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Effect of Bur Roughness on Bond to Sclerotic Dentin With Self-etch Adhesive Systems

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Clinical Relevance

The use of diamond burs with different roughness did not increase the bond strength of self-etch systems and etching pattern in sclerotic dentin. Clinicians should avoid using this procedure when applying self-etch adhesive.

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ABSTRACT

Purpose: **To evaluate the effect of bur roughness on bond strength values and conditioner pattern of two-step self-etch adhesives applied on sclerotic dentin.**

Methods: **The roots of 48 bovine incisors were removed and the crowns were divided into four groups: the control group (CO) teeth were left untreated or the teeth were slightly roughened with coarse-, medium-, or fine-grit diamond burs. Next, the teeth were subdivided and Clearfil SE Bond (CSE) and Adper SE Bond (ASE) were applied according to the manufacturers' instructions. Composite resin (Opallis) buildups were incrementally constructed on the bonded surfaces. After storage for 24 hours in distilled water at 37°C, the teeth were sectioned into sticks (area of 0.8 mm^2). The sticks were stressed until failure by tensile forces (0.5 mm/min). Additionally, eight bovine teeth were treated as previously described, and after adhesive application, the surface was rinsed off and examined by scanning electron**

microscopy to measure the relative number of open tubules (OT). Data (MPa) were analyzed by two-way analysis of variance and Tukey test (*p*=0.05).

Results: CO showed the highest bond strength values ($p<0.05$). As regards OT, the lowest mean was observed for CO ($p<0.05$) and the highest was found after application of CSE or ASE ($p<0.05$).

INTRODUCTION

The basic bonding mechanism to dental substrates is essentially an exchange process involving replacement of the minerals removed from hard dental tissue by resin monomers, which upon setting become micromechanically interlocked in the porosities created. Presently two bonding mechanisms with resin monomers are used with modern adhesive systems: the etch-and-rinse and the self-etch approaches.[1,2]

Regardless of the bonding strategy, bonding to pathologically altered substrates such as sclerotic dentin generally leads to compromised bonding.[3,4] Sclerotic dentin has partially or totally obliterated dentinal tubules as a result of the continuous deposition of peritubular dentin,[5,6] and it is generally present in noncarious cervical lesions.[3,4,6,7] The micromorphological features of this altered dentin substrate are potential obstacles to resin infiltration, which include the hypermineralized surface layer, an additional partially mineralized surface bacterial layer, and intratubular mineral casts that are comparatively more acid-resistant.[3,8,9]

In vitro studies[4,7,10,11] have demonstrated that for etch-and-rinse adhesives, bond strength values in sclerotic dentin are 25%-40% lower than that achieved in sound dentin as a result of the presence of an acid-resistant hypermineralized surface layer. The causes of this mineral deposition are multifactorial, including occlusal stresses, chronic stimuli of low intensity and high frequency, and bacterial colonization.[6]

Hypermineralization inside the dentinal tubules also hinders the formation of resin tags and thus promotes the formation of a thinner and less homogeneous hybrid layer.[8,9,12,13] It has been suggested[3,4,8] that removal of the upper hypermineralized surface layer by bur grinding or stronger acids offers a possible strategy with which to improve micromechanical retention in sclerotic dentin. Several researchers have investigated the effect of these approaches (ie, increasing phosphoric acid conditioning time[10,14-16] and roughening the dentin surface[10,17,18] when using etch-and-rinse systems) and have obtained controversial results.

With regard to self-etch adhesives, phosphoric acid pretreatment[4,13] or roughening the sclerotic surface with diamond burs has also been reported[13] to increase the hybrid layer thickness. Nevertheless, the formation of a thicker hybrid layer cannot be interpreted as a material advantage because the thickness of this layer is not related to high bond strength values.[2] To the best of our knowledge, the effect of roughening dentin with a bur on the bond strength of self-etch adhesives to sclerotic dentin has not yet been investigated. Therefore, the aim of this study was to evaluate the effect of different diamond bur grits on the bond strength of self-etch systems to sclerotic dentin and the etching pattern produced by self-etch systems on the diamond bur–treated dentin surfaces.

MATERIALS AND METHODS

Tooth Selection and Preparation

Fifty-six extracted bovine incisors with exposed sclerotic dentin, clinically characterized by a vitreous appearance,[12,19] were used in this study. They were extracted from the mandibles of three-year-old animals who had been slaughtered on a commercial scale for meat consumption. After harvesting, they were stored in distilled water at 4°C for no longer than one week before being used in this experiment. The roots of all teeth were sectioned and the coronal pulp was removed.

Experimental Design and Restorative Procedure

Forty teeth were then embedded in chemically activated resin, with the exposed dentin surfaces parallel to the horizontal plane. Next, the dentin surfaces of all teeth were cleaned with a detergent (Tergentol, Inodon, Porto Alegre, RS, Brazil). The teeth were randomly assigned into eight experimental groups (n=5) according to the combination of the main factors "adhesive system" (two levels) and "surface treatment" (four levels).

In the control group, dentin surfaces were left untreated. For experimental groups the teeth were slightly roughened with a coarse-grit (#1035, KG Sorensen, São Paulo, SP, Brazil; mean particle size 151 μm), medium-grit (#1035, KG Sorensen; mean particle size 91 μm), or fine-grit (#1035F, KG Sorensen; mean particle size 46 μm) diamond bur mounted in a high-speed hand-piece under water

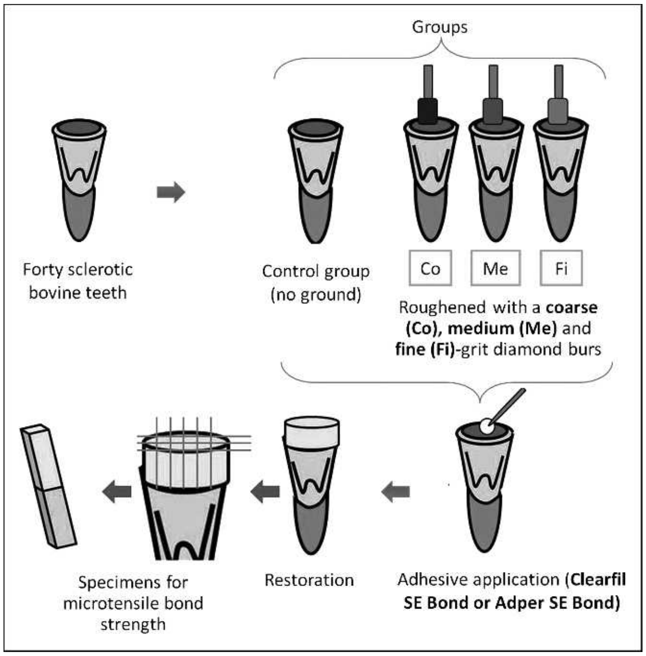

Figure 1. *Flowchart of the microtensile bond strength test.*

cooling for a period of 10 seconds (Figure 1). The teeth were prepared with 10 passes with the bur across the dentin surface under copious air-water spray. Before performing the sample surface preparation, the single operator was training on the surface of an analytical balance to determine the equivalent manual pressure that would be placed on the surface during surface preparation (Mettler, type H6, Columbus, OH, USA). The pressure applied was equivalent to approximately 300 ± 55 g.

After this, the adhesive systems Clearfil SE Bond (CSE; Kuraray, Okayama, Japan) and Adper SE Plus (ASE; 3M ESPE, St Paul, MN, USA) were applied according to the manufacturers' instructions (Table 1). After the bonding procedure, all teeth received a microhybrid composite restoration (Opallis, FGM Produtos Odontológicos, Joinville, SC, Brazil) in two increments of 2 mm that were each light polymerized for 40 seconds using a halogen light-curing unit set at 400 mW/cm^2 (VIP, Bisco, Schaumburg, IL, USA), without use of a matrix, as conventionally performed for the microtensile bond strength test.[11,13] The specimens were stored in water at 37°C for 24 hours.

Microtensile Bond Strength Test

The specimens were cut perpendicularly with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain five to 10 resin-dentin beams (0.8 mm × 0.8 mm cross-sectional dimensions on average) from each tooth for microtensile testing (μTBS) (Figure 1). The final width and thickness of the bonded area were measured to the nearest 0.01 mm with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). Specimens were attached to a Geraldelli jig[20] with cyanoacrylate adhesive and stressed under tension (Kratos Dinamometros, Co-

Table 1: *Adhesive Systems, Batch Number, Composition, and Application Mode*

Adhesive Systems	Composition	Mode of Application
Clearfil SE Bond (Kuraray, Okayama, Japan); Lot No. 00795A	Primer: MDP, HEMA, dimethacrylate monomer, water, catalyst Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyst	1. Apply primer and leave for 20 s. 2. Dry thoroughly with mild air flow. 3. Apply bond. 4. Gentle air flow. 5. Light polymerize for 10 s at 400 mW/cm^2.
Adper SE Plus (3M ESPE, St Paul, MN, USA); Lot No. 8BG/8BF	Bottle A: water, HEMA, surfactant, pink colorant Bottle B: UDMA, TEGDMA, TMPTMA, HEMA phosphates, MHP, bonded zirconia nanofiller, initiator system based on camphorquinone	1. Apply one coat of bottle A. This goes on pink, acting as a guide to achieving full coverage when applying the adhesive. 2. Apply bottle B and the pink color disappears, indicating where the adhesive is placed. 3. Agitate for 20 s. 4. Air-dry for 10 s. 5. Light polymerize for 10 s at 400 mW/cm^2.

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogenphosphate; MHP, methacrylated phosphates; TEGDMA, triethylene glycol dimethacrylate; TMPTMA, hydrophobic trimethacrylate; UDMA, urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,30,5-trimethylhexaan.

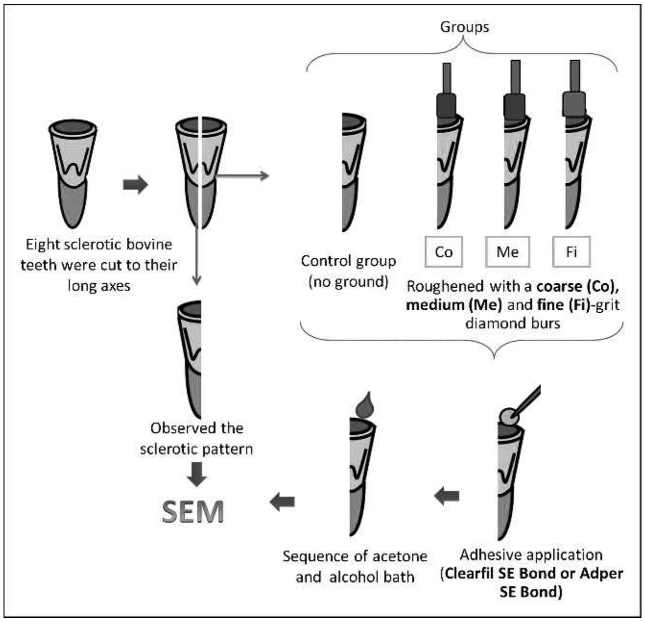

Figure 2. *Flowchart of the etching pattern examined by scanning electron microscopy (SEM).*

tia, São Paulo, Brazil) at 0.5 mm/min until failure. Bond strengths were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the specimens was classified as adhesive/mixed if failure occurred at the resin-dentin bond interface with or without cohesive failure of the neighboring substrates and as cohesive if the failure occurred at the substrate (resin or dentin). The classification was done under a stereomicroscope at 100× magnification (Olympus SZ40, Tokyo, Japan).

Statistical Analysis

All bond strength values obtained from the same tooth were averaged, and the tooth was considered the statistical unit of the present investigation.[21,22] The data were then submitted to two-way analysis of variance (ANOVA) (adhesive vs surface treatment) and post hoc Tukey tests. The level of significance was preset at α=0.05.

Etching Pattern Examined by Scanning Electron Microscopy (SEM)

A total of 16 bovine teeth were used for this part of the experiment. The teeth were cut perpendicular to their long axes using a slow-speed diamond saw (Isomet) in order to obtain two dentin halves. They were then randomly divided into two lots of halves: one half was used to evaluate the degree of dentin obliteration (control group), while the other half was treated according to one of the four following groups (n=2): 1) adhesive only application (without bur) and slightly roughened with a 2) coarse-grit, 3) medium-grit, or 3) fine-grit diamond bur, followed in each case by adhesive application. The adhesive application was performed as described in the "Experimental Design and Restorative Procedure" section. However, the adhesives were not light-cured after application (Table 1). Each surface was rinsed off with a sequence of acetone bath (five minutes), deionized water (five minutes), 96% alcohol bath (five minutes), and deionized water (five minutes) in order to remove all resin monomers from the surface (Figure 2).[23,24]

Specimens were then fixed in 2.5% glutaraldehyde in 0.1 M phosphate-buffered solution for 24 hours at room temperature before being dehydrated in ascending grades of ethanol and submitted to chemical drying in hexamethyldisilazane (SSX-550, Shimadzu, Tokyo, Japan). Finally, the samples were sputter-coated with gold (Sputtering SCD050, Bal-Tec, Balzers, Liechtenstein) and examined by SEM (SSX-550, Shimadzu) at 12 kV operated in secondary electron mode.

For the SEM analysis, three pictures were taken of each half. All of the electron micrographs were taken at the same working distance using the same magnification.[19] The measurement was performed as follows: the total area of each image was recorded using the UTHSCSA ImageTool 3.0 software (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA) by a blinded researcher. Then, in the same image, the open tubule area was delineated by a software tool, summed, and the relative ratio between the open tubule area vs total area was calculated to give the relative percentage of open tubule area of each specimen. The three readings were averaged for statistical purposes. The relative percentage of open tubule area was evaluated by two-way ANOVA (adhesive vs surface treatment) and post hoc Tukey tests. The level of significance was preset at α=0.05.

RESULTS

Microtensile Bond Strength Test

Five to 10 resin-dentin specimens were obtained from each tooth. The failure modes of all experimental groups are shown in Table 2. The majority of the specimens (86.6%) presented adhesive/mixed failures. Dentin and resin failures were observed in 1.5% and 5.9% of the specimens, respectively. A small number of premature failures (6.0%) were observed.

Table 2: *Number of Specimens and Percentage (%) of Failure Modes for All Experimental Groups*

Adhesive	Failure Type	Control	Diamond Bur Granulation		
			Coarse	Medium	Fine
Clearfil SE Bond	A/M	17 (10.8)	37 (23.4)	40 (25.3)	39 (24.6)
	D	3 (2.0)	0 (0)	0 (0)	0 (0)
	R	7 (4.4)	3 (2.0)	0 (0)	0 (0)
	PF	1 (0.6)	5 (3.0)	4 (2.6)	2 (1.3)
Adper SE Bond	A/M	25 (22.5)	24 (21.6)	23 (20.7)	27 (24.4)
	D	0 (0)	0 (0)	1 (0.9)	0 (0)
	R	1 (0.9)	2 (1.8)	2 (1.8)	1 (0.9)
	PF	0 (0)	3 (2.7)	1 (0.9)	1 (0.9)

Abbreviations: A/M, adhesive or mixed failure; D, dentin cohesive failure; PF, premature failures; R, resin cohesive failure.

With regard to resin-dentin bond strength, only the main factor surface treatment was statistically significant (p=0.022). For both adhesives, the highest mean bond strength values were observed for the control group. However, this group was similar to the groups treated with coarse- and fine-grit diamond burs (p>0.05). The lowest resin-dentin bond strength was found when the medium-grit diamond bur was used (p<0.05) (Table 3).

Etching Pattern

Table 4 shows the relative percentage of open tubule area for all groups. Statistical analysis revealed that the cross-product interaction adhesive vs surface treatment was not statistically significant (p=0.43); however, both main factors were statistically significant (p=0.006 and p=0.0001, respectively, for adhesive and surface treatment).

For both adhesives, the lowest mean percentage of open tubules was observed for the control group before treatment in comparison with all groups (p<0.05). However, the use of primer significantly increased the percentage of open tubules for all groups (p<0.05). The use of a diamond bur showed an intermediate value of open tubules, regardless of the diamond bur coarseness (p>0.05). As regards adhesives, Clearfil SE Bond showed higher mean percentage values of open tubules in comparison to Adper SE Bond (p<0.05).

Table 3: *Mean Values and Standard Deviation of Microtensile Bond Strength (MPa) and Statistical Analysis[a] of All Experimental Groups*

Adhesive	Control A	Diamond Bur Granulation		
		Coarse A	Medium B	Fine A
Clearfil SE Bond	32.5 ± 10.7	26.3 ± 3.4	25.6 ± 3.6	24.1 ± 5.3
Adper SE Bond	30.1 ± 7.5	23.1 ± 4.7	16.0 ± 5.2	27.7 ± 5.6

[a] *Groups identified with the same capital letter are not statistically different (Tukey test, p>0.05).*

Table 4: *Mean Values and Standard Deviations of the Relative Percentage of Open Tubule Area (%) and Statistical Analysis[a] for All Experimental Groups*

Adhesive	Before Treatment	Control	Diamond Bur Granulation		
			Coarse	Medium	Fine
Clearfil SE Bond (a)	5.5 ± 2.6 A	22.8 ± 4.6 C	13.7 ± 4.3 B	10.4 ± 1.8 B	13.1 ± 5.1 B
Adper SE Bond (b)	4.5 ± 2.4 A	21.1 ± 4.6 C	9.3 ± 4.0 B	9.4 ± 2.2 B	8.7 ± 3.2 B

[a] *Groups with the same capital or lowercase parenthetical letters are not statistically different (Tukey test, $p>0.05$).*

Figure 3 shows SEM of the dentin surfaces of all the experimental groups. Very superficial open dentinal tubules were observed in the adhesive-only application (without bur) control groups (Figure 3C,D) in comparison to the other experimental conditions, in which diamond burs were used (Figure 3E-J). That is, the primers of the adhesive system used were unable to condition the dentin surface after it was roughened with the bur (Figure 3E-J).

DISCUSSION

The difficulty of obtaining human teeth with sclerotic features represents the reason why we opted to use bovine teeth in the present study. Previous studies revealed that both human and bovine dentin substrates have a similar morphology,[19,25-27] and superficial similarity could point at the possibility of using bovine sclerotic dentin as a replacement for human sclerotic dentin in bond strength tests, which allows researchers to use teeth from bovine animals to evaluate the behavior of adhesives systems.[28,29]

The results of the present investigation showed that the use of diamond burs of different grit sizes to roughen the sclerotic dentin did not improve the bond strength between the sclerotic dentin and self-etch adhesives. The findings of previous literature indicated that diamond bur roughening would produce a more irregular surface and a greater area of intertubular dentin available for adhesion.[3,10] Furthermore, Eliguzeloglu and others[13] showed that tag formation produced by self-etch adhesives was much more pronounced when the sclerotic dentin

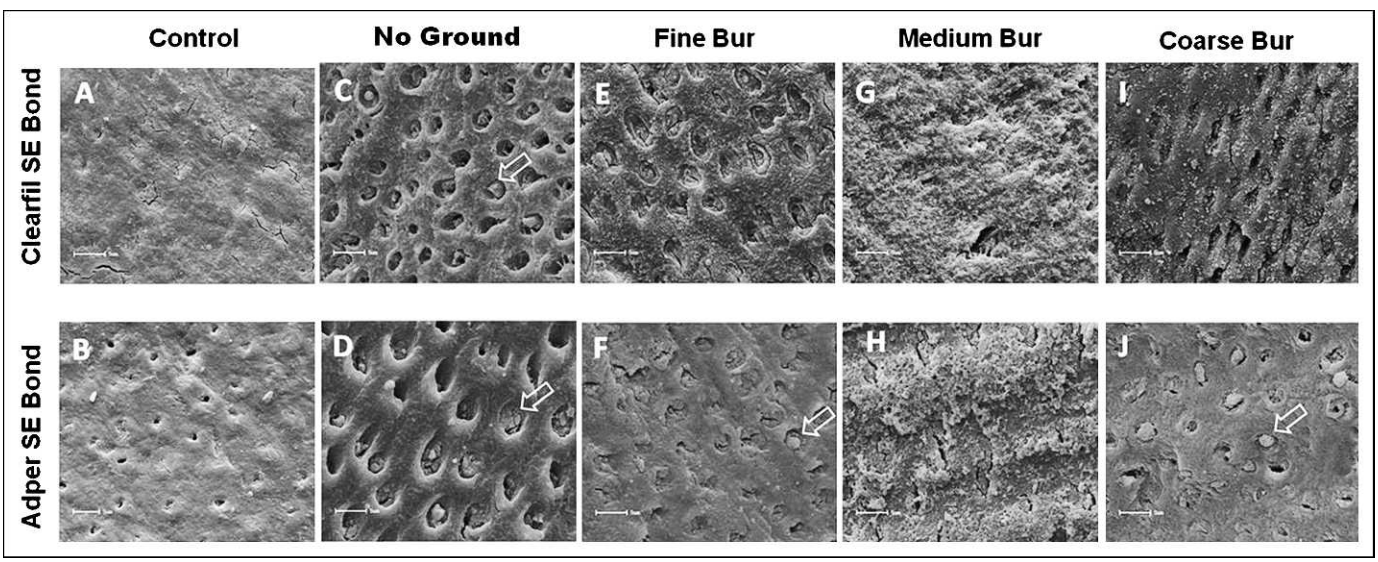

Figure 3. *Scanning electron micrographs of the sclerotic dentin surfaces in a control group before treatment (A and B) and after adhesive application with and without bur (Clearfil SE Bond [C, E, G, and I] and Adper SE Bond [D, F, H, and I]). Observe that in sclerotic dentin, complete obliteration of dentin tubules was shown (A and B). However, a large number of dentin tubules were exposed after application of the adhesive systems (C, Clearfil SE Bond; and D, Adper SE Bond). Note that in these groups, sclerotic dentin with "sclerotic" casts extending from open dentin tubules was shown (arrows). The surface of the sclerotic dentin after being roughened with different diamond burs (E, F: fine; G, H: medium; and I, J: coarse) was shown to be partially covered.*

had been previously roughened with a diamond bur prior to adhesive application.

Although the results of the present investigation are not in agreement with those of previous studies, we cannot rule out the fact that the authors of those other studies have only evaluated the bonding performance micromorphologically. It has been demonstrated that the hybrid layer thickness[3,10] or the amount of tag formation[13] are not correlated with improved resin-dentin bond strength.[2]

In fact, mechanical preparation may lower the thickness of the hypermineralized surface layer and remove some of the residual sclerotic casts that superficially obliterate the dentin tubules, but a tenacious smear layer is invariably formed along the lesion surface, consisting of hypermineralized remnants that are likewise resistant to acid dissolution,[4] and this can be observed in Figure 1.

The features of this smear layer, such as thickness and coarseness, depend on the bur used for cavity preparation.[30,31] This has led to investigators recommending the use of extra-fine diamond burs[30] or tungsten carbide burs[31] for cavity preparation of sound dentin, as they produce thinner smear layers. While this may yield improved bond strength of self-etch systems to sound dentin, this does not seem to be applicable to sclerotic dentin, according to the results of the present study. It was observed that the bond strength after bur roughness did not improve, and in some cases, a decrease in bond strength was found. There are no clear explanations for the lesser results found for the medium diamond bur, and future studies need to be conducted to test this hypothesis.

The formation of a smear layer that consists of acid-resistant hypermineralized dentin chips and whitlockite crystals derived from the sclerotic casts creates additional diffusion barriers to self-etch adhesives.[3] The retention of diffusion barriers, either in the form of an intact hypermineralized layer or an acid-resistant smear layer, resulted in some decline in the success of bonding to sclerotic dentin.[32]

Although this issue is somewhat controversial in the literature, there are studies[31,33-36] indicating that thick smear layers may hinder the infiltration of resin monomers into the underlying substrate and reduce the resin-dentin bond strength values produced by self-etch systems. Thus, the findings of this study indicate that when self-etch adhesives are to be applied to sclerotic dentin, clinicians should avoid cavity preparation, particularly with the use of a medium-grit diamond bur. This may be feasible when restoring noncarious cervical lesions. If no cavity preparation is performed, the sclerotic dentin surface is devoid of smear layer and the hypermineralized layer is left intact.[4] In this case, although it does not increase the bond strength values, there are a larger number of tubules available for adhesion.

However, this may not be the case with sclerotic dentin beneath caries lesions, where cavity preparation, in most of the cases, is mandatory. Other clinical approaches should be investigated in order to improve the bond strength of self-etch systems and instrumented sclerotic dentin. For instance, when observing the results of the etch-and-rinse adhesive, roughening/removal of the sclerotic superficial dentin was of help in obtaining more uniform etching and resin infiltration into intertubular sclerotic dentin,[17] and this procedure is responsible for improving the bond strength to sclerotic dentin.[10] Thus, the adjunctive use of an acid solution on ground sclerotic dentin before applying a self-etch adhesive will probably improve the bond strength, as has been suggested by several authors.[1,3,4] Future studies need to be conducted to test this hypothesis.

With regard to the adhesive system tested in the present study, the smear layer thickness produced in sound dentin had no influence on the microtensile bond strength values for the mild two-step self-etch adhesive, such as Clearfil SE Bond.[33-36] Adper SE Plus (also known as Adper Scotchbond SE in Europe) is a new material, and there is little information available on it; however, recent studies[37,38] have indicated that the bond strength values to sound dentin are similar between them, a finding that is in agreement with the present results in sclerotic dentin.

CONCLUSIONS

The bonding performance of self-etch systems in sclerotic dentin cannot be improved by roughening the dentin with diamond burs. Depending on the grit size of the diamond bur used, a decline in the bond strength of self-etch systems may occur when they are applied to sclerotic dentin.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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