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Bond Strength and Quality of the Hybrid Layer of One-step Self-etch Adhesives Applied with Agitation on Dentin

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Clinical Relevance

Vigorous application on the dentin surface is an excellent tool capable of improving the immediate performance of simplified self-etch adhesive systems.

SUMMARY

Objectives: This study evaluated the effect of the application mode on the microtensile resin-dentin bond strength (µTBS) and silver nitrate uptake (SNU) of three one-step self-etch adhesives systems (Clearfil S^3 Bond [S3], Xeno III [XE] and Adper Prompt L-Pop [AD]).

Materials and Methods: The occlusal enamel of 30 caries-free extracted molars was removed with a slow-speed diamond saw under copious water-cooling in order to expose a flat dentin surface. The adhesives were applied passively or with agitation. After light-curing (600 mW/cm^2 for 10 seconds), composite buildups were constructed incrementally and the specimens were stored in water (37°C/24 hours). The specimens were longitudinally sectioned in the "x" and "y" direction to obtain bonded sticks (0.8 mm^2) to be tested immediately in tension at 0.5 mm/minute. For SNU, two bonded sticks from each tooth were coated with nail varnish, placed in silver nitrate and polished down with SiC paper. The µTBS and SNU data were submitted to two-way ANOVA and Tukey's test (α=0.05).

Results: Regarding the µTBS, only the main factor mode of application was statistically significant (*p*=0.01). All adhesives showed higher µTBS when applied with agitation. Regarding SNU, only the main factor adhesive was statistically significant (*p*=0.001). A higher amount of

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silver nitrate uptake, located in both the hybrid and adhesive layer, occurred for AD, regardless of the mode of application.

Conclusions: Application with agitation on the dentin surface is a clinical tool capable of improving the resin-dentin bond strength of one-step self-etch adhesives; however, this clinical approach does not improve the hybrid layer resistance to silver nitrate uptake.

INTRODUCTION

Bonding to dental hard tissues can be accomplished using one of two main adhesion strategies: the etch-and-rinse or the self-etch approach. These approaches have been used to bond resin-based materials to enamel and dentin with a number of steps. Efforts have been directed toward reducing the number of steps and reducing the technique sensitivity associated with the bonding procedure.[1]

One-step self-etch adhesive systems (also known as all-in-one adhesives or non-rinsing adhesives) require shorter clinical application time, are less technique sensitive and are user-friendly. The elimination of separate etching and rinsing steps simplified the bonding technique and has been responsible for the increased popularity of these systems in daily practice.[2] These systems do not require removal of the smear layer and smear plugs, as they are incorporated into the hybrid layer complex.[3]

Unfortunately, different research centers have shown that some one-step self-etch adhesives exhibit relatively low bond strength values to both enamel and dentin when compared to two-step self-etch or etch-and-rinse systems.[1,3] According to Pashley and others[4] and Yiu and others,[5] mixtures that contain high concentrations of solvents, such as one-step self-etch adhesives, may impair polymerization of the monomers within the demineralized tooth substrates.[6] The higher the remaining solvent content, the lower the mechanical properties of the polymer formed.[7-9] The incomplete removal of solvents may prevent attainment of an adequate degree of conversion, which, in turn, is responsible for the low mechanical properties of these adhesives[10] and low resin-dentin bond strengths.[9,11,15] Thus, attention should be given to the removal of solvents from one-step self-etch adhesives before the light-curing step.

Theoretically, discrepancies between the depth of demineralization and the depth of resin infiltration can be avoided by use of self-etch adhesives.[12] The rationale behind the use of the self-etch approach assumes that acidic resin monomers will penetrate beyond the smear layer into mineralized dentin,[13] relying on their ability to infiltrate through the smear layers and partially dissolve hydroxyapatite to generate a resin-infiltrated zone with minerals incorporated.[14] If the simplified self-etch adhesive fails to penetrate beyond the smear layer and reach the mineralized dentin to form a hybridized complex, the strength of the bond may be compromised.[15]

Thus, any attempt to allow for an increased rate of water and solvent evaporation, along with deeper monomer infiltration, is likely to improve the strength of the polymer formed within the collagen fibrils and allow for the attainment of higher bond strength values and low nanoleakage within the adhesive and hybrid layer. Miyazaki and others[16] suggested that the active primer application may be helpful in removing the smear layer, thus improving the micromechanical interlocking and chemical interaction with the underlying dentin, regardless of adhesive acidity. Agitation can provide a consistent etch and enhance the interaction of acid monomers with tooth structure, dispersing etching byproducts into the hybrid layer.[17] Agitation can increase the moieties kinetics and allow for better monomer diffusion inward, while the solvents are diffusing outward and are indicated for etch-and-rinse adhesive systems.[18-19]

Only a few studies have evaluated the effects of application with agitation on the surface of self-etch systems on dentin. The ones that have usually evaluated two-step self-etch adhesive systems have reached controversial results.[17,20-21] Therefore, the current study evaluated the microtensile resin-dentin bond strength and silver nitrate uptake pattern of three one-step self-etch systems applied passively or with agitation on the surface. This study tested the following null hypotheses: 1) the mode of adhesive application does not affect the bond strength of one-step self-etch systems to dentin, 2) the mode of adhesive application does not affect the silver nitrate uptake pattern of one-step self-etch systems to dentin, 3) there is no significant difference in bond strength or silver nitrate uptake among the adhesives tested.

METHODS AND MATERIALS

Thirty extracted, caries-free human third molars were used. The teeth were collected after completion of the patients' informed consent. The University of Ponta Grossa Review Board approved this study. The teeth were disinfected in 1% thymol stored in distilled water and used within six months of extraction. The enamel from the occlusal surface of the teeth was ground by means of a wet #180-grit SiC paper perpendicular to the long axis of the tooth in order to expose a flat dentin surface. The enamel-free, exposed occlusal dentin surfaces were further polished on wet #600-grit silicon-carbide paper for 60 seconds to standardize the smear layer.

Three one-step self-etch adhesive systems were selected according to their pH values, which were provided by the manufacturers or in the literature.[1-2] Clearfil S^3 Bond (S3, Kuraray Medical Inc, Osaka, Japan)–mild (pH>2); Xeno III (XE, Dentsply Caulk, Milford, DE, USA)–moderate (1<pH<2) and Adper Prompt L-Pop (AD, 3M ESPE, St Paul, MN, USA)–aggressive (pH<1).[1-2] Their composition, application mode and batch number are described in Table 1.

A single operator applied all the adhesive systems on the dentin as follows:

1) *Passive application*: In this group, the adhesive was only spread over the entire surface for approximately three to five seconds and was left undisturbed for 15 to 20 seconds according to the manufacturer's recommendation (Table 1). Then, an air stream was applied for 10 seconds at a distance of 20 cm. The air-dry pressure that was used was 40 psi (0.27 MPa).

2) *Application with agitation*: The adhesive was rigorously agitated on the entire dentin surface for approximately 15 to 20 seconds according to the manufacturer's recommendation (Table 1). The microbrush was scrubbed on the dentin surface under manual pressure (equivalent to approximately 34.5 ± 6.9 g).[19,22] An air stream was applied for 10 seconds at a distance of 20 cm. The pressure of the air-dry used was 40 psi (0.27 MPa).

A VIP light-curing unit set with a light intensity of 600 mW/cm^2 (BISCO, Inc, Schaumburg, IL, USA) was used throughout the restorative procedure. Following the adhesive application, resin composite buildup "crowns" (Opallis, FGM, Joinville, SC, Brazil) were constructed in 1.5 mm increments and light-cured for 40 seconds each. Five teeth were used for each experimental group.

Microtensile Bond Strength Test

After storing the bonded teeth in distilled water at 37°C for 24 hours, they were longitudinally sectioned in both the "x" and "y" directions across the bonded interface with a diamond saw in a Labcut 1010 machine (Extec Corp, Enfield, CT, USA) under water cooling at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.8 mm^2. The number of premature debonded sticks (D) per tooth during specimen preparation was recorded. The specimens originated from the areas immediately above the pulp chamber, and their remaining dentin thickness (RDT) was measured with a caliper to the nearest 0.01 mm and recorded (Absolute Digimatic). The cross-sectional area of each stick was also measured with a digital caliper and recorded for subsequent calculation of the microtensile bond strength (Absolute Digimatic, Mitutoyo, Tokyo, Japan). Two sticks from each tooth were separated for silver nitrate uptake analysis under scanning electron microscopy (SEM). All the other sticks were used for the microtensile bond strength test.

Each bonded stick was attached to a modified device for microtensile testing with cyanoacrylate resin (Zapit,

Table 1: *Adhesive Systems: Composition, Groups and Application Mode*

Adhesive Systems	Composition	Mode of Application Groups	Application Mode (*)
Adper Prompt L-Pop (AD-3M ESPE, St Paul, MN, USA)	**Liquid 1** (red blister): Methacrylated phosphoric esters, Bis-GMA, initiators based on camphorquinone and stabilizers	Agitation (manufacturer's recommendation)	a, b1, c, b1, c, d
	Liquid 2 (yellow blister): Water, HEMA, polyalkenoic acid and stabilizers	Passive	a, b2, c, b2, c, d
Xeno III (XE-Dentsply Caulk, Milford, DE, USA)	**Liquid A** (green cap): HEMA, Purified water, Ethanolurethane, dimethacrylate resin, BHT, highly dispersed silicon dioxide	Agitation	a, b3, c, d
	Liquid B (black cap): Phosphoric acid modified polymethacrylate resin, PEM-F, modified methacrylate resin, UDMA, Camphorquinone, Ethyl-4-dimethylaminobenzoate	Passive (manufacturer's recommendation)	a, b4, c, d
Cleafil S^3 Bond (S3- Kuraray, Osaka, Japan)	**Adhesive:** MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl- camphorquinone, silanated colloidal silica, ethyl alcohol and water	Agitation	b3, c, d
		Passive (manufacturer's recommendation)	b4, c, d

*(*) **a**: Dispense equal amounts of Liquid A,1 and Liquid B,2 and mix liquid in mixing well thoroughly (5 seconds); b1: Apply one coat with application with agitation for 15 seconds; **b2**: Apply one coat passively and leave undisturbed for at least 15 seconds; **b3**: Apply one coat with application with agitation for 20 seconds; **b4**: Apply one coat passively and leave undisturbed for at least 20 seconds; **c**: air-dry for 10 seconds at 20 cm; **d**: light-cure (10 seconds-600 mW/cm^2).*

Bis-GMA: bisphenol A diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; BHT: Butylated hydroxy toluene; PEM-F: pentamethacryloyloxyethylcyclohexaphosphazene monofluoride; UDMA–urethane dimethacrylate; MDP – 10-Methacryloyloxydecyl dihydrogen phosphate

Dental Ventures of North America, Corona, CA, USA) and subjected to tensile force in a universal machine (Emic, São José dos Pinhais, PR, Brazil) at 0.5 mm/minute. The failure modes were evaluated at 400x (HMV-2, Shimadzu, Tokyo, Japan) and classified as cohesive (failure exclusively within dentin or composite—C), adhesive (failure at the resin/dentin interface—A) or adhesive/mixed (failure at resin/dentin interface that included cohesive failure of the neighboring substrates—A/M).

Silver Nitrate Uptake

The bonded sticks were coated with two layers of nail varnish applied to within 1 mm of the bonded interfaces. The specimens were rehydrated in distilled water for 10 minutes prior to immersion in the tracer solution for 24 hours. Ammoniacal silver nitrate was prepared according to the protocol previously described by Tay and others.[12] The sticks were placed in the ammoniacal silver nitrate in darkness for 24 hours, rinsed thoroughly in distilled water and immersed in photo developing solution for eight hours under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface.

All the sticks were wet-polished with 600 SiC paper to remove the nail varnish. The specimens were then placed inside an acrylic ring that was attached to double-sided adhesive tape and embedded in epoxy resin. After the epoxy resin had set, the thickness of the embedded specimens was reduced to approximately half by grinding with silicon carbide papers under running water. The specimens were polished with 1000, 1200, 1500, 2000 and 2500-grit SiC papers and 1 µm and 0.25 µm diamond paste (Buehler Ltd, Lake Bluff, IL, USA) using a polishing cloth. They were ultrasonically cleaned and demineralized in 50% phosphoric acid for three seconds, followed by immersion in 1% NaOCl for 10 minutes.

The specimens were then washed, air dried for 24 hours, mounted on aluminum stubs and gold coated (Sputter Coater IC 50, Shimadzu). The resin-dentin interfaces were analyzed using a scanning electron microscope (SSX-500, Shimadzu) operated in back-scattering electron mode. The working distance was 10 mm and the accelerating voltage (ACCV) was 15 kV with a small spot medium.

Three pictures from each specimen were taken. The first picture was taken in the center of the stick. The remaining two pictures were taken 0.3 mm to the left and 0.3 mm to the right of the first one. All were taken by a technician who was not aware of the experimental conditions under evaluation. The relative percentage of silver nitrate uptake within the adhesive and hybrid layer areas was measured in all pictures using UTHSCSA ImageTool 3.0 software (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA).

Statistical Analysis

The experimental unit in the current study was the tooth. The mean microtensile bond strength (MPa) and silver nitrate uptake (%) of all sticks from the same tooth were averaged for statistical purposes. The microtensile bond strength and silver nitrate uptake means for every testing group was expressed as the average of the five teeth used per group. The prematurely debonded specimens were included in the tooth mean for microtensile bond strength. The average value attributed to specimens that failed prematurely during preparation was arbitrary and corresponded to approximately half of the minimum bond strength value that could be measured in this study (ca 6.8 MPa).

Before submitting the data to the appropriate statistical analysis, the Kolmogorov-Smirnov test was performed to assess whether the data followed a normal distribution, and the Barlett's test for equality of variances was performed to determine if the assumption of equal variances was valid.[23]

After observing the normality of the data distribution and the equality of the variances, the microtensile bond strength (MPa) and silver nitrate uptake (%) data were subjected to two-way analysis of variance (Adhesive vs Mode of application) and a post-hoc test (Tukey's test at α=0.05) was used for pairwise comparisons.

RESULTS

The mean cross-sectional area ranged from 0.87 mm^2 to 0.98 mm^2 and no difference among the groups was detected (p>0.05). The remaining dentin thickness (RDT) for all specimens ranged from 2.6 to 3.1, indicating that the interfaces were located in medium dentin.[24] The percentages of specimens with premature debonding and the frequency of each fracture pattern

Table 2: *Number and Percentage of Specimens (%) According to Fracture Pattern Mode and the Premature Debonded Specimens From Each Experimental Condition (*)*

Mode of Application	*Adper Prompt L-Pop*			*Xeno III*			*Clearfil S^3 Bond*		
	A/M	*C*	*PDS*	*A/M*	*C*	*PDS*	*A/M*	*C*	*PDS*
Passive	48(90.5)	1(2.0)	4(7.5)	50(84.7)	0(0.0)	9(15.3)	37(77.1)	0(0.0)	11(22.9)
Agitation	42(82.4)	4(7.8)	5(9.8)	36(70.6)	8(15.7)	7(13.7)	54(96.4)	0(0.0)	2(3.6)

(*)A/M–adhesive/mixed fracture mode; C–cohesive fracture mode; PDS—premature debonded specimens.

mode are shown in Table 2. Differences between the adhesives were detected regarding fracture pattern. For XE and S3, a lower percentage of premature debonding occurred when application with agitation was performed. For AD, the number of premature debonded specimens was independent of application mode.

Table 3: *Overall Microtensile Bond Strength Values and the Respective Standard Deviations (MPa) Obtained in Each Experimental Condition, As Well As Statistical Significance (*)*

Mode of Application	*Adper Prompt L-Pop*	*Xeno III*	*Clearfil S^3 Bond*
Passive	17.2 ± 4.2 B	16.5 ± 3.4 B	19.4 ± 4.5 B
Agitation	29.9 ± 4.2 A	26.3 ± 4.7 A	31.8 ± 3.9 A

(*) Groups with the same letter are not significantly different (Tukey's test, p>0.05).

Table 4: *Mean Percentage of Silver Nitrate Uptake and the Respective Standard Deviations (%) Obtained in Each Experimental Condition, As Well As Statistical Significance (*)*

Mode of Application	*Adper Prompt L-Pop*	*Xeno III*	*Clearfil S^3 Bond*
Passive	52.4 ± 12.3 A	25.3 ± 10.2 B	20.1 ± 10.5 B
Agitation	58.7 ± 22.7 A	16.8 ± 4.3 B	15.7 ± 2.6 B

(*) Groups with the same letter are not significantly different (Tukey's test, p>0.05).

Microtensile Bond Strength Test

The Kolmogorov-Smirnov test confirmed the normal distribution of data in all three groups and the Bartlett's test showed no significant differences among variances in all three groups (p=0.231). The overall microtensile bond strength values for all adhesives are shown in Table 3. The cross-product Adhesive vs Mode of Application was not statistically significant, as well as the main factor Adhesive (p>0.05). Only the main factor Mode of Application was statistically significant (p=0.01). For all adhesives, higher resin-dentin bond strength values were observed in groups where the adhesives were applied with agitation (p=0.01).

Silver Nitrate Uptake

The Kolmogorov-Smirnov test confirmed normal distribution of data in all three groups and the Bartlett's test showed no significant differences among the variances in all three groups (p=0.152). The overall silver nitrate uptake values for all adhesives are shown in Table 4. The cross-product Adhesive vs Mode of Application was not statistically significant, as well as the main factor Mode of Application (p>0.05). Only the main factor Adhesive was statistically significant (p=0.01). The amount of silver nitrate uptake in the Adper Prompt L-Pop groups was twice or three times greater than that observed in the other groups (p=0.01). Representative secondary electron SEM images at the resin-dentin interfaces for the experimental conditions are depicted in Figure 1.

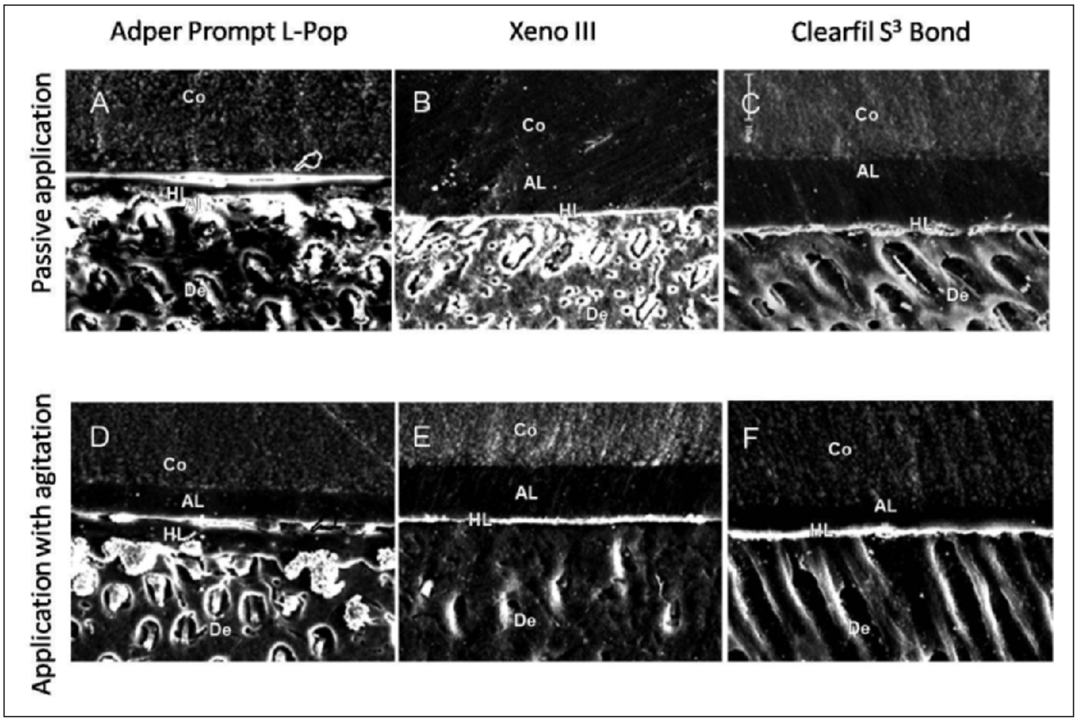

Figure 1. *Representative secondary electron SEM images of the resin-dentin interfaces bonded with Adper Prompt L-Pop (A and D), Xeno III (B and E) and Clearfil S^3 Bond (C and F). Passive application is represented by Figures 1A to 1C and active application by Figures 1D and 1F. The amount of silver penetration in Figure 1A and 1D (Adper Prompt L-Pop) was higher and largely occurred at the hybrid layer (white hand in Figure 1A and black hand in Figure 1D). The amount of silver penetration in Figures 1B and 1E (Clearfil S^3 Bond) and Figure 1C and 1F (Xeno III) was lower than in Figures 1A and 1D, with few dentin tubules infiltrated by silver nitrate (Co=composite; AL=adhesive layer; HL=hybrid layer and De=dentin).*

The amount of silver penetration for Adper Prompt L-Pop was higher and occurred both in the hybrid layer and adhesive layer (Figures 1A and 1D), regardless of the mode of application. The amount of silver penetration for Clearfil S^3 Bond (in Figures 1B and 1E) and Xeno III (Figures 1C and 1F) was lower than what was observed for AD. Silver uptake largely occurred in the hybrid layer, as no silver deposit was seen in the adhesive layer formed by CS3 and XE.

DISCUSSION

Although the one-step, self-etch adhesive system is marketed as simplified, a more complex chemistry is necessary to blend hydrophilic and hydrophobic monomers, solvents, water and additives.[1-2] Water is indispensable, because it provides the ionization medium for self-etch activity. Other solvents, such as acetone and ethanol, are necessary to dissolve both hydrophilic and hydrophobic monomers into one phase within the one-step self-etch solution,[25] but they may also help water to evaporate upon completion of the self-etch process.

Currently, almost all one-step self-etch adhesives contain HEMA, a well-known co-monomer that prevents phase separation and acts as a wetting agent and diffusion promoter for resin into exposed collagen.[1,26] One of the drawbacks of HEMA is that it firmly retains water in hydrogels from where it is hardly removed by air-drying.[10] These hydrogels may be retained within the micro-domains of the adhesive layer. They may act as hydrophilic solids that continue to draw water from the underlying vital dentin after polymerization.[27] Because of their relatively high permeability, single-step self-etch adhesives were reported to behave as permeable membranes after polymerization,[28-29] allowing the diffusion of water from the hybridized dentin to the adhesive surfaces.[2]

The silver nitrate uptake results identified silver deposits in the hybrid layer for all one-step adhesive systems tested, regardless of the application technique.[2,30] The silver nitrate uptake probably represents regions of suboptimal conversion within the polymer matrix due to incomplete removal of the solvent.[31] Water is known to inhibit the polymerization of adhesive resins,[10] and incompletely polymerized resins have affinities for specific stains.[32]

These areas of increased permeability within a polymerized resin matrix, in which water is incompletely removed from primed dentin or adhesive layers, results in regions of incomplete polymerization and/or hydrogel formation that might result in reductions in resin-dentin bond strength. In addition, these regions may permit higher diffusion of water fluxes within the hybrid layers, which might accelerate water sorption and the extraction of unpolymerized or degraded monomers.[33]

It is difficult to evaporate water from these all-in-one adhesives, and there are two ways to increase solvent evaporation from the primer mixture after its placement on the dentin surface: either by use of compressed air or by application with agitation. The use of compressed air depends on different variables, including time of use, distance between the air-spray device and the surface, pressure of air-spray and variations in the voltage, among others. Recently, several studies have proposed the use of prolonged periods of air-drying and good results were obtained with this technique.[34-36] However, controversy exists regarding the benefits of this step. When strong air-drying is used, water and solvents are evaporated quickly, resulting in a viscous resinous material with entrapped air bubbles remaining on the dentin surface. This might weaken the mechanical properties of the polymer, again lowering the bond strengths.[34] Also, this procedure reduces the thickness of the adhesive layer, making it more susceptible to polymerization inhibition by oxygen.[37] Nunes and others[38] observed that attempting to remove more solvents did not increase the extent of polymerization significantly, suggesting that other problems are impairing polymerization of one-step self-etch adhesives.

The distance between the air-spray device and the surface is another important point to be considered. Different authors evaluated variables regarding air-blowing pressure and application time for several one-step self-etch systems.[34,39] All of these authors indicated that the adhesives were applied according to the manufacturer's instructions. Only Hiraishi and others[39] and Ikeda and others[36] more adequately described the application procedures used in their studies, but unfortunately, none of them described the air syringe distance to the surface and the manufacturer's instructions of the adhesives employed.

Although the manufacturers usually recommend the application of a gentle or strong air stream, the pressure of the air emerging from the air syringe device can be highly variable, depending on how distant it is from the tooth substrate. This is why the distance from the dentin surface and the air-syringe was standardized in the current investigation. The best way to control the pressure of the air that emerges from the air syringe could be getting closer or further from the substrate, as most of the air syringe buttons are not sufficiently sensitive to deliver different air pressures to the tooth surface.

On the other hand, application of adhesives with agitation on the surface may speed up solvent evaporation in the same way as described above, while at the same time causing a higher rate of monomer incorporation inside the smear layer. The application with agitation of one-step self-etch systems is likely to carry fresh acidic resin monomers to the basal part of the etched dentin, producing a more aggressive demineralization, facilitating diffusion of the monomers and promoting a better interaction with the smear layer and underlying dentin.[16] This procedure can increase the moieties' kinetics and allow for better monomer diffusion inward, while solvents are diffusing outward. This finding was already demonstrated for simplified etch-and-rinse adhesives systems.[18-19] Although this procedure—application under agitation—allowed for an increase in resin-dentin bond strengths, it did not reduce nanoleakage within the bonding interface.[40] The agitation might have produced a better cross-linked polymer, but the chemical nature of the monomers that compose the polymer was not altered by the alternative application mode. This means that the polymer still contains hydrophilic domains capable of binding with water and, consequently, form silver nitrate deposits under the experimental conditions of the current study.

In addition, this study demonstrated that the highly acidic AD showed the highest amount of silver nitrate uptake among the adhesives tested. Although not evaluated in the current investigation, one might hypothesize that AD is likely more hydrophilic than the other self-etch adhesives. This would explain the high silver uptake due to the higher water sorption and that more hydrophilic adhesive compositions are prone to increasing bound water within the polymer matrix.[41-43] As the increase in water sorption is not caused by a reduced degree of conversion or crosslinking density of the polymer[42] within the resin matrices, the resin-dentin bond strengths of Adper Prompt L-Pop were similar to the other adhesives evaluated under the same mode of application, despite its higher nanoleakage.

Some authors recognized that application with agitation works well only for mild self-etch adhesive systems.[20] These authors believe that strong or moderate self-etch adhesives are acidic enough to etch and prime through the smear layer and into the underlying intact dentin.[44] In the current investigation, the authors have evaluated three self-etch adhesives, based on their acidity: AD is a highly acidic one-step self-etch system (pH ≅ 0.35), XE is a moderate one-step self-etch (pH ≅ 1.0) and S3 is a mild self-etch (pH ≅ 2.0).[1-2] The current investigation demonstrated that the application with agitation improved adhesive performance, regardless of the adhesive acidity.

Finally, it is worth mentioning inclusion of the premature debonded specimens in bond strength values. Instead of including them as "zero," as some research groups have already done,[45-46] the authors have assigned an arbitrary mean that corresponded to half of the minimum value that could be measured in the current investigation. The rationale behind this approach is that specimens that debonded before being tested could present an estimated "bond strength" value that would be somewhere in the range between zero and the minimum bond strength value that was measured in a specific study.[47] The specific values from the study should be taken under evaluation, since this may vary due to the universal testing machine used, operator ability, experimental conditions and more. For instance, Bouillaguet and others[48] showed that most specimens that debonded during preparation could have had bond strengths as high as 13 MPa, while Pashley and others[49] suggested that values as low as 4 MPa could not be measured using microtensile tests.

Inclusion of the premature debonded specimens as "zero" in the bond strength mean increases the standard deviation of the specific group, and this might result in the evaluation of the group being difficult due to altering the normal distribution of the data. However, regardless of the approach used in the data treatment, researchers should always provide the number/percentage of pre-test failures per group in order to add new information and avoid misinterpretation of their data.

CONCLUSIONS

It seems that, at least from an immediate viewpoint, improvements in bond strength may be achieved when one-step self-etch adhesives are applied with agitation on the dentin substrate, although this seems to not increase the hybrid layer resistance to silver nitrate uptake. However, further studies are still required to extend the use of this technique to other one-step self-etch adhesives available on the market. Studies of aged specimens are needed to identify whether the use of an application with agitation on the surface can be a useful tool to preserve resin-dentin interfaces from degradation after long-term function.

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