhesion of etch-and-rinse bonding systems [6,16,17].

This research examined the effect of chemo-mechanical or bur excavation methods and their respective created smear layer on the adhesion of self-etching primer systems and an etch-and-rinse adhesive to dentin. The micro-tensile bond strength and the micromorphologic characteristics of prepared surfaces and resin-dentin interfaces were evaluated. The hypothesis tested was that the use of different cavity preparation techniques may affect the adhesion of bonding agents to dentin.

MATERIALS AND METHODS

Specimen Preparation and Experimental Groups

Extracted, caries-free human third molars (wisdom teeth) were used in this study, according to protocols approved by the institutional review board of the Piracicaba School of Dentistry, University of Campinas (150/2002). Teeth were stored for no more than 3 months in thymol-saturated water at 5°C to be decontaminated and to avoid cross-contamination to laboratory equipment and personnel. Ninety-six teeth were transversally sectioned with a diamond blade (Buehler Ltd., Lake Bluff, IL, USA) under water irrigation to remove occlusal enamel and expose flat, mid-coronal dentin surfaces (Figs. 1A and 1B). Teeth were randomly assigned to one of sixteen experimental groups ($n = 6$), according to the type of dentin treatment (carbide or diamond bur excavation; chemo-mechanical excavation; smear layer-free surfaces) and adhesive system applied (Table 1).

A two-step, alcohol-based, etch-and-rinse system was selected (Single Bond, 3M ESPE, St. Paul, MN, USA), which was applied to moist dentin after etching with 36% phosphoric acid for 15 seconds to remove the smear layer and to demineralize the superficial dentin. In addition, three two-step self-etching systems (Tyrian SPE/One-Step Plus, Bisco Inc., Schuamburg, IL, USA; Clearfil SE Bond,

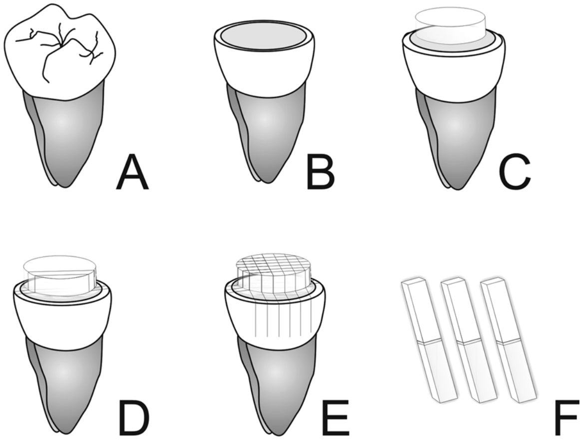


FIGURE 1 Schematic representation of specimen preparation: (A) intact tooth; (B) exposed mid-coronal dentin surface; (C) restored tooth; (D and E) sectioned tooth; and (F) beams or specimens (formed by composite bonded to dentin).

Kuraray Medical Inc., Kurashiki, Japan; and Unifil Bond, GC Corp., Tokyo, Japan) were chosen, which were applied directly to dry, unetched prepared surfaces, with different degrees of aggressiveness in their abilities to demineralize subsurface intact dentin. Clearfil SE Bond and Unifil Bond are considered mild self-etching adhesives (pH of approximately 2), while, Tyrian SPE/One-Step Plus has a very low pH (<1) and high concentration of acidic hydrophilic monomers.

Dentin Treatment Protocols

The depth of the dentin preparations was approximately 0.5 mm for all methods, which were in accordance with the following protocols:

- *Carbide bur* The entire dentin surface was uniformly prepared with a ten-blade cylindrical tungsten carbide bur (H297.314.012, Brasseler, Lemgo, Germany) for 15 seconds. The bur was mounted in a dental turbine (Extra Torque 605, Kavo Dental GmbH, Biberach, Germany) at high speed, under water cooling. New burs were used after every four preparations.
- *Diamond bur* The entire dentin surface was uniformly prepared with a cylindrical diamond bur (837.314.012, Brasseler, Lemgo, Germany) for 15 seconds at high speed under water irrigation.

TABLE 1 Compositions of Adhesive Systems Used in this Study

Adhesive	Composition	Procedures
Single Bond (etch & rinse)	Etchant: 35%phosphoric acid. Adhesive: Bis-GMA, HEMA, ethanol, water, UDMA, Bisphenol A glycerolate, polyalkenoic acid copolymer, dimethacrylate, camphorquinone (pH = 4.3).	a(15s); b; c; e(2 coats); c; l(10s)
Clearfil SE Bond (self-primer)	Primer: Water, ethanol, MDP, HEMA, dimethacrylate hydrophilic, camphorquinone, N, N-diethanol p-toluidine (pH = 2.0). Adhesive resin: MDP, Bis-GMA, HEMA, dimethacrylate hydrophobic, camphorquinone, N, N-diethanol p-toluidine, silanated colloidal sílica.	d; f(20s); c; g; l(10s)
Tyrian SPE/One-Step Plus (self-primer)	Primer: 2-Acrylamido-2-methyl propanesulfonic acid, Bis (2-(methacryloyloxy) ethyl) phosphate, ethanol (pH = 0.5). Adhesive: Biphenyl dimethacrylate, HEMA, acetone, glass frit.	d; h; i(20s); j; e; c; l(10s)
UniFil Bond (self-primer)	Primer: HEMA, 4-MET, ethanol, water (pH = 2.2). Adhesive resin: UDMA, HEMA, TEGDMA, silanated colloidal silica.	d; f; k(20s); c; g; l(10s)

Abbreviations—Bis-GMA: bisphenol-glycidyl-methacrylate; HEMA: 2-hydroxyethylmethacrylate; TEGDMA: triethylene glycol dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 4-MET: 4-methacryloxyethyl trimellitate anhydride; UDMA: urethane dimethacrylate.

Procedures—a: acid etch; b: rinse surface; c: gently air dry; d: air dry; e: apply one-bottle adhesive system; f: apply primer; g: apply adhesive; h: mix liquid A and B; i: apply mixture; j: remove excess with foam pellets; k: leave undisturbed; l: light cure.

The bur was mounted in a dental turbine (Extra Torque 605, Kavo Dental GmbH, Biberach, Germany) at high speed, under water cooling. New burs were used after every four preparations.

- *Chemo-mechanical excavation* Carisolv gel (MediTeam Dental, Göteborg, Sweden) is composed of amino-acids and sodium hypochlorite solution, which was applied to the entire dentin surfaces for 30 seconds. The volume of the gel applied to each tooth was 0.1 mL. Dentin was excavated with a non-cutting Carisolv hand instrument (#4) for 45 seconds (no bur was used). The dentin surface was rinsed with a tap water spray for 20 seconds to remove the gel.
- *Highly polished surface free of smear layer* The entire dentin surface was wet ground (1000- and 1200-grit aluminum oxide abrasive paper, 3M de Brazil, Sumaré, SP, Brazil) and polished with 6, 3, 0.5, and 0.25 μm diamond pastes (Arotec, Cotia, SP, Brazil). Each abrasive paper or diamond paste was used for 30 seconds on a polishing machine (APL-4, Arotec, Cotia, SP, Brazil). Teeth were ultra-sonicated (Ultrasonic Clean 1440D, Odontobrás, Ribeirão Preto, SP, Brazil) in distilled water for 12 minutes after each abrasive paper or diamond paste [18].

Micro-Tensile Bond Strength

Sixty-four teeth were used to perform the micro-tensile bond test. Sixteen teeth were randomly selected from each dentin treatment and designated to receive the application of one of four adhesive systems. Bonding systems were applied to prepared dentin and light-cured (630 mW/cm^2, XL 3000, 3M ESPE, St. Paul, MN, USA), in accordance with manufacturers' recommended directions (Table 1). The light-curing unit tip was always held 3 mm from the adhesive or composite resin surface. Resin composite build-ups were constructed incrementally on the polymerized bonding agent in five 1-mm thick layers using a micro-hybrid composite (Clearfil AP-X, shade A2, Kuraray Medical Inc., Kurashiki, Japan) (Fig. 1C). The next layer of composite was placed immediately after curing of the last one. The hand pressure during the build-up technique was the same for restorations of all teeth. Each layer was light-cured for 40 seconds using the same light-curing unit as before. Restored teeth were stored in distilled water at 37°C for 24 h and vertically, serially sectioned using a diamond blade (Buehler Ltd., Lake Bluff, IL, USA) in both mesio-distal and bucco-lingual directions across the bonded interface (Figs. 1D and 1E). The number of sections varied according to the size of the teeth (5 to 6 sections each side). After sectioning, several bonded beams (rectangular shape) with a cross-sectional measurement of 0.8 mm^2 (Fig. 1F) were obtained.

Six beams were randomly selected from each restored tooth. Each bonded beam was fixed to the grips of a micro-tensile testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA) and tested in tension at 0.5 mm/min until failure in a universal testing machine (4411, Instron Co., Canton, MA, USA). After fracture, the cross-sectional area of the debonded interface was measured to the nearest 0.01 mm with a digital caliper (Starret mod. 727-6/150, Starret, São Paulo, Brazil) and used to calculate test results in units of stress (MPa). Means of the six beams were calculated for each restored tooth fragment. Data were analyzed statistically by two-way ANOVA and the Tukey-Kramer post-hoc test. Statistical significance was established at $\alpha = 0.05$.

SEM Evaluations

The roots of thirty-two teeth were severed and the crown section was longitudinally cut (buccal-lingually) into two halves. Sixty-four tooth fragments were thus obtained. To evaluate the treated dentin surface morphology under scanning electron microscopy (SEM) (VP 435, Leo, Cambridge, England), sixteen fragments were randomly selected ($n = 4$). Dentin surfaces were treated with the four dentin treatment protocols (carbide bur, diamond bur, chemo-mechanical, and smear layer-free) as before. Fragments were dehydrated in ascending acetone concentrations (30%, 50%, 70%, 90% and 100%), critical-point dried (CPD 030, Balzers, Balzer, Liechtenstein), sputter-coated with gold (MED 010, Balzers, Balzer, Liechtenstein), and examined under SEM (VP 435, Leo, Cambridge, England). Representative areas (longitudinal surfaces) of the treated dentin were photographed at 5,000×.

For SEM evaluation of the interfacial micromorphology, forty-eight dental fragments were used. The dentin surfaces were treated according to the described protocols and the adhesive systems were applied in accordance with manufacturers' instructions ($n = 3$). Restored teeth were vertically, serially sectioned into some 2.0 mm thick slabs. The slabs were hand-polished with 600-, 1200-, and 2000-grit SiC paper (Norton Abrasivos, São Paulo, SP, Brazil) followed by diamond pastes (6, 3, 1 and 0.25 μm) (Buehler Ltd., Lake Bluff, IL, USA). Slabs were rinsed and polishing debris was ultrasonically removed during a 12-minute cleaning after each polishing step. After polishing, slabs were etched with 50% phosphoric acid solution for 15 seconds, washed, and treated with 0.1% with NaOCl for 10 minutes. Slabs were dehydrated in ascending acetone concentrations, critical-point dried, sputter-coated with gold, and examined under SEM. Representative areas (transverse surfaces) of the composite-dentin interfaces were photographed at 5,000×.

Fractured specimens from the microtensile test were gently removed from the grips, ultrasonically cleaned (Ultrasonic Clean 1440D, Odontobrás, Ribeirão Preto, SP, Brazil), allowed to air-dry overnight, sputter coated (MED 010, Balzers, Balzer, Liechtenstein), and observed under SEM (VP 435, Leo, Cambridge, England) to determine the failure site and fracture modes.

RESULTS

Two-way ANOVA indicated significant differences for the factor type of dentin treatment ($p < 0.01$), adhesive system ($p < 0.01$), and the interaction between factors ($p < 0.01$). The mean tensile bond strength and standard deviation values are shown in Table 2. Tukey's test showed that the bond strength of Single Bond and Tyrian SPE/One-Step Plus adhesive systems to dentin were not affected by dentin treatments. Clearfil SE Bond and Unifil Bond systems yielded higher bond strength values when applied to smear layer-free surfaces than when applied to cut or excavated dentin. The bur-cut dentin with carbide or diamond drills resulted in similar bond strength performance for all adhesive systems.

Figure 2A shows a flat carbide bur-treated dentin surface covered with the fine smear layer. The diamond bur produced coarse scratches across dentin and a thicker smear layer. Particles from the cutting procedure were found over dentin together with the smear layer

TABLE 2 Results of Tensile Bond Strengths for Experimental Groups

	Methods of tooth preparation			
Adhesive systems	Carbide bur	Diamond bur	Chemo-mechanical	Smear layer-free surface
Tyrian SPE/ One-Step Plus	25.3 (4.0) BCa	25.9 (2.1) BCa	20.7 (4.7) Ba	23.7 (1.8) Ba
Clearfil SE Bond	43.5 (7.9) Aab	34.6 (7.1) ABb	23.7 (2.5) ABc	47.3 (6.9) Aa
Unifil Bond	24.3 (6.0) Cb	23.9 (7.1) Cb	22.6 (4.1) Bb	37.1 (6.4) Aa
Single Bond	34.7 (6.7) ABa	37.6 (6.0) Aa	33.6 (6.7) Aa	39.5 (1.4) Aa

Groups having different letters (upper case = column; lower case = row) are significantly different.

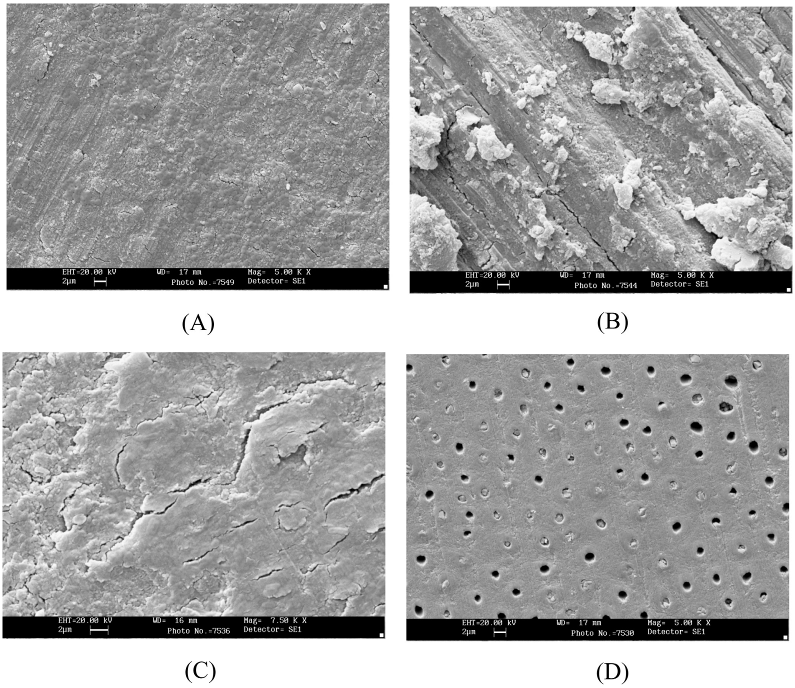


FIGURE 2 SEM of the prepared dentin surfaces: (A) carbide bur; (B) diamond bur; (C) chemo-mechanical method and (D) surface polished free of smear layer.

(Fig. 2B). Figure 2C shows a dense smear layer over the dentin surface prepared with the Carisolv chemo-mechanical method. The smeared debris did not form a continuous layer, but no opening of the dentinal tubules was visible. Conversely, the polished surface was free of smear layer. The smear plugs were present in most of the tubules (Fig. 2D).

Resin-dentin interdifusion zone and resin tags were noted in all bonded interfaces, which were created using self-etching and etch-and-rinse adhesive systems (Figs. 3A–F). The thickness of the hybrid layer and length of resin tags varied according to dentin treatment and the type of adhesive system. The predominant failure pattern in experimental groups was adhesive exhibiting adhesive layer and composite resin (Fig. 4A). SEM of bonded specimens treated with the Carisolv gel showed clear groove marks due to the excavation method (Fig. 4B).

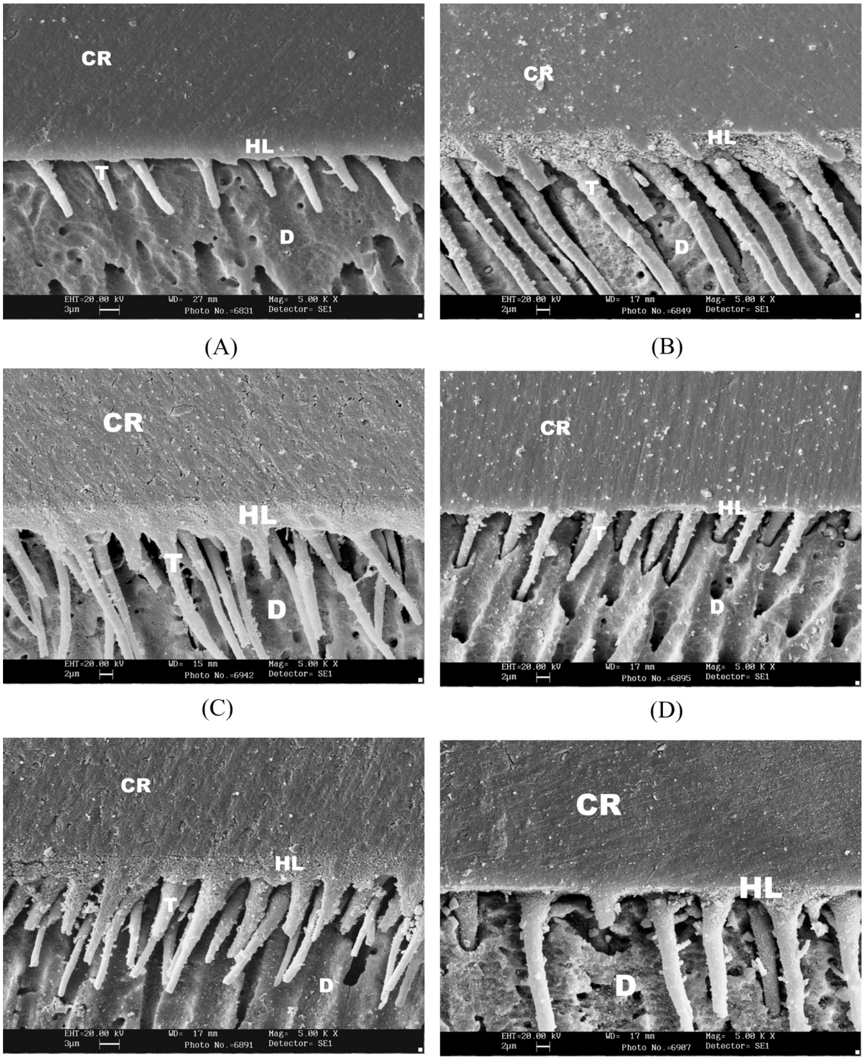
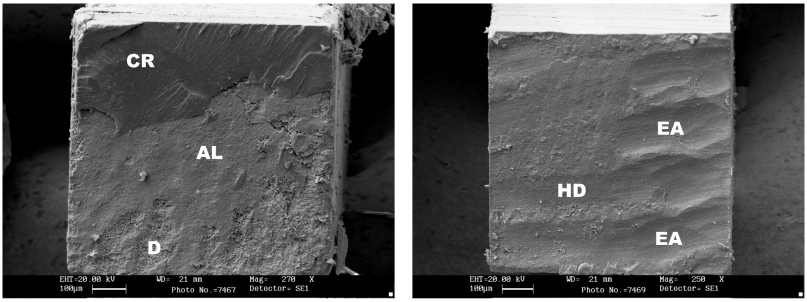


FIGURE 3 SEM of the bonded interfaces: (A) resin-dentin interface of Unifil Bond applied to carbide bur-cut dentin; (B) resin-dentin interface of Tyrian SPE/One-Step Plus bonded to diamond bur-cut dentin; (C) resin-dentin interface of Single Bond applied to chemo-mechanical excavated dentin; resin-dentin interfaces of (D) clearfil SE Bond, (E) Tyrian SPE/One-Step Plus, and (F) single Bond bonded to dentin surface polished and free of smear layer (CR-composite resin; HL-hybrid layer; D-dentin).



(A) (B)

FIGURE 4 SEM photomicrographs illustrating fractured surfaces: (A) fractured dentin-adhesive (Single Bond) interface on the dentin side, which was prepared with a diamond bur (CR-composite resin; AL-adhesive layer; D-dentin); (B) chemo-mechanical excavation method created grooved marks on treated surface (EA). Fractured dentin-adhesive (Tyrian) interface on the dentin side showed that the failure occurred cohesively in hybrid layer (HD-hybridized dentin).

DISCUSSION

The self-etching primer bonding mechanism comprises demineralization and diffusion of the acidic component through the smear layer into the underlying dentin. The hybrid layer is completely formed after bonding resin is applied to primed dentin [19]. As self-etching primer systems are applied to smear layer-covered dentin, concerns have been expressed regarding the manner of dentin surface preparation before bonding procedures [5–7,20]. Studies have shown that the quality and quantity of the smear layer formed vary according to the method used to cut or abrade the dentin during cavity preparation [10,12,21]. In the present study, differences were noted among the surface textures obtained by using different techniques (Fig. 2).

Dentin finishing with aluminum oxide abrasive papers (1200- and 2000-grit) followed by polishing with diamond pastes created a highly polished surface with the absence of, or a very thin, smear layer. Moreover, some smear plugs were also observed in most of the tubules (Fig. 2D) [18]. These surfaces were used as control, considering that acidic monomer from self-etching primers did not have difficulty in reaching the sound dentin. A thick smear layer may interfere with self-etching primers diffusing into the underlying intact dentin, buffering the acidic monomer, and increasing the pH, affecting the capacity to demineralize dentin. The size of smear layer debris and irregularly shaped particles can also impair smear layer hybridization and their incorporation into the hybrid layer [16].

The Unifil Bond self-etching primer represented the highest pH value among acidic primers tested, and contained 4-methacryloxyethyl trimellitate anhydride (4-MET) as acidic primer, and HEMA as an additional hydrophilic component. Unifil Bond applied to smear layer-free surfaces yielded the highest bond strength among the four prepared surfaces. Conversely, the other tooth preparation methods (burs and chemo-mechanical) reduced the bond strength. Irrespective of how the surfaces were prepared, dentin was always covered by a smear layer, as a result of the action of burs or chemo-mechanical excavation (Figs. 2A–C). Figure 3A shows the interfacial micromorphology of Unifil Bond applied to carbide bur-treated dentin at high speed. A well-defined hybrid layer and resin tags formation were not observed, indicating a weak interaction between the composite and bur-cut dentin, which could compromise adhesion and produce low bond strength. Except for smear layer-free surfaces, Unifil Bond had similar bond strengths to Tyrian SPE/One-Step Plus, although the adhesive systems presented significant differences in their compositions and pH of adhesive solutions.

Tyrian SPE/One-Step Plus produced the lowest mean bond strength among dentin-bonding agents applied to smear layer-free surfaces. This is the most acidic self-etching primer among the tested adhesive systems (pH 0.5); however, the absence of a smear layer and the primer acidity did not increase the bond strength to dentin. Studies have shown that more acidic self-etching systems presented more hydrophilic components as observed in the composition of Tyrian SPE/One-Step Plus self-etching system; however, more adhesive resin infiltration into dentin promoted by the acidity of adhesive systems and the amount of hydrophilic monomers is not related to high bond strength [19,22]. Figures 3D and 3E show the difference in hybrid layer thickness between Clearfil SE Bond and Tyrian SPE/One-Step Plus with the higher bond strengths for the thin hybrid layer, which was formed by Clearfil SE Bond.

The carbide and the diamond burs produced different micromorphologic characteristics (Figs. 2A and 2B); however, the adhesive systems applied to bur-prepared dentin surfaces resulted in similar bond strengths. Differences in the features of smear layers formed by different burs also have been observed by Eick *et al*. [23], Tao *et al*. [10], Santini & Mitchell [11], and Oliveira *et al*. [12]. Some authors have suggested that the presence and quality of the smear layer produced by burs affects the bond strength to dentin. Coarse instruments, such as coarse grit abrasive paper and coarse diamond burs can produce a thicker and looser smear layer [10,13,16,21], which is responsible for reducing the bond strength of self-etching primer systems to dentin [12,14–17]. Among the self-etching primers tested, Clearfil SE Bond showed better performance in terms of bond strength to both bur-cut dentin substrates.

Although the chemo-mechanical method is indicated to be used on caries-infected dentin, Carisolv gel was applied onto intact dentin since it can also reach and treat sound dentin walls during caries removal and cavity preparation. Excavations with Carisolv have also been shown to produce a smear-like debris covering the dentin surfaces [24–26] (Fig. 2C). Moreover, some reports have related protein denaturation by dissolution of non-cross linked collagen and other components from dentin [25,27]. However, studies showed that the use of a chemo-mechanical caries removal method does not adversely affect self-etching and etch-and-rinse adhesive systems bonding to dentin [28,29]. In the present study, lower bond strength values were obtained for Clearfil SE Bond and Unifil Bond self-etching systems when they were applied to chemo-mechanically excavated-dentin. The mildest form of these self-etching adhesives may not be enough to overcome the effects of Carisolv gel, which has a very high pH (pH 11). These systems present similar pH and a much higher pH than Tyrian SPE/One-Step Plus; however, no significant difference in bond strength to treated-dentin was observed among the self-etching primer systems.

This study used sound dentin surfaces in an attempt to evaluate only the effects of dentin preparation and the smear layers formed, since the modified dentin substrate, such as sclerotic, caries-infected or caries-affected dentin, are considerably unfavorable substrates for bonding [23]. Studies have shown low bond strengths for Single Bond and Clearfil SE Bond when applied on abnormal dentin [4,29], which could alter and impair the results of this study.

The bond strength of two adhesive systems was not affected by the manner of dentin surface preparation prior to bonding procedures. Single Bond is a two-step etch-and-rinse adhesive and represents a combination of hydrophilic primer, hydrophobic adhesive resins, and organic solvent in a single adhesive solution; however, with a previous, separate, phosphoric acid etching step [19,30]. The etching system (32–37% phosphoric acid) was reported to remove the smear layer and demineralize dentin by removing hydroxyapatite and exposing collagen fibrils in a few microns (3–7 μm) of the most superficial layer of dentin [31], thus eliminating the effects of smear layer debris and prepared dentin surfaces on bond strength [6,16,17]. Thus, the hybrid layer thickness formed by Single Bond did not vary following to the dentin preparation (Figs. 3C and 3F). As the Tyrian SPE/One-Step Plus self-etching system is considered a strong and aggressive self-etching adhesive [22], the SPE primer acidity was enough to overcome the effects of dentin preparation and the different smear layers formed, yielding similar bond strength results and hybrid layer formations with similar thickness (Figs. 3B and 3E).

CONCLUSIONS

In accordance with the methodology used, and based on the results obtained and statistical analysis, this study showed that the effect of chemo-mechanical or bur excavation methods on the tensile bond strength to dentin was material-dependent, and indicated a decrease for two self-etching systems (Unifil Bond and Clearfil SE Bond) and no influence for Tyrian SPE/One-Step Plus and Single Bond etch-and-rinse adhesive systems. However, Tyrian SPE/One-Step Plus showed very different results when compared with Single Bond in terms of bond strength.

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REFERENCES

[1] Cate, T., *Oral Histology* (Elsevier, Amsterdam, 2007), 8th ed.
[2] Perdigão, J., *Dent. Clin. N. Am.* **46**, 277–301 (2002).
[3] De Munck, J., Van Landuyt, K., Peumans, M., Poitevin, A., Lambrechts, P., Braem, M., and Van Meerbeek, B., *J. Dent. Res.* **84**, 118–132 (2005).
[4] Arrais, C. A. G., Giannini, M., Nakajima, M., and Tagami, J., *Eur. J. Oral Sci.* **112**, 458–464 (2004).
[5] De Munck, J., Van Meerbeek, B., Yudhira, R., Lambrechts, P., and Vanherle, G., *Eur. J. Oral Sci.* **110**, 322–329 (2002).
[6] Van Meerbeek, B., De Munck, J., Mattar, D., Van Landuyt, K., and Lambrechts, P., *Oper. Dent.* **28**, 647–660 (2003).
[7] Trajtenberg, C. P., Pereira, P. N. R., and Powers, J. M., *Am. J. Dent.* **17**, 331–336 (2004).
[8] Kakaboura, A., Masouras, C., Staikou, O., and Vougiouklakis, G., *Quintessence Inter.* **34**, 269–271 (2003).
[9] Rafique, S., Fiske, J., and Banerjee, A., *Caries Res.* **37**, 360–364 (2003).
[10] Tao, L., Pashley, D. H., and Boyd, L., *Dent. Mater.* **4**, 208–216 (1988).
[11] Santini, A. and Mitchell, S., *Quintessence Inter.* **29**, 737–747 (1998).
[12] Oliveira, S. S. A., Pugach, M. K., Hilton, J. F., Watanabe, L. G., Marshall, S. J., and Marshall, Jr., G. W., *Dent. Mater.* **19**, 758–767 (2003).

[13] Hosoya, Y., Shinkawa, H., Suefiji, C., and Nozaka, K., *Am. J. Dent.* **17**, 359–364 (2004).
[14] Koase, K., Inoue, S., Noda, M., Tanaka, T., Kawamoto, C., Takahashi, A., Nakaoki, Y., and Sano, H., *J. Adhes. Dent.* **6**, 97–104 (2004).
[15] Ogata, M., Harada, N., Yamaguchi, S., Nakajima, M., Pereira, P. N. R., and Tagami, J., *Oper. Dent.* **26**, 375–382 (2001).
[16] Tay, F. R., Carvalho, R. M., Sano, H., and Pashley, D. H., *J. Adhes. Dent.* **2**, 99–116 (2000).
[17] Ogata, M., Harada, N., Yamaguchi, S., Nakajima, M., and Tagami, J., *Oper. Dent.* **27**, 447–454 (2002).
[18] Arrais, C. A. G., Micheloni, C. D., Giannini, M., and Chan, D. C. N., *J. Dent.* **31**, 59–66 (2003).
[19] De Munck, J., Van Landuyt, K., Peumans, M., Poitevin, A., Lambrechts, P., Braem, M., and Van Meerbeek, B., *J. Dent. Res.* **84**, 118–132 (2005).
[20] Chaves, P., Giannini, M., Ambrosano, G. M. B., *J. Adhes. Dent.* **3**, 191–196 (2002).
[21] Koibuchi, H., Yasuda, N., and Nakabayashi, N., *Dent. Mater.* **17**, 122–126 (2001).
[22] Tay, F. R. and Pashley, D. H., *Dent. Mater.* **17**, 296–308 (2001).
[23] Eick, J. D., Wilko, R. A., and Anderson, C. H., *J. Dent. Res.* **49**, 1359–1368 (1970).
[24] Banerjee, A., Kidd, E. A. M., and Watson, T. F., *J. Dent.* **28**, 179–186 (2000).
[25] Cederlund, A., Lindskog, S., and Blomlöf, J., *Acta Odontol. Scand.* **57**, 185–189 (2000).
[26] Yazici, A. R., Özgünaltay, G., and Dayangaç, B., *Oper. Dent.* **27**, 360–366 (2002).
[27] Wennemberg, A., Sawase, T., and Kultje, C., *Eur. J. Oral Sci.* **107**, 297–306 (1999).
[28] Kubo, S., Li, H., Burrow, M. F., and Tyas, M. J., *Oper. Dent.* **27**, 387–395 (2002).
[29] Çehreli, Z. C., Yazici, A. R., Akca, T., and Özgünaltay, G., *J. Dent.* **31**, 429–435 (2003).
[30] Reis, A. F., Oliveira, M. T., Giannini, M., de Goes, M. F., and Rueggeberg, F. A., *Oper. Dent.* **28**, 689–694 (2003).
[31] Perdigao, J., Lambrechts, P., Van Meerbeek, B., Tome, A. R., Vanherle, G., and Lopes, A. B., *Dent. Mater.* **12**, 262–271 (1996).