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A Novel Enamel and Dentin Etching Protocol Using α-hydroxy Glycolic Acid: Surface Property, Etching Pattern, and Bond Strength Studies

D Cecchin • AP Farina • CMP Vidal • AK Bedran-Russo

Clinical Relevance

Glycolic acid is a mild etchant that effectively etches enamel and dentin surfaces, a necessary step to successfully placing dental adhesive restorations.

SUMMARY

Objectives: To determine the use of α-hydroxy glycolic acid (GA) as a surface pretreatment for dental restorative applications. The etching pattern of GA pretreatment of dental hard tissues was assessed by surface microhardness and scanning electron microscopy (SEM). The effectiveness of GA surface etching on the enamel and dentin resin bond strengths was assessed using two etchant application modes (rubbing and no rubbing) and three adhesive systems (Single Bond [SB], One Step Plus [OSP], and Scotchbond Universal [SBU]).

Methods: Knoop microhardness measurements were carried out on polished enamel and dentin surfaces before and after treatment with 35% GA, 35% phosphoric acid (PA), or distilled water (control group) for 30 seconds. The microtensile bond strength test was carried out on enamel and dentin. Ultrastructural analysis of the surface and interfacial interaction was qualitatively accomplished using SEM.

Results: Etching with either PA or GA significantly decreased the enamel microhardness, with GA being significantly less aggressive than PA (*p*<0.001), while both acids showed similar decreases in dentin microhardness (*p*=0.810). SEM revealed similar etching patterns of GA and PA, while apparently a thinner hybrid layer was observed for GA groups. In

Doglas Cecchin, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Restorative Dentistry, College of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil

Ana Paula Farina, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Restorative Dentistry, College of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil

Cristina MP Vidal, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Operative Dentistry, College of Dentistry and Dental Clinics, University of Iowa, Iowa City, IA, USA

*Ana K Bedran-Russo, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA

*Corresponding author: 801 South Paulina Street, Room 531, Chicago, IL 60612, USA; e-mail: bedran@uic.edu

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dentin, the bond strengths were statistically similar between PA and GA groups, regardless of the etchant application mode ($p>0.05$). However, rubbing of GA enhanced the bond strength to enamel. PA and GA significantly increased the SBU bond strength to enamel when compared to SB and OSP ($p<0.05$).

Conclusions: GA effectively etched enamel and dentin surfaces, resulting in bond strength values similar to those associated with traditional PA. GA is a suitable enamel and dentin surface etchant for adhesive restorative procedures.

INTRODUCTION

Resin composite materials are widely used to conservatively restore enamel and dentin after caries or trauma and/or to satisfy esthetic demands. The use of dental adhesive is a necessary step to effectively bond resin composites to dental hard tissues. Dental adhesives rely on micromechanical retention of a resin polymer to enamel and dentin substrates. Such a mechanism occurs through a surface conditioning step, resulting in the superficial tissue demineralization and infiltration of resin monomers, which form resin microtags and a hybrid layer.[1,2] Dental enamel is a highly mineralized tissue composed of 96 wt% mineral, ~3 wt% water, and only ~1 wt% residual biomacromolecules.[3] On the other hand, dentin presents an intricate mineralized organic matrix comprised of ~70 wt% mineral, ~20 wt% organic components (mainly type I collagen), and ~10 wt% water.[4] While clinically successful, the adhesion of hydrophobic and hydrophilic blends of resins to the dentin organic matrix is based on a complex and technique-sensitive mechanism contributing to the short service life of resin composite restorations.[2,5] Because dentin is the bulk tissue, the complex composition and structure of dentin serve as important constraints for technological advances in adhesive dentistry.

Phosphoric acid (PA) in the form of liquid or gel, and with concentrations ranging from 30% to 40%, has been almost exclusively used as a surface conditioner of enamel and dentin prior to the placement of dental adhesives.[1] PA surface treatment increases surface wettability, roughness, and hardness of enamel and dentin.[6] Adequate resin bond strength to PA-etched enamel is highly predictable.[7,8] In dentin, PA demineralizes the peritubular and intertubular dentin, exposing a matrix rich in type I collagen fibrils.[2,9] The high acidity of PA may induce structural changes in dentin collagen[6] and activates the inactive proforms of endogenous dentin proteases associated with resin-dentin degradation.[10] It has been shown that the depth of dentin demineralization does not correlate with bonding effectiveness,[10] leading to strategies such as short application times,[6] lower acid concentrations,[11] and use of alternative dental surface conditioners.[12]

Glycolic acid (GA) is the smallest of a group of naturally occurring organic acids known as α-hydroxy acids. GA is widely used in dermatology to promote skin chemical peeling,[13] while poly(lactic-co-glycolic acid) has been used to promote wound healing in skin and bone.[14] GA is colorless, odorless, and water soluble[15,16] and is reported to elevate collagen synthesis and fibroblast proliferation in *in vivo* and *in vitro* studies.[16-18] Because of such characteristics, GA is potentially attractive for dental applications as a surface conditioner during restorative procedures. Such application is explored for the first time in this study to promote surface demineralization of enamel and dentin for resin adhesion to dental surfaces.

The aim of this study was to determine the effectiveness of GA as a dental surface conditioner for adhesion of commercially available adhesive systems to enamel and dentin. The enamel and dentin surface demineralization pattern was assessed by microhardness measurement and ultrastructural surface morphology using scanning electron microscopy (SEM). Microtensile bond strength (μTBS) to enamel and dentin was evaluated using three different adhesive systems and two application modes (rubbing and no rubbing). The null hypothesis tested was that surface conditioning of enamel and dentin using GA would result in similar surface etching pattern, surface microhardness, and adhesive bond strength to standard phosphoric acid etching.

METHODS AND MATERIALS

Study Design

The microhardness test was proposed to determine the demineralization effect of experimental etchants in enamel and dentin. The pattern of surface etching was investigated under SEM. The effectiveness of GA etching for resin adhesion was investigated by μTBS test on enamel and dentin using different etchant application modes (rubbing and no rubbing) and three adhesives (Adper Single Bond [SB; 3M ESPE, St Paul, MN, USA], One Step Plus [OSP; Bisco Inc, Schaumburg, IL, USA], and Scotchbond

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Table 1: *Materials, Application Mode, and Manufacturer (Batch No.) of Acid Solutions and Adhesive Systems*

Material	Application Procedure	Composition[a]	Manufacturer [Batch No.]
35% Phosphoric acid	Enamel: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and dry. Dentin: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and blot dry.	Dilution in distilled water from a 85% phosphoric acid solution.	Sigma [MKBR6573V]
35% Glycolic acid	Enamel: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and dry. Dentin: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and blot dry.	Preparation in distilled water using 97% Glycolic acid powder.	Sigma [BCBH8450V]
Adper Single Bond	1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s. 2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely. 3. Polymerize the adhesive with a curing light for 40 s.	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water, and ethanol.	3M ESPE (St Paul, MN, USA) [N561025]
Scotchbond Universal Adhesive	1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s. 2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely. 3. Polymerize the adhesive with a curing light for 40 s.	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane.	3M ESPE (St Paul, MN, USA) [475230]
One Step Plus	1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s. 2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely. 3. Polymerize the adhesive with a curing light for 40 s.	Bis-GMA, HEMA, BPDM, acetone. Fillers: 8.5% wt. glass ionomer.	Bisco (Schaumburg, IL, USA) [1400005745]
Filtek™ Supreme Ultra	Apply three layers of 1mm each light cured for 40 s.	Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA. Filler: Aggregated zirconia /silica clusters (20 nm silica and 4-11nm zirconia particles), average cluster 0.6-1nm; nonagglomerated /non-aggregated 20 nm silica and 4-11 nm zirconia. Filler load: 78.5% wt (63.3% vol).	3M ESPE (St Paul, MN, USA) [6028A2B]

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Composition of the materials, as provided by the manufacturers.*

Universal Adhesive [SBU; 3M ESPE]). The morphology of the adhesive interfaces was assessed by SEM. Preparation of acid solutions, composition of adhesives, application modes, and manufacturers are detailed in Table 1.

Extracted human central incisors and molars were selected, cleaned, and kept frozen (−20°C) until use. Studies on enamel and dentin were carried out on the polished enamel surface of central incisors and the occlusal dentin surface of third molars, respectively.

Preparation of Etchant Solutions

A liquid formulation of 35% GA (pH 1.2) was prepared using stock solution (70% GA; Sigma-Aldrich, St Louis, MO, USA). To eliminate potential effects of additives in commercial formulations, a

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liquid formulation of 35% PA (pH 0.12) was prepared using a stock solution (85% PA; LabChem Inc, Pittsburgh, PA, USA).

Microhardness Measurements

Enamel and dentin surfaces were exposed, embedded in epoxy resin, and polished on a water-cooled polishing unit (EcoMet 3000, Buehler, Lake Bluff, IL, USA) with abrasive paper (400-, 600-, and 1200-grit) followed by 0.9, 0.6, 0.3, and 0.1 μm diamond alumina suspensions (Metaldi Supreme, Buehler Ltd). The polished specimens were cleaned ultrasonically in deionized water for 15 minutes to remove residual polishing material. Specimens from the same tooth were etched either with 35% PA or 35% GA for 30 seconds under passive application mode (Table 1) (n=8). An unetched surface was included as a control group. Surface microhardness measurements of dentin and enamel were carried out on a microhardness instrument (Leco, LM700at, St Joseph, MI, USA) using a Knoop hardness tip and 25*g* load force for 15 seconds.[19] The microhardness of each specimen was determined by 20 indentations done after surface treatment.

Surface Etching Pattern—SEM

The surfaces of enamel and dentin were prepared as described above and etched using either active or passive application modes (n=3). The specimens were dehydrated in ascending concentrations of ethanol, fixed in hexamethyldisilazane (Sigma-Aldrich),[20] mounted on aluminum stubs, and gold sputter-coated (SEM Coating Unit E5150, Polaron Equipment, Hatfield, PA, USA), and the micromorphology of the surfaces was assessed in an SEM (S-3000N Hitachi, Tokyo, Japan).

μTBS and Adhesive Interface Morphology by SEM

Occlusal dentin of molars and buccal surfaces of upper central incisors were prepared for resin bond strength studies to dentin and enamel, respectively. The buccal surface of upper incisors was flattened with #320 grit. The occlusal enamel of molars was removed with a low-speed diamond saw (Isomet 1000, Buehler Ltd) under water-cooling to expose midcoronal dentin. Both the exposed enamel and dentin surfaces were ground with 600-grit abrasive paper for 60 seconds under wet conditions to produce a standard smear layer.[21] Incisors and molars were randomly divided into 12 groups (n=8) according to the etching protocols and adhesive strategies. Acid etching was done as described in Table 1 using either active or passive mode, and adhesive systems application was performed strictly in accordance with the respective manufacturer's instructions. After etching and bonding procedures, a nanohybrid resin composite (Filtek™ Supreme Ultra, 3M ESPE) was built in three increments of 1 mm each and light-cured for 40 seconds. All adhesive systems and resin composite were light-cured using a halogen light-curing unit operated at 600 mW/cm^2 (Optilux, Demetron Res Corp, Danbury, CT, USA).

After bonding procedures, teeth were stored in distilled water for 24 hours at 37°C and then were sectioned longitudinally across the bonded interface with a low-speed diamond saw (Isomet 1000, Buehler Ltd) under water irrigation to obtain resin-enamel or resin-dentin specimens with a cross-sectional area of approximately 0.8 mm^2. Four resin-dentin beams were obtained from each tooth, totaling 32 beams tested per group. The specimens were glued to a jig and tested in tension at a crosshead speed of 1 mm/min using a microtensile tester machine (Bisco, Schaumburg, IL, USA). The debonded interfaces were visualized under a stereomicroscope at magnifications up to 40×, and failure mode was classified as adhesive, cohesive in dental substrate (enamel or dentin), cohesive in composite, or mixed failures.[22]

Three additional resin-dentin specimens from each group were randomly selected for interfacial analysis using SEM. Specimens were embedded in epoxy resin and gloss polished using carbide paper and diamond pastes. Processing for SEM was carried out as described above and specimens were visualized in the same microscope.

Statistical Analysis

Normal distribution of the microhardness data was confirmed by Kolmogorov-Smirnov test. Enamel and dentin microhardness were analyzed by one-way analysis of variance (ANOVA) and post hoc Tukey tests ($p<0.05$). The Levene test shows that the enamel and dentin bond strength data are not normally distributed ($p=0.027$ and $p<0.001$, respectively). The bond strength of each tooth (n = 8 per group) was averaged from 4 tested beams and the enamel and dentin bond strength data were analyzed by three-way ANOVA test. When applicable, a one-way ANOVA followed by post hoc Games Howell test was performed comparing all groups ($p<0.05$). Failure mode distribution was evaluated by Chi-square test ($p<0.05$).

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Table 2: *Dentin and Enamel Knoop Microhardness Values (KHN) (Mean [Standard Deviations]) for the Etching Protocols*[a]

Groups	Microhardness, KHN	
	Enamel	Dentin
Control	311.89 (18.90) A	62.20 (3.28) A
35% Phosphoric acid	200.87 (11.56) C	49.47 (2.22) B
35% Glycolic acid	287.35 (4.83) B	48.99 (1.70) B

[a] *Means followed by different letters are significantly different at* $p < 0.05$. *Valid comparison only to column values.*

RESULTS

The microhardness results are shown in Table 2. Statistically significant differences were observed among groups for enamel and dentin ($p<0.001$). Surface treatment with either PA or GA resulted in a statistically significant decrease in the enamel microhardness ($p<0.001$ and $p=0.022$, respectively), while GA was significantly less aggressive than PA ($p<0.001$). Both acids reduced the dentin surface microhardness when compared to the control, with no statistically significant differences between PA and GA ($p=0.810$).

The enamel and dentin surfaces etched with PA and GA revealed a similar etching pattern. It is evident that both etchants exposed enamel prism rods as a result of the preferential interprismatic apatite dissolution (Figure 1a-d). Both etchants removed the smear layer, dissolved intratubular dentin, and exposed collagen fibrils, which is more evident within the dentin tubules (Figure 1e-h). No visual variations could be observed between active and passive applications.

The enamel and dentin μTBS results are depicted in Tables 3 and 4, respectively. On enamel, there were no statistically significant interactions among factors (adhesives vs acids vs application modes, $p>0.05$) and between acids ($p=0.602$). Statistically significant differences were found between application modes ($p=0.003$) and adhesive systems ($p<0.001$). Rubbing the etchant yielded significantly higher bond strength when compared to use of the no rubbing mode ($p=0.0030$). The enamel bond strength of SBU was significantly higher than that of SB and OSP ($p<0.001$), with no statistically significant different between SB and OSP ($p=0.228$). On dentin, significant interactions were observed among factors (adhesives vs acids vs application modes, $p<0.05$), except for adhesive vs acid ($p=0.882$). Overall, SBU presented the highest bond strength values, with no statistical difference compared to OSP ($p=0.110$). The dentin bond strength of the three adhesive systems was differently affected by the acid and application mode, as detailed in Table 4. Most notably, GA significantly increased the bond strength of SB and OSP when compared to PA under rubbing mode ($p>0.05$), and the bond strength of SBU was not significantly affected by the etchant ($p<0.05$).

Statistically significant differences in failure mode distribution were observed for enamel ($p=0.0106$) and dentin ($p<0.0001$) adhesive interfaces. No cohesive failures were observed for enamel. Adhesive failure was predominant for SB and OSP, and there was an increase in mixed failures for SBU, for both GA and PA (Table 3). In dentin interfaces, all failure modes were observed, but mixed and cohesive modes in resin failures were predominant.

Representative SEM images of the resin-enamel interface are shown in Figure 2. The interfaces of all adhesives exhibited similar morphology, with the presence of resin tags and micro-tags.

No visual differences were observed between etchant application modes (rubbing or no rubbing). However, the adhesive layers were thicker for the SBU when compared to the SB and OSP. Representative SEM images of the resin-dentin interfaces are shown in Figure 3. The presence of resin tags in dentin tubules was seen in all groups. An apparently thicker hybrid layer could be noted in PA when compared to GA.

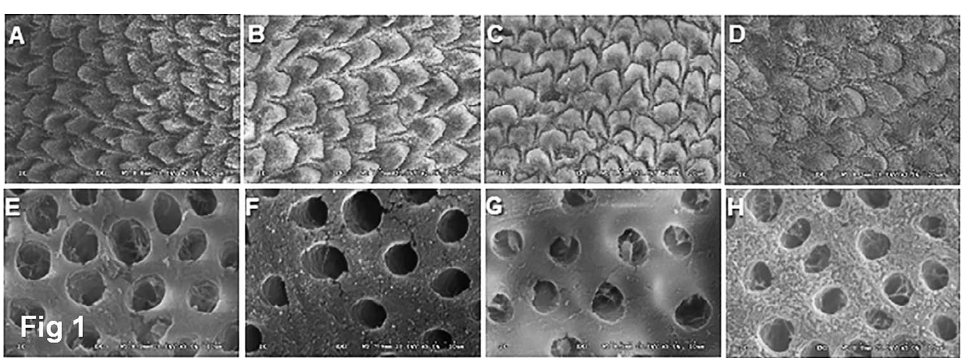

Figure 1. *Micromorphology of enamel and dentin surfaces. (a) PA no rubbing in enamel. (b) PA rubbing in enamel. (c) GA no rubbing in enamel. (d) GA rubbing in enamel. (e) PA no rubbing in dentin. (f) PA rubbing in dentin. (g) GA no rubbing in dentin. (h) GA rubbing in dentin.*

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Table 3: *Results of the Microtensile Bond Strength (μTBS) to Enamel (Means [Standard Deviations]) and Failure Mode of the Experimental Groups[a]*

Adhesive System	Enamel-resin Bond Strength			
	Phosphoric Acid		Glycolic Acid	
	Rubbing δ	No Rubbing	Rubbing δ	No Rubbing
Single Bond[b]	35.37 (7.98) (15A/15M)	35.83 (5.54) (17A/13M)	33.69 (8.49) (17A/13M)	37.76 (8.33) (15A/15M)
One Step Plus[b]	33.03 (10.42) (20A/10M)	35.80 (9.92) (18A/12M)	30.97 (8.35) (21A/9M)	35.65 (7.46) (20A/10M)
Scotch Bond Universal[a]	48.50 (9.23) (8A/22M)	50.99 (11.39) (6A/24M)	47.95 (8.30) (8A/22M)	50.58 (9.58 (3A/27M)

[a] *There were no statistically significant interactions among the study factors, adhesive vs application time vs etchant ($p>0.05$). Thus, letters and symbols next indicate statistical differences among pooled data of the study variable. Superscripted lowercase letters indicate statistically significant differences between dental adhesives ($p<0.05$). Symbol (δ) indicates statistical difference between rubbing and non-rubbing ($p<0.003$). Failure mode: A, adhesive; and M, mixed.*

DISCUSSION

In addition to being simple and clinically acceptable, acid etching is a required step in bonding techniques. The rationale for the current study is based on exploring alternative material with which to etch enamel and dentin using a less aggressive and potentially more biocompatible acid. The treatment with GA etched the surfaces of enamel and dentin, leading to significant changes in the surface microhardness and etching pattern. Such changes favored bonding of the three different commercially available dentin adhesives to enamel and dentin, with bond strength values comparable to those of PA-treated surfaces. Thus, the null hypothesis was partially rejected.

In this study, the decrease in enamel microhardness mean values was significantly greater for PA than for GA, while similar decreases in the microhardness of dentin were observed for both acids. The lower pH of PA (pH=0.12) compared to GA (pH=1.2) may have accounted for a more aggressive pattern of demineralization of the highly mineralized enamel. Furthermore, the pK_a of PA is only slightly superior to that of GA (2.16 and 3.83, respectively); thus, the acid dissociation is relatively strong for both solutions. Similar superficial demineralization patterns were observed for both acid solutions (Figure 1), although the interfacial microscopy data support a deeper demineralization of dentin by PA (Figure 3). Moreover, bond strength values for GA etching in enamel and dentin were comparable to those obtained with PA etching, with or without rubbing (Tables 3 and 4), suggesting that the depth of demineralization did not have a significant effect on the bond strength of the investigated dental adhesives. Taken together, these findings indicate that GA can be used to etch enamel and dentin in a clinically relevant condition while being relatively less aggressive than PA.

In addition to removal of minerals, PA can also interfere with dentin organic components. It has the potential to activate endogenous gelatinolytic/collagenolytic enzymes (matrix metalloproteinases and cysteine cathepsins) able to cleave collagen at the bonded interface over time, accelerating the degradation of the adhesive interface.[23-25] Such activation is of particular concern in dentin and the pH of the etching solution.[26] The effect of GA on the activation of endogenous proteases needs to be further investigated.

The findings demonstrate that acid rubbing during the etching procedure can significantly affect

Table 4: *Results of the Microtensile Bond Strength (μTBS) to Dentin (Means [Standard Deviations]) and Failure Mode of the Experimental Groups[a]*

Adhesive System	Dentin-resin Bond Strength			
	Phosphoric Acid		Glycolic Acid	
	Rubbing	No Rubbing	Rubbing	No Rubbing
Single Bond[b]	48.55 (6.92) B (1A/16M/ 1CC/11CD)	47.52 (8.15) B (5A/10M/ 5CC/11CD)	57.50 (6.90) A (2A/8M/ 7CC/13CD)	47.84 (8.20) B (7A/11M/ 9CC/3CD)
One Step Plus[ab]	46.15 (7.05) B (7A/8M/ 8CC/7CD)	52.75 (8.29) A (4A/11M/ 10CC/5CD)	53.63 (7.97) A (4A/15M/ 6CC/5CD)	51.77 (12.69) AB (4A/17M/ 7CC/2CD)
Scotch Bond Universal[a]	55.11 (6.18) A (3A/8M/ 8CC11CD)	48.05 (7.87) B (6A/12M/ 11CC/1CD)	58.15 (7.99) A (3A/9M/ 12CC/6CD)	52.51 (9.22) AB (5A/6M/ 11CC/8CD)

[a] *Different superscripted lowercase letters indicate statistically significant differences among dental adhesives ($p<0.05$). Different uppercase letters indicate statistically significant differences among groups in each row ($p<0.05$). Failure mode: A, adhesive; M, mixed; CC, cohesive in composite; and, CD, cohesive in dentin.*

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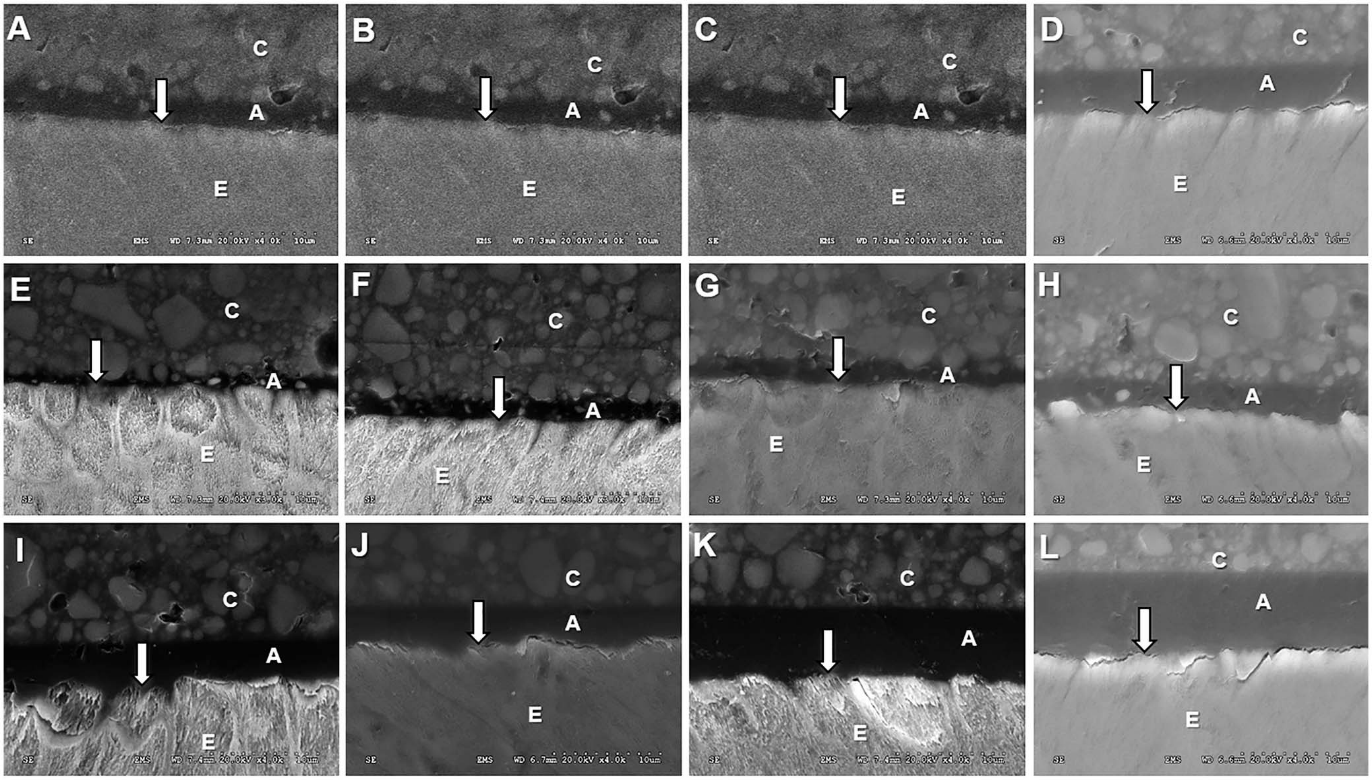

Figure 2. *Representative micrographs of resin-enamel interfaces for the different strategies (PA and GA etching; rubbing and no rubbing) and of three adhesive systems (SB, OSP, and SBU). (a) PA no rubbing and SB. (b) PA rubbing and SB. (c) GA no rubbing and SB. (d) GA rubbing and SB. (e) PA no rubbing and OSP. (f) PA rubbing and OSP. (g) GA no rubbing and OSP. (h) GA rubbing and OSP. (i) PA no rubbing and SBU. (j) PA rubbing and SBU. (k) GA no rubbing and SBU. (l) GA rubbing and SBU. A = adhesive layer; C = composite resin; E = enamel; the adhesive/enamel interface is indicated by arrows.*

the adhesive bond strength to enamel for both PA and GA. Previous studies[27-30] have shown that the active application mode of adhesive itself may improve the immediate and long-term bonding performance of self-etching systems to dentin. Active application of the adhesive improves performance of universal adhesives to enamel when compared to passive mode, with results similar to those associated with the etch-and-rinse technique.[30] In the present study, SEM analyses showed no apparent morphological differences in PA- or GA-etched surfaces with or without rubbing that could explain the outcomes. It may be possible that rubbing of the acid on enamel may have disturbed the exposed hydroxyapatite crystals and chemical composition and weakened the micromechanical retention of the enamel microtags, apparently more significantly than was seen with the GA-etched surfaces. Further characterization of mineral composition at the enamel surfaces and enamel microtag bonding mechanisms of GA to enamel could clarify the effect of application mode.

Hence, adhesive failures were predominant with SB and OSP, implying that the weak link was the bond between the resin and enamel. SBU exhibited predominantly mixed failures (Figure 3), with similar distribution between acid and acid application mode. Although mode of failure in dentin showed variation among the three adhesives, there was no difference in the failure pattern between the GA and PA groups (Table 4).

The enamel and dentin bond strength results indicate that GA is effective in promoting adhesion to dental tissues, regardless of the application mode and adhesive chemistry. It is important to emphasize that the complex composition of dentin warrants a sophisticated bonding mechanism and protocol, in which the water/solvent evaporation after demineralization is crucial and is frequently not done completely.[31] Since GA has lower depth of demineralization than PA, it is plausible that deep areas of residual water/solvent are not as frequent as in PA-treated dentin. In addition, the formation of an apparently thinner hybrid layer was evident when using the GA (Figure 3). However, hybrid layer

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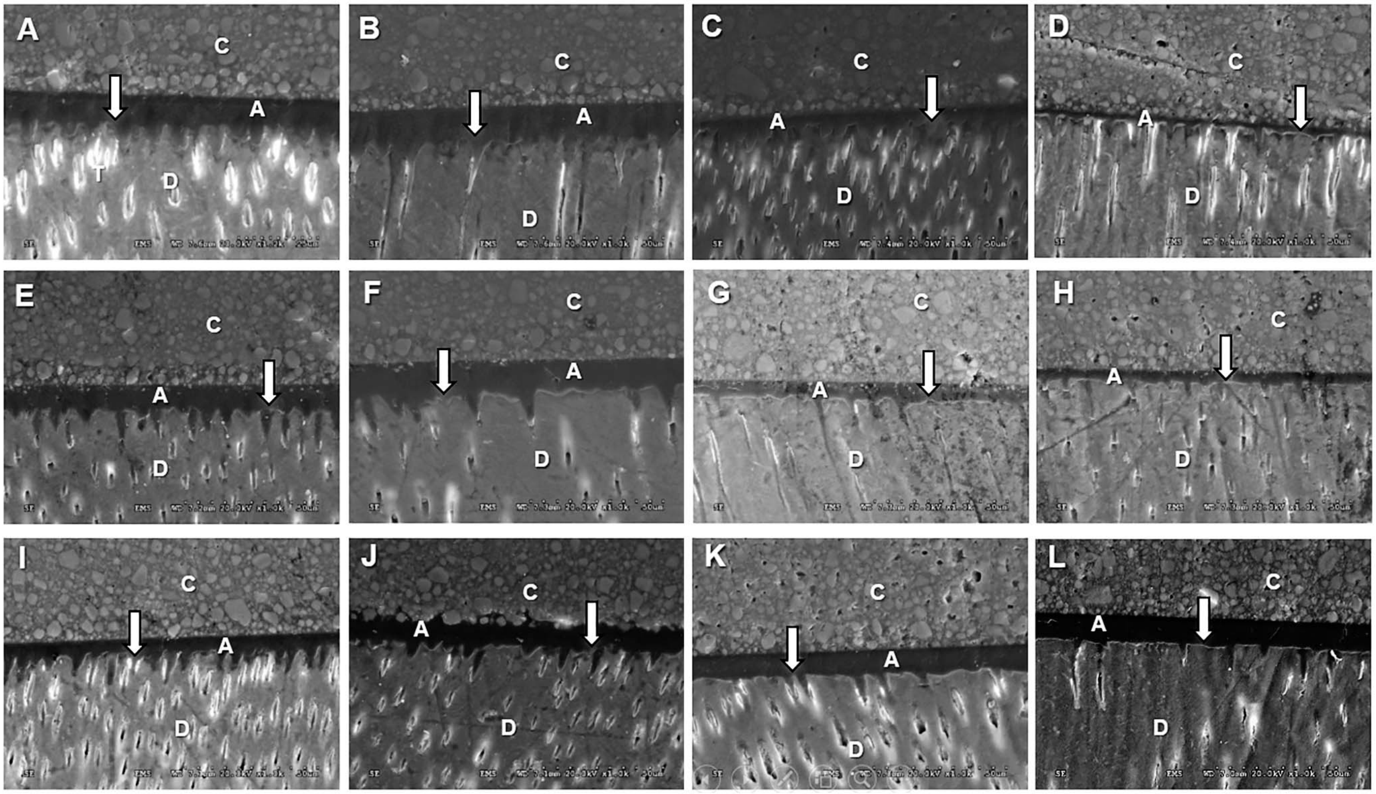

Figure 3. *Micrographs of resin-dentin interfaces for the different strategies (PA and GA etching; rubbing and no rubbing) and of three adhesive systems (SB, OSP, and SBU). (a) PA no rubbing and SB. (b) PA rubbing and SB. (c) GA no rubbing and SB. (d) GA rubbing and SB. (e) PA no rubbing and OSP. (f) PA rubbing and OSP. (g) GA no rubbing and OSP. (h) GA rubbing and OSP. (i) PA no rubbing and SBU. (j) PA rubbing and SBU. (k) GA no rubbing and SBU. (l) GA rubbing and SBU. A = adhesive layer; C = composite resin; D = dentin; H = hybrid layer; T = resin tag.*

thickness does not correlate with bond strength or better bonding quality,[32] as was also observed in the dentin bond strength data. It can be speculated that the less aggressive GA associated with the formation of thinner hybrid layers might reduce the discrepancies between demineralization and adhesive infiltration, potentially resulting in less exposed partially demineralized dentin areas prone to degradation. Long-term data and permeability studies to evaluate the hybrid layer overtime are necessary to confirm such an assumption.

CONCLUSIONS

The demineralization with either PA or GA significantly decreased enamel and dentin surface microhardness and produced similar surface demineralization morphology. The etching with GA in enamel and dentin resulted in immediate bond strength values comparable to those of PA using three different adhesive systems and adhesion strategies. GA and PA exhibited similar etching effectiveness at clinically relevant application times.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Illinois at Chicago. The approval code for this study is 2011-0312.

Conflict of Interest

The authors of this manuscript 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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